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Faculty of Nature and Life Sciences **كليـة علوم الطبيعة و الحياة**
Department of Agronomic Sciences **قسم العلوم الزراعـية**

THESIS

For the graduation of

Doctorate in Agronomic Sciences

Option

« Agricultural Production and Sustainable Agricultural Development »

Presented by

Chemouri Soumia

Theme

Impact of weeding methods on the floristic diversity and valorization of weeds in citrus orchards in the region of Tlemcen (Northwest of Algeria)

The 07th of March 2021, In front of the dissertation committee:

Quality	Last and first names	Grade	Attachment structure
Chairman	Riazi Ali	Prof.	University of Mostaganem, Algeria
Examiner	Benkhelifa Mohammed	Prof.	University of Mostaganem, Algeria
Examiner	Taibi Ahmed	Prof.	University of Tipaza, Algeria
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Co-supervisor	Tefiani Choukri	MCA	University of Tlemcen, Algeria
Guest of honor	Garcia Berrios Julián	Prof.	University of Santiago De Compostela, Spain

Academic year: 2020/2021

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If you want to go quickly, go alone. If you want to go far, go together.

"African Proverb"

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LIST OF ACRONYMS AND ABBREVIATIONS

° K: Kelvin degree
µl: Microliter
AA: Antioxidant activities
ABTS: 2, 2'-Azino-bis (3-ethylbenzothiazoline-6-sulphonic acid
ADF: Acid detergent fiber
Al: Aluminum
$AlCl_3$: Aluminum chloride/aluminum trichloride
Am.: American
ANOVA: Analysis of variance
PCA: Principal Component analysis
As.: Asiatic
Atl.: Atlantic
ATP: Adenosine triphosphate
B: Boron
BHT: 3,5-Di-tert-butyl-4-hydroxytoluene
C: Chemical weeding
C_A : Chemical plot after treatment
Ca: Calcium
$CaCO_3$: Calcium carbonate
C_B : Chemical plot before treatment
CE: Catechin equivalent
CF: Crude fiber
Ch: Chamaephyte
Circumbor.: Circumboreal
Circummed.: Circummediterranean
CO_2 : Carbon dioxide
Cosm.: Cosmopolite
Cox: Oxidized carbon
CP: Crude protein
Cr: Chromium
Ct: Control without weeding
Ct_A : Control plot after treatment
Ct_B : Control plot before treatment
Cu: Copper
DM: Dry matter
DNA: Deoxyribonucleic acid
DPPH: 2, 2- diphenyl-1-picrylhydrazyl
DSA: Direction of agricultural services
DW: Dry weight
EDTA: 2, 2′, 2″, 2‴-(Ethane-1, 2-diyldinitrilo) tetraacetic acid
EDTAE: EDTA equivalent
End.: Endemic
Eur.: European
Euras.: Eurasiatic
FAs: Fatty acids
Fe: Iron
$FeCl_2$: Iron (II) chloride/ferrous chloride
FW: Fresh weight
g a.i./ha: Gram active ingredients per hectare
G.: Geophyte
g: Gramm
GAE: Gallic acid equivalent
H.: Hemicryptophyte
H2.: Biannual
ha: Hectare
HCL : Hydrochloric acid
Ibero-Maur.: Ibero-Mauritanian
IC_{50}: Concentration providing 50% of inhibition
IPA: Isopropyl amine salt
Ir.-Tour.: Irano-Torranian
JI: Jaccard's Index
Kg: Kilogram
M: Mechanical weeding
m: Meter
M_A : Mechanical plot after treatment
Macar.: Macaronesian
Mar.: Moroccan
M_B : Mechanical plot before treatment
Med.: Mediterranean
Mg: Magnesium
mg: Milligram
min: Minute

ml: Milliliter
mm: Millimeter
Mn: Manganese
Mo: Molybdenum
MTL: Maximum tolerable level
N. Af.: North African
N: Nitrogen
Na: Sodium
NDF: Neutral detergent fiber
NDFD: NDF digestibility
nm: Nanometer
NPh.: Nanophanerophyte
NRC: National Research Council
NS: Not significant
NSC: Nonstructural carbohydrates
NW: Northwest
ºC: Celsius degree
OM: Organic matter
OMD: Organic matter digestibility
P: Phosphorus
P: Precipitations
P_2O_5: Phosphorus pentoxide
Paleo-Temp.: Paleo-Temperate
Paleo-Trop.: Paleo-Tropical
Pb: Lead
PC1: First Principal Component
PC2: Second Principal Component
PC3: Third Principal Component
PCA: Principal Component Analysis
Ph.: Phanerophyte
Prod: Production
PSM: Plant secondary metabolites
q.: Quintals
Q_2 : Pluviothermique quotient
QE : Quercetin equivalent
RE: Rutin equivalent
RNA: Ribonucleic acid
rpm: Revolution per minute
S. Af.: South African
S: Sulfur
SC: Structural carbohydrates
SEM: Standard error of the mean
Sib.: Siberian
Subcosm.: Subcosmopolite
Subtrop.: Subtropical
Sup: Superficies
T: Temperature
TDN: Total digestible nutrients
TE: Trolox equivalent
Th.: Therophyte
Thermocosm.: Thermocosmopolite,
Trop.: Tropical
UV: Ultraviolet
v/v: Volume/volume
W.-Med.: West-Mediterranean
w/v: Weight/volume
WS$_P$AS: Winter, spring, autumn, summer
Zn: Zinc
µmol: Micromole

THESIS ABSTRACT

The present thesis deals with the impact of mechanical and chemical weeding on the composition of weed flora in citrus orchards as well as the valorization of 20 % of this flora. The first part of this study was grounded on 168 surveys before and after each weeding (mechanical and chemical) in comparison to a control without weeding. A total of 88 species, belonging to 71 genera and 30 botanical families were identified. The Mediterranean elements (64%), therophytes (51%) and dicotyledons (64%) were dominant. the ANOVA revealed the existence of a significant spatial-temporal difference in the mean species richness between the stations (control *vs.* chemical and mechanical). In addition, the species richness before weeding tended to be higher than that after it. The second part was interested in the evaluation of the antioxidant activities (DPPH, ABTS and chelating power) and the phenols and flavonoids content of 19 weed species (representing 20 % of the total flora) that aimed at valorizing citrus weeds. Four species (*I. viscosa* L., *S. montana* L., *R. peregrina* L. and *R. tingitana* L.) were characterized by their richness in phenols and flavonoids and as good DPPH scavengers. Principal component analysis permitted to find two main groups: that in which *I. viscosa* L., *R. tingitana* L. and *S. montana* L. possessed relative high amounts of total phenols and good capacity for scavenging ABTS free radicals, whereas *M. sylvestris* L., *E. spinosa* (L.) Campd., *H. helix* L. and *R. peregrina* L. were good scavengers of DPPH free radicals and chelators of ferrous ions. This can be the first step for possible mixtures of these extracts in order to evaluate the possible synergistic effect among them, acting simultaneously as free radical scavengers and chelating agents. The third part treated the evaluation of the nutritive value and the mineral composition of 19 weeds which aimed to valorize these species to be used as fodder for animal nutrition. The results showed that *O. pes-caprae* was the species exhibiting the best NDF-Digestibility. As for the mineral composition, *Ch. coronarium* showed the highest K and Ca levels while *E. spinosa* presented the higher Mg and Zn concentrations. For Fe, Ch. segetum was the richest species. Also, *I. viscosa* had the best Mn content and *H. helix* recorded the higher P level. These results suppose the potential use of weeds in pharmaceutical, industrial, and cosmetic industries but also for animal feeding.

Keywords: Weeds, weeding techniques, valorization, antioxidant activity, nutritive value.

RÉSUMÉ DE LA THÈSE

La présente thèse traite l'impact du désherbage mécanique et chimique sur la composition de la flore adventice dans les vergers d'agrumes ainsi que la valorisation de 20% de cette flore. La première partie de cette étude porte sur 168 relevés floristiques réalisés avant et après chaque mode de désherbage en comparaison avec un témoin sans désherbage. Au total, 88 espèces appartenant à 71 genres et 30 familles botaniques ont été recensés et identifiés. Les éléments méditerranéens (64%), les thérophytes (51%) et les dicotylédones (64%) prédominent. L'ANOVA a révélé l'existence d'une différence spatio-temporelle significative dans la richesse moyenne en espèces entre les stations (témoin *vs.* chimique et mécanique). De plus, la richesse en espèces a tendance à être plus élevée avant qu'après le désherbage. La seconde porte sur l'évaluation des activités antioxydantes (DPPH, ABTS et le pouvoir chélatant) et à la teneur en phénols et flavonoïdes de 19 espèces d'adventices (représentant 20% de la flore totale) visant à valoriser les adventices d'agrumes. Quatre espèces (*I. viscosa* L., *S. montana* L., *R. peregrina* L. et *R. tingitana* L.) ont été caractérisées par leur richesse en phénols et flavonoïdes et comme de bons piégeurs de DPPH. L'analyse des composants principaux a permis de trouver deux groupes principaux : celui dans lequel *I. viscosa*, *R. tingitana* et *S. montana* possédant des quantités relativement élevées de phénols totaux et une bonne capacité à piéger les radicaux libres ABTS, tandis que *M. sylvestris* L., *E. spinosa* (L.) Campd., *H. helix* L. et *R. peregrina* se sont révélés de bons piégeurs de radicaux libres DPPH et chélateurs d'ions ferreux. Cela peut être la première étape pour d'éventuels mélanges de ces extraits afin d'évaluer le possible effet synergique entre eux, agissant à la fois comme piégeurs de radicaux libres et agents chélateurs. La troisième partie a traité l'évaluation de la valeur nutritive et de la composition minérale de 19 adventices qui visait à valoriser ces espèces pour être utilisées comme fourrage dans la nutrition animale. Les résultats ont montré que *O. pes-caprae* L. est l'espèce qui représente la meilleure digestibilité des NDF. Quant à la composition minérale, *Ch. coronarium* a montré les niveaux les plus élevés de K et du Ca tandis que *E. spinosa* a présenté des concentrations plus élevées de Mg et de Zn. Pour Fe, *Ch. Segetum* L. s'est avérée l'espèce la plus riche. En outre, *I. viscosa* possède la meilleure teneur en Mn et *H. helix* a enregistré le niveau de P le plus élevé. Ces résultats supposent une utilisation potentielle des adventices dans les domaines pharmaceutiques et biotechnologiques, la phytoremédiation et aussi dans l'alimentation animale, etc.

Mots-clés : Adventices, techniques de désherbage, valorisation, activité antioxydante, valeur nutritive.

ملخص الأطروحة

تتناول الأطروحة الحالية تأثير إزالة الأعشاب الضارة الميكانيكية والكيميائية على تكوين نباتات الحشائش في بساتين الحمضيات بالإضافة إلى تثمين 20٪ من هذه النباتات. يتعلق الجزء الأول من هذه الدراسة بـ 168 مسحا نباتيا تم إجراؤها قبل وبعد كل طريقة لإزالة الأعشاب الضارة مقارنة بشاهد بدون إزالة الأعشاب الضارة. في المجموع ، تم تحديد 88 نوعًا تنتمي إلى 71 جنسًا و 30 عائلة نباتية. تسود عناصر البحر الأبيض المتوسط (64٪) والنباتات الحرارية (51٪) وثنائية الفلقة (64٪) في بستان الحمضيات. كشف التحليل الاحصاءي باستعمال تحليل التباين (ANOVA) عن وجود اختلاف مكاني وزماني كبير في متوسط ثراء الأنواع بين المحطات (الشاهد مقابل الكيميائية والميكانيكية). بالإضافة إلى ذلك ، فإن ثراء الأنواع يميل إلى أن يكون أعلى من قبل إزالة الأعشاب الضارة. يتعلق الجزء الثاني بتقييم الأنشطة المضادة للأكسدة (DPPH و ABTS والقوة المخلبة) ومحتوى الفينولات والفلافونويد في 19 نوعًا من الحشائش (تمثل 20٪ من إجمالي النباتات) التي تهدف إلى تعزيز أعشاب الحمضيات. . أربعة أنواع (*R. peregrina* L. and *R. tingitana* L., *S. montana* L., *I. viscosa* L.) تميزت بغناها بالفينولات والفلافونويدات وباعتبارها كاسحات جيدة في إزالة الجذور الحرة لـ DPPH. سمح تحليل المكونات الرئيسية بالعثور على مجموعتين رئيسيتين: تلك التي تمتلك فيها كميات عالية نسبيًا من إجمالي الفينولات وقدرة جيدة على تنظيف الجذور الحرة ABTS (*I.viscosa*, *R. tingitana*, *S. montana*) بينما قد ثبت أن (*M. sylvestris* L., *E. spinosa* (L.) Campd., *H. helix* L., *R. peregrina*) كانوا كاسحات جيدة للجذور الحرة DPPH ومخلبات الأيونات الحديدية. يمكن أن تكون هذه هي الخطوة الأولى للخلطات المحتملة من هذه المقتطفات من أجل تقييم التأثير التآزري المحتمل بينها ، حيث تعمل ككاسحات للجذور الحرة وعوامل مخلبية. وتناول الجزء الثالث تقييم القيمة الغذائية والتركيب المعدني لـ 19 حشائشاً ، والتي تهدف إلى تعزيز هذه الأنواع لاستخدامها كعلف في تغذية الحيوانات. أظهرت النتائج أن *O. pes-caprae* L. هو النوع الذي يمثل أفضل قابلية هضم لـ NDF. أما بالنسبة للتركيب المعدني ، فقد أظهرت *Ch. coronarium* أعلى مستويات K و Ca بينما أظهرت *E. spinosa* تركيزات أعلى من Mg و Zn. بالنسبة لـ Fe ، تبين أن *Ch. segetum* L. كانت أغنى الأنواع. بالإضافة إلى ذلك ، كان لدى *I. viscosa* أفضل محتوى من Mn وسجلت *H. helix* أعلى مستوى P. تفترض هذه النتائج استخدامًا محتملاً للأعشاب الضارة في مجالات الأدوية والتكنولوجيا الحيوية ، والمعالجة النباتية وأيضًا في علف الحيوانات ، إلخ.

الكلمات المفتاحية: الحشائش الضارة ، تقنيات التطهير ، التثمين ، النشاط المضاد للأكسدة ، القيمة الغذائية.

General Introduction

The intra-tellian agricultural basins of the Northwest of Algeria possess an important agricultural potential. The slopes of Aïn Témouchent still retain the largest vineyard in Algeria; the Sig irrigated perimeter remains the largest olive orchard in the country; the irrigated plain El Mohammadia is one of the most important citrus-growing areas at national scale; the agricultural plain of Eghriss (Mascara) constitutes the first potato-producing region in Algeria while half of lands of the country's pulses is sown in the region and where cereals dominate (Kazi Tani, 2010).

Apart from yield losses caused by abiotic factors (climatic, edaphic, cultivation techniques, etc.), crops are subject to the effect of biotic factors and in particular weeds. Weeds are plants growing in unwanted areas that can cause a negative impact on surrounding vegetation and/or the users of the land. Weed species associated with any ecosystem depends on the crop grown, used management practices, environmental characteristics, biotic and abiotic factors (Kelton and Price, 2000).

Weeds reduce crop yields by competing for water, nutrients, and light. Some weeds release toxins that inhibit crop growth, and others may harbor insects, diseases or nematodes that attack crops. Some plants possess characteristics that make them more competitive than others. In order to reduce detrimental effects of weeds to agricultural crops, different management strategies are practiced, nevertheless, the weed management strategies are focused currently on sustainable agricultural and environmental conservation. Management strategies include a variety of cultural practices and ideas that not only control the weed flora but also help to preserve the environment. (Kelton and Price, 2000).

Mechanical weed control is an efficient method in row crops, organic farming, and minor crops (such as vegetables, fruits, or some seed crops). Mechanical weeding can assure effective weed management even if the other methods are not possible and can outperform them in some cases. mechanical weed control is done by several forms, ranging from handheld tools to the most advanced vision-guided hoes (Hussain et *al.*, 2018). In the past, mechanical weeding presented lower efficacy of intra-row weed control. However, recent progresses have resulted in the development of several implements (such as finger weeders, brush weeders, torsion weeders, mini ridgers, and computer-vision-guided hoes) that provide excellent control of intra-row weeds. (Hussain et *al.*, 2018). The mechanical weeding affects the weed density (Usman et *al.*, 2010).

Chemical weed control is practiced via herbicides (synthetic organic chemicals that do not occur naturally in the environment). Herbicides provide an effective control of weeds. However, their uses are reduced due to environmental and health issues, in addition to weeds resistance generated by the recurrent use of chemical weeding. (Labreuche et *al*., 2014). Both chemical and mechanical weeding affect the floristic diversity and they can decrease the species richness of the weed flora. Basically, good management of weeds is conditioned by a good knowledge on these species, which makes up the weed flora of each crop. For this purpose, several studies were conducted in Algeria (Melakhessou et *al*., 2020, Eddoud et *al*., 2018, Fertout-Mouri, 2018).

Beside the negative effects many weeds are useful for food, feed, or medicine. Nothing that the same plant can be a weed in one place and a beneficial plant in another.

As food, weeds are consumed by humans as green vegetables in many African, Asian, and European countries, usually by farmer's families and by rural populations, given their high nutritional value (Khan et *al*., 2013, Romojaro et *al*., 2013, Cruz-Garcia and Price, 2012).

For medicinal purposes, weeds like many plants produce a vast range of secondary metabolites (phenols, flavonoids, etc.) which are recognized to have medicinal properties, including antioxidant activity. The species, plant organs, extraction type, period and region of harvest are determining factors for the secondary metabolite contents and their corresponding antioxidant activities (Sriti Eljazia et *al*., 2018, Păltinean et *al*., 2017, Chahmi et *al*., 2015, Jan et *al*., 2014). In this context several studies were conducted to study secondary metabolites and biological activities of weeds (Haziyah Ishak et *al*., 2018, Abudunia et *al*., 2017).

To feed animals, weeds present a high forage quality, an interesting nutritional source for livestock and a high concentration of minerals such required for a healthy development of animals (Gutiérrez, et *al*., 2008, Kamalak et *al*., 2005). In this framework, divers' studies were realized on their mineral and proximate composition to be valorized from a forage viewpoint (Nashiki et *al*., 2005, Marten and Andersen, 1975). The stage of maturity at harvest plays a major role in determining the quality of a forage, in addition to the weather conditions (Ramirez Lozano, 2015, Newman et *al*., 2009).

Weeds are diverse and very abundant in nature and in cultivated fields, belonging to different botanical. Since they are generally considered as undesirable and usually targeted to be eliminated, they could be valorized to be used as a source of secondary metabolites and antioxidants or as fodder for livestock. Within this framework, the present thesis fits.

The main objectives of the current thesis are:

1. Study of the weed flora associated with citrus orchards by comparing the impacts of mechanical and chemical weeding on their floristic composition compared to a control without weeding.
2. Evaluation of the capacity of the aqueous extracts of nineteen weed species, from the inventory of the weed flora of citrus orchards from Algeria (Northwest of Algeria) for scavenging free radicals such as 2, 2- Diphenyl-1-picrylhydrazyl (DPPH) and 2, 2'-Azino-bis (3-ethylbenzothiazoline-6-sulphonic acid (ABTS), as well as for chelating metal ions,) , and the establishment of the link between these antioxidant activities and phenol and flavonoid content. The aim is valorizing these weeds through their applications as antioxidants in cosmetic, food and pharmaceutical industries,
3. Evaluation of the nutritional value and mineral composition of nineteen weed species from the inventory of weed flora in citrus orchards in Algeria (Northwestern Algeria). The aim is to enhance the value of these weeds through their valorization as a source of nutrients in animal feeding or fertilization in the case of their return to the soil.

This thesis includes two main sections, Literature Review and Experimental Trials.

Literature Review: This section is made up of 4 chapters.

Chapter I: Description of the study area.

Chapter II: Weed flora and weed management.

Chapter III: Secondary metabolites and antioxidant activity.

Chapter IV: Minerals and nutritive value.

Experimental Trials: This section contains 3 chapters.

Chapter V: Impact of mechanical and chemical weeding on the floristic diversity of citrus orchards in Tlemcen (Northwestern Algeria).

Chapter VI: Total phenol and flavonoid contents and the *in vitro* antioxidant activities of nineteen weed species from Northwest of Algeria.

Chapter VII: Nutritional value and mineral composition of nineteen weed species growing in citrus orchards for their use as potential source of nutrients in ruminants feeding

Section 1

Literature Review

Chapter I

Description of the study area

Literature Review

Chapter I

Description of the study area

Contents

1. Introduction

The study of the research environment is important to any scientific study and generally represents the first step in any investigation. It allows to give some descriptive elements that can highlight on the factors and aspects likely to influence the study and its results. In this part, a description (geomorphological, geological and pedological characteristics and hydrography) of the study area will be provided, as well as a bioclimatic synthesis based on meteorological data for 34 years (1981-2015). Also, the crop and animal productions of the study area will be presented. The meteorological and the production data of the study region were provided by the direction of agricultural services of the Tlemcen region (DSA).

2. Geographical delimitation of the study area

The study area corresponds to Tlemcen region, which is geographically located in the extreme Northwest of Algeria at 1 º 27 ’and 1 º 51’ west longitude and 34 º 27 ’and 35 º 18’ north latitude. It is bounded by the Mediterranean Sea to the north, the wilaya of Naama to the south, the Algerian-Moroccan border to the west, the wilaya of Sidi Bel Abbès to the south-east and by the wilaya of Ain Témouchent to the north-east. It covers an area of 9,017.69 km^2, it includes 20 daïras subdivided into 53 municipalities. The relief of the wilaya of Tlemcen presents an orographic heterogeneity offering a significant diversity of landscapes. The experimental trials of the current thesis were conducted in the plain of Hennaya which is located to the North of Tlemcen city in Tafna basin.

3. Geomorphological and geological characteristics

In the geological and geomorphological context, four main geographical sectors have been described for the study area (Figure I.1): The coast, the interior plains, the Tlemcen mountains, and the high steppe plains. The coastline is mainly represented by the Traras mountains and the Sebaa Chioukh Hills, occupies an area of 211000 ha. The Traras massif is formed by a series of parallel ridges with an altitude varying from 500 to 1000 m and culminating at 1136 m at Jebel Fillaoucène. The Traras mountains are mainly composed of limestone, marl, marly limestones, and basaltic and schist rocks. The Sebaa Chioukh hills are located to the east of the Traras mountains with an altitude between 600 and 800 m (Benest, 1985, Guardia, 1975).

The interior or Tellian plains are formed mainly by flat geomorphological structures (Figure I.2), extend over an area of 32100 ha (Mekkioui, 1989). The average altitude of these plains oscillates between 200 and 400 meters. These are lands for agricultural purposes.
The mountains of Tlemcen form a real natural barrier between the steppe and the Tell (Figure I.3), at an altitude of 800 to 1,700 meters, which peak at 1,843 m at Jebel Tenouchfi, not exceeding 20 km in width (Tinthoin, 1948). This mountain range is part of the Jurassic system starting in Morocco at Moulouya and extending to the mountains of Saida and Tiaret (Boudy, 1955). The sandstones of Boumediene, the limestones of Zarifet, the dolomites of Terni, the Marno-limestones of Raouraï, the limestones of Lato, the Marno-limestones of Hariga and the sandstones of Merchiche present the stratigraphic series of the Tlemcen mountains (Benest, 1985). The Tlemcen steppe highlands constitute a tabular area with an average altitude of 1100 m. They form a geomorphological unit characteristic of the Atlas domain, occupying an area of 24800 ha (Mekkioui, 1989). The Quaternary terrain that constitutes the vast tabular expanse is represented by two distinct formations: the old Quaternary alluvium and the recent Quaternary (Bouabdellah, 1991).

4. Pedological characteristics

The soils of the study region are diverse due to the important lithological geomorphological and climatic variability. Overall, the region of Tlemcen is generally characterized by fertiallitic, brown and calcareous soils (Kaid Slimane, 2000). The coastline is characterized by unsaturated soils, decalcified soils, limestone humus soils, calcic soils, and balanced soils (Mesli, 2001).

For the soils of the Tellian plains, they are soils subject to leaching phenomena. The presence of red colluvial soils, brown calcareous soils and soils formed on tuff or friable limestone is recorded. The mountains of Tlemcen are represented by red Mediterranean soils, leached and podzolic soils. Finally, the soils of the steppe zone are based on marly formations sometimes associated with limestone and gypsum flows (Mesli, 2001, Tinthoin, 1948).

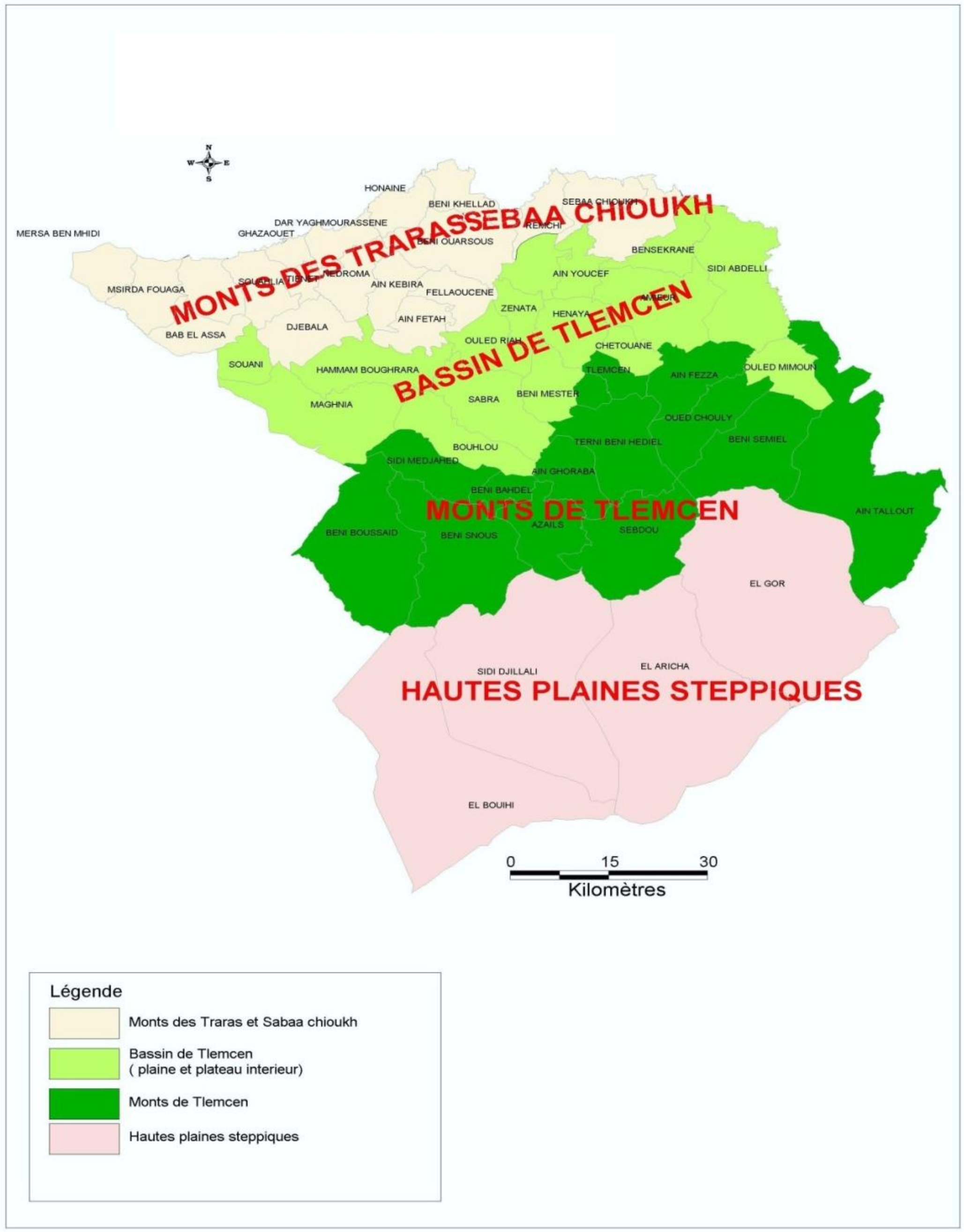


Figure I.4. Geographical sectors of the study area (Bouabdellah, 2008).

Translation of the legend: Monts de Traras et Sebaa Chioukh: Mountains of Traras and Sebaa Chioukh, Basin de Tlemcen (plaines et plateaux intérieurs) : Tlemcen bassin (interior plains and plateaus), Monts de Tlemcen: Mountains of Tlemcen, Hautes plaines steppiques : High steppe plains.

5. Hydrography

The rivers of Algeria are spasmodic and intermittent, for which they have prevailed the name of Arabic origin "wadi". The study region is marked by an important hydrographic network which consists of main and secondary wadis. There are several tributaries and chaâbat which feeds the main wadis (Tinthoin, 1948). The main rivers are wadi Tafna, Isser, and Sekkak.

The Tafna wadi is the most important watercourse in the wilaya of Tlemcen with 170 km long where it takes it source in the mountains of Tlemcen. The course of this wadi can be subdivided into three parts: upper Tafna, middle Tafna and lower Tafna (Bouanani, 2004).

The upper Tafna, the wadi begins in the wadi Ouriach and asserts itself after the junction of a large number of ramifications dug in the Jurassic lands and descending from ridges reaching 1500 m. These ramifications meet around Sebdou at an altitude of 900 m. From this place and up to Sidi Medjahed, the wadi follows a course in a deep valley dug in the Jurassic lands. In this mountainous region, wadi Tafna receives wadi Khemis (right bank) and wadi Sebdou (left bank). The middle Tafna, from Sidi Medjahed, the wadi penetrates into the tertiary basin and flows in a shallow valley in more or less clayey soils. This part of the tertiary basin is crisscrossed by numerous tributaries (wadi Mouilah, Boukiou, Boumessaoud, Zitoun and Isser). The lower Tafna, the lower course of the Tafna stretches from the Tahouaret gorges towards the village of Pierre Chat to the beach of Rachgoune in the Mediterranean Sea, over 20 km.

The wadi Isser is the second in size, it originates from the source of Ain Isser (South of Ouled Mimoun), in the synclinal gutter of Meurbah in the valley of Béni Smiel. This wadi covers an area of 1140 km^2 with a maximum altitude of 1625 m. The length of the main thalweg is 81 km.

The downstream limit of the basin coincides with the El Izdihar dam of Sidi Abdelli (put in service in January 1989). Its two main tributaries are wadi Tallout and wadi Lakhdar. As for its confluence with wadi Tafna, it takes place in the plain of Remchi at an altitude of 80 m.

Wadi Sekkak is a left bank tributary of the Isser wadi (itself a right bank tributary of the Tafna) with which it flows north of Aïn Youcef, originates on the Terny plateau south of Tlemcen at the source of Aïn Rhannous (Bouanani, 2004, Chebbani, 1996).

The Sikkak wadi basin includes two very distinct sectors: in the north and in the center, depressions filled with tertiary and quaternary sediments, and a mountainous area which includes to the South, the Terny plateau surrounded by several high massifs culminating in Djebel Nador (1579 m) (Bouanani, 2004).

6. Flora and vegetation

On the coast, the nature of the climate explains the predominance of the tree layer of certain forest species such as Thuja of Berber (*Tetraclinis articulata* Wahl.), Aleppo pine (*Pinus halepensis* L.) and red juniper *(Juniperus phoenicea* L.*)*. While the interior plains (Maghnia, Hennaya, Remchi, etc.) have a potential in soil of high agro-pedological value which is dominated by agricultural activities. For the mountains, the vegetation is characterized by a diversity of physiognomic structures and composition in the shrubby and bushy strata due to the geographic, geological, and climatic variety offered by the Tlemcen mountains. Berber Atlas cedar (*Cedrus atlantica* Endl.), Aleppo pine (*Pinus halepensis* L.), holm oak (*Quercus ilex* L.), kermes oak (*Quercus coccifera*), cork chain are the main species that form the forest cover.

For the mounts of Tlemcen, Babali et *al.*, (2013) reported the presence of Oak Forests, represented by four major species of the genus Quercus (*Quercus ilex* L., *Quercus suber* L., *Quercus coccifera* L. and *Quercus faginea* subsp. tlemcenensis (DC.) Maire and Weiller), Conifers represented by Thuja (*Tetraclinis articulata* (Vahl) Masters).

As for the steppe, the vegetation cover is distinguished in two main groupings, the first is mainly made up of meadows-forest to near-steppe stands formed by a series of holm oak and Aleppo pine with an undergrowth made up of rosemary, dwarf palm and Alfa tree in mountainous areas. The second characterizes the steppe environment itself, made up of hydrophilic or halophilic vegetation.

7. Agricultural potential

The study region has a total agricultural area (S.A.T) of 537274 ha, while the used agricultural area (S.A.U) is around 350285 ha and which represents 65% of the S.A.T of the total region. Overall, the use of inputs (fertilizers, herbicides, etc.) and driving and operational machinery in agriculture in the study region remains low.

Table I.1. Partition of agricultural lands in the study region (DSA, 2016).

	S. A. T (ha)	S. A. U (ha)	Irrigated lands	Plowed lands	Permanent crops	Greenhouse crops	Other lands used by agriculture	
							Grazing lands	Non-product lands
Hennaya	9502	9284	1797	8047	1237	0	0	218
Total region	537274	350285	28700	309569	40716	215.00	153964	33025

S.A.T: Total agricultural area, S.A.U: Used agricultural area. Superficies in hectare (ha).

7.1. Vegetal production

Tlemcen is an agricultural region where several crops are practiced. The irrigated lands represent 8.19%, which are mainly concentrated in the plains and interior plateaus. Cereal culture is practiced in the coast, the interior plains and around the mountains of Tlemcen. Whereas grazing and breeding animals are mainly concentrated in the high steppe.

The production reached for the various crops from 2005 to 2016 is shown in Table I.2. The data were provided by the DSA of Tlemcen.

Vegetable production occupied the first rank with an average production of 2698149.4 q, from 2005 to 2016, followed by cereals with a mean production of 1184536.4 q. Forage production ranked third with 839873.6 q. Fruit production was in the next position (533950.3 q). Next came olives production with an average of 252789 q. The following was citrus production with 209676.4 q. After ranked viticulture (average of 147745.5 q). Dry vegetables registered 90696 q. At the end, the fig production took place with a mean value of 18051.81 q.

Table I.2. Crops production of the region of Tlemcen (DSA, 2016).

Production type	Crops production by crop year (quintals, q)										
	2005-2006	2006-2007	2007-2008	2008-2009	2009-2010	2010-2011	2011-2012	2012-2013	2013-2014	2014-2015	2015-2016
Cereals	258000	535160	131000	1567150	1308100	521500	1694200	2610000	1449790	2355000	600000
Forage	371000	673590	360150	558000	673050	652830	797020	1158020	1118850	1033200	1842900
Dried leguminous vegetables	32106	44120	20000	81910	97950	94685	111800	154830	144195	124050	92010
Vegetable crops	3020050	1538700	1808460	2138800	2218000	2130000	2151340	2950700	3294100	4188000	4241493
Fruit crops	366600	234570	253180	364300	375200	498600	440140	720060	769300	989778	861725
Fig trees	17700	13245	13695	18850	22800	19250	17670	27450	18600	20000	9310
Viticulture	125935	77400	88520	113600	95930	106440	152550	200000	186100	241269	237456
Industrial cultures	0	0	0	0	0	0	0	0	0	0	0
Citrus	121200	113200	120000	140700	150290	182100	180000	244700	326240	364080	363930
Olive	124500	86500	79900	130300	192440	269030	105000	220000	338000	550000	685000

7.2. Citrus production

7.2.1. In Tlemcen

The irrigated superficies are occupied principally by arboriculture, more particularly citrus crops that are cultivated in the plains and interior plateaus. The citrus crops dominate in the valorised perimeters (irrigated perimeters) in the Tafna, Isser and Sikkak valleys. The insufficiency of irrigation water remains the limiting factor for citrus fruit production in the region. The citrus production in the study region recorded an average production of 209676 q from 2005 to 2016 (Table I.2).

Hennaya covers 9284 ha of the total S.A.U of Tlemcen, representing 2.65% of these superficies (Table I.1). Concerning citrus fruits, Hennaya occupies the first position with an area of 590 ha thus representing 23% of the total area occupied by citrus fruits in the Tlemcen region. Maghnia ranks second with 559 ha (22%) and after comes Remchi, occupying 468 ha (18%). El Fhoul was the next with 227 ha (9%), and finally Chetouane take the fifth position with 205 ha (8%) (Table I.2).

7.2.2. In Hennaya

For Hennaya, the citrus superficies represent 28% of the S.A.U of the region (2568 ha), with an average production of 91,376.67 q for the period from 2014 to 2017. The main varieties cultivated are Thomson navel, Washington navel and Valencialate (Table I.3).

Table I.3. Citrus production in Hennaya (Tlemcen, Algeria), for a period 2012-2017 (DSA, 2017).

Citrus varieties		2012-2013	2013-2014	2014-2015	2015-2016	2016-2017
Thomson navel	Sup	272	352	360	360	367
	Prod	18240	53280	44400	44400	74000
Washington navel	Sup	113	131	130	130	130
	Prod	8160	18000	12000	12000	25000
Clementine	Sup	52	52	52	52	55
	Prod	3000	5000	5000	5000	5000
Mandarin	Sup	2	2	2	2	2
	Prod	100	200	200	200	200
Portuguese	Sup	2	2	2	2	2
	Prod	240	200	200	200	200
Valencialate	Sup	74	76	76	76	76
	Prod	5920	7600	7600	7600	15200
Lemon	Sup	21	21	21	21	21
	Prod	1520	2850	2000	2000	5000
Total	Sup	536	636	636	643	653
	Prod	37180	87130	78130	71400	124600

Sup: Superficies in hectare (ha), Prod: Production in quintals (q).

7.3. Livestock and poultry

The study region counted over the period from 2005 to 2016 an average of 32636 head of beef cattle, 20483 head of dairy cattle, 508425 head of sheep, 37671 head of goats, 784063 head of laying hens, and 9994870 head of meat hens (Table I.4). The breeding of animals is practiced mainly in the high steppe plains under grazing. Ruminants breeding and poultry are the most dominants in the region. Ruminant breeding is mainly practiced in extensive, relying on the rangelands to ensure feeding.

During the agricultural year 2015-2016, four zones El-Aricha (86637 head), Bouihi (75120 head), El-Gor (73670 head) and Sidi Djillali (73548 head) had the most important sheep herds in terms of number, and it is in these regions that this type of breeding is practiced. These four regions alone account for 48% of the total sheep herd in the study region.

Terny, Maghnia, El-Gor and Ain Ghoraba are the areas which have the largest cattle herd (beef and dairy cattle) in Tlemcen, they alone represent 51% of the total of the wilaya. Whereas Boughrara, Ain Ghoraba, Sidi Djillali and Terny occupied the first positions in terms of the importance of the goats' herd, they represent 26% of the total region.

For poultry, the production of laying hens is mainly concentrated in Ain Tellout, Zenata, Bab Assa and Hennaya, thus representing 62% of the total in the study region. As for meat hens, Djebala, Ain fezza, Beni Ouarsous and Nedroma are the areas which have the greatest production of broilers, thus representing 41% of the total region.

7.4. Animal production

The study region produces several animal products which ensure the self-sufficiency of its inhabitants. The important part is chiefly provided by rural areas. The animal productions (red meats, white meats, milk, Eggs, wool, and honey) of the region, from 2005 to 2016 are presented in Table I.5.

The region under study recorded in the period from 2005 to 2016 an average production of 14,941 tons of red meat, 15292 tons of white meat, 54679 liters of milk, 148053 units of eggs, 462 tons of wool and 141 tons of honey (Table I.5). Eggs and milk productions occupied the first ranks in the study region.

Table I.4. Livestock and poultry potential of the region of Tlemcen (DSA, 2016).

Livestock and poultry	Unit	Crop year (2005-2016)										
		2005-2006	2006-2007	2007-2008	2008-2009	2009-2010	2010-2011	2011-2012	2012-2013	2013-2014	2014-2015	2015-2016
Beef cattle	Head	26800	26000	26750	27400	28600	29980	30400	34500	43020	45550	40000
Dairy cattle	Head	-	-	-	16200	16600	17620	17950	18900	25680	26810	24100
Sheep	Head	395000	370600	420000	440000	460000	521000	515000	520000	532300	768770	650000
Goats	Head	30200	33500	34800	34600	35500	37280	37600	41500	43200	43200	43000
Laying hens	Head	-	-	-	646300	691000	663000	750000	870000	896200	878000	878000
Meat hens	Head	-	-	-	6083200	7945000	8917900	10490900	13365000	11116960	10500000	11540000

-: not provided.

Table I.5. Animal production of the region of Tlemcen (DSA, 2016).

Designation	Unit	Crop year (2005-2016)										
		2005-2006	2006-2007	2007/2008	2008-2009	2009-2010	2010-2011	2011-2012	2012-2013	2013-2014	2014-2015	2015-2016
Red meats	T.	6400	7002	7766	6833.4	7612	76100	7,990	9,033	9749	11138	14723
-Sheep meats	T.	4700	5153	5680	5026	5410	54300	5,400	6,034	6840	7447	10878
-Beef meats	T.	1520	1650	1820	1520	1932	20000	2,210	2,536	2523	3304	3426
-Goat meats	T.	180	199	266	287.4	270	1800	3,800	463	386	387	419
White meats	T.	8275	9573	6790	8950	11530	16600	19,950	23,660	21300	21110	20472
Milk	Liter	33900	32000	30000	36323.6	38100	42100	48700	73130	79260	88530	99426
Eggs	Unit	111600	136800	130500	140500	148500	158900	165,000	207,500	198	210710	218378
Wool	T.	400	241	324	410	420	430	440	600	600	616	600
Honey	T.	42	93.5	66	150	185	160	150	200	163	180	165

T.: Tons.

8. Bioclimatic synthesis

8.1. Weather and climatic conditions

The climate is defined as the set of successive states of the atmosphere in a given region and during a given period. It is generally studied over thirty years (Emberger, 1952). For the climate of the region of Tlemcen, it belongs to the Mediterranean climate with a diversity of bioclimatic stages recorded at the local scale according to the exposure, the topography, the proximity of the sea, the altitude, and the plant cover (Benabadji and Bouazza 2000, Aimé 1991).

For the study area, the data collected at the Zenata meteorological station during a period from 1981 to 2015 was used, in order to characterize, the average monthly temperatures, the average monthly precipitation and the seasonal distribution of precipitation. The characteristics of this meteorological station are shown in the Table I.6.

Table I.6. Geographic coordinates of the meteorological station and period of observations.

Station	Altitude	Longitude W	Latitude N	Period
Zenata	249 m	W 1 ° 27'	35 ° 01 N'	1981-2015

8.2. Average monthly precipitations P (mm)

The term "precipitation" includes all meteoric waters that fall on the surface of the earth, whether in liquid (rain) or solid (snow, hail) form (Emberger, 1952). The study of precipitations is important, because in the Mediterranean region, the main limiting factor is water, although it is not the only one. In Algerian agriculture, precipitations are the only water source for cultures called dry crops (Kazi Tani, 2010). The average monthly precipitations recorded in the station is presented in the Figure I.2.

The Figure I.2 revealed an irregularity in the monthly distribution of precipitations on an annual scale. The maximum of precipitations is very marked in November, December, January, February, and March; these months total more than two thirds of the precipitations. While the minimum precipitations are recorded in the three summer months (June, July, August) which receives only insignificant amounts (dry period).

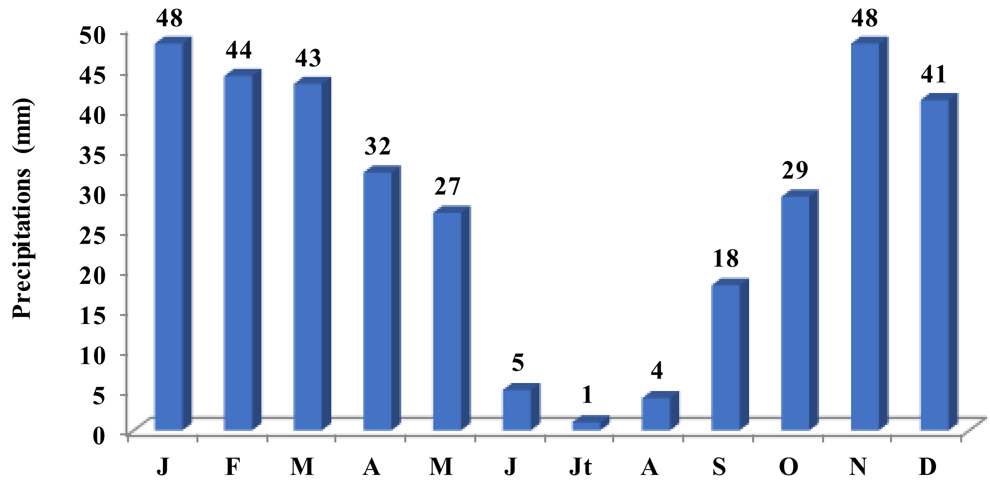


Figure I.5. Average monthly precipitations P (mm).

8.3. Seasonal distribution of precipitations P (mm)

The seasonal distribution of precipitation P (mm) is a concept which consists in calculating the sum of precipitation by season and in carrying out the seasonal classification in order of decreasing rainfall by designating each season by its initial (Winter (W), Spring (SP), Summer (S) and autumn (A)). The seasonal precipitations of the Zenata station are summarized in the Figure I.3.

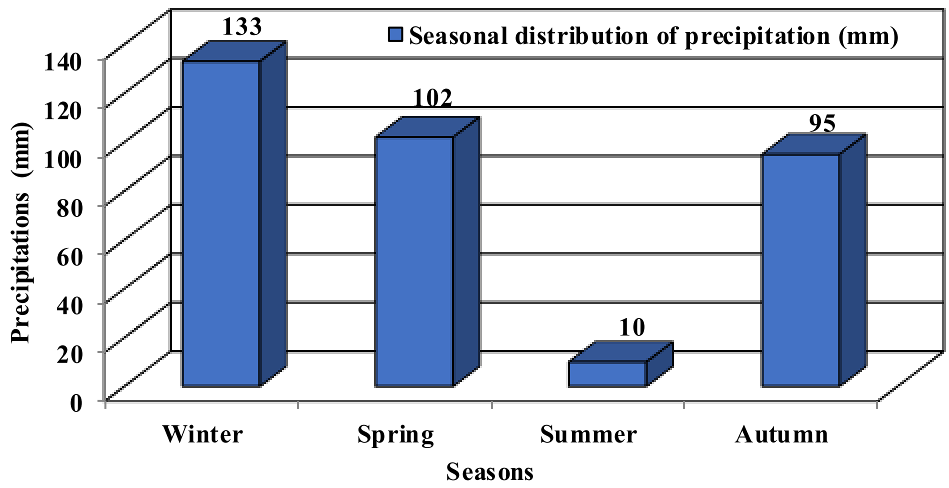


Figure I.6. Seasonal distribution of precipitations.

The station of Zenata receives 340 mm of precipitations annually. The study region is subject to a seasonal "WSPAS" regime, the maximum precipitation is recorded in winter with a rate of 39.11% of total annual average precipitation, while spring occupies the second position with a rate of 30% (Figure I.3).

8.4. Average monthly temperatures T (° C)

After precipitations, temperature is the second important factor in the climate. The monthly and annual temperature variations are used to establish the isothermal lines either by year or by month. This variation takes place between the minima and the maxima (Emberger, 1952). The monthly average temperatures of the station are given in Figure I.4.

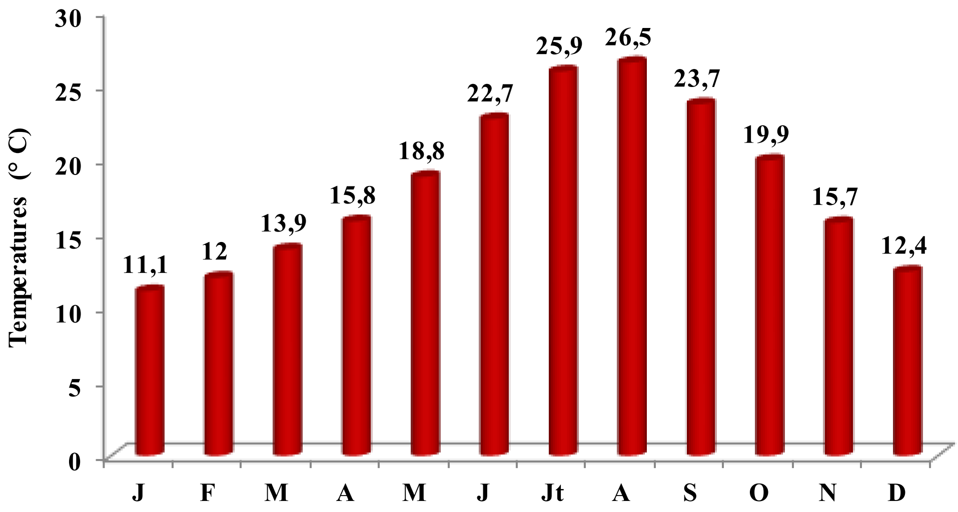


Figure I.7. Average monthly temperatures T(° C).

The average annual temperature is around 18.2 ° C. The coldest month corresponds to January (11.1 ° C) while the warmest month coincides with August (26.5 ° C).

8.5. Ombrothermic diagram of Bagnouls and Gaussen (1953)

Bagnouls and Gaussen (1953) developed a climatic classification of the Mediterranean region which is established by in plotting on a graph:

- On the x-axis, the months of the year.
- On the y-axis on the right, the monthly precipitation in mm.
- On the y-axis on the left, the average monthly temperatures in ° C at a on a scale twice that of precipitation.

The diagram shows the dry and wet periods. According to Bagnouls and Gaussen (1953), a month is dry if the total precipitation (mm) is less than or equal to twice the temperature (° C). A month is considered dry when the temperature curve is higher than that of precipitation. The ombrothermic diagram of the Zenata station is presented in Figure I.5. The study area is characterized by a dry season of 6 months, from May to October (Figure I.5).

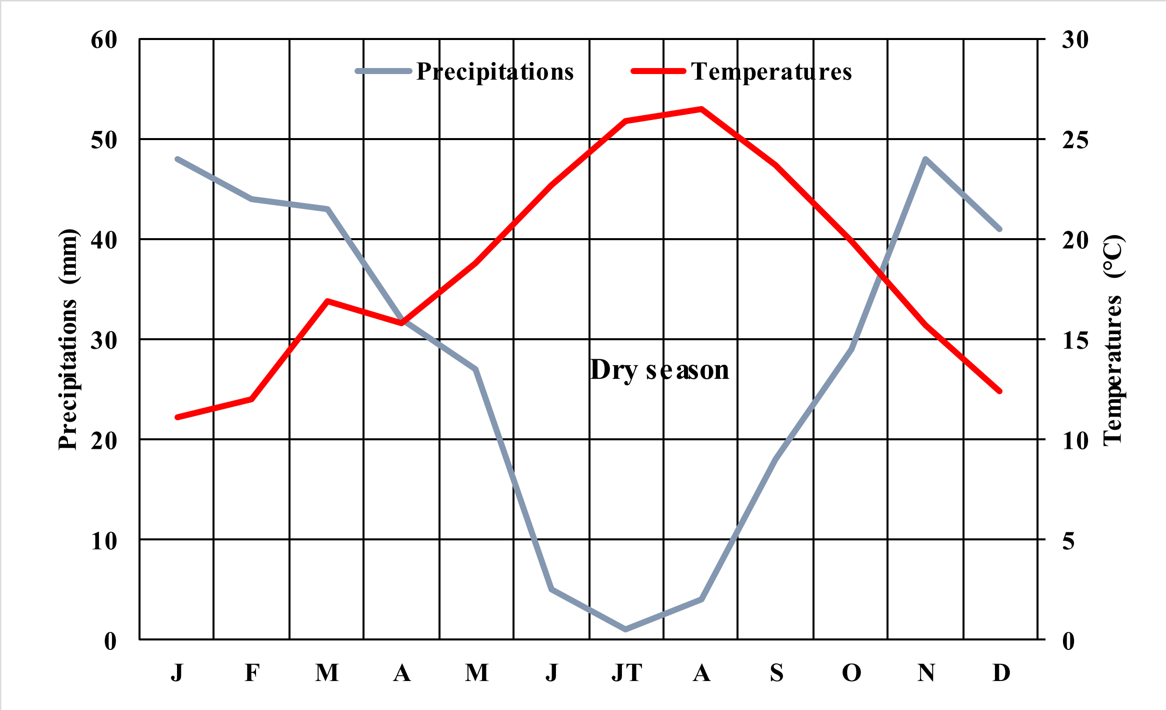


Figure I.8. Ombrothermic diagram of the study region (Zenata station) from 1981 to 2015.

8.6. Quotient pluviothermique (Q_2) and climagram of Emberger

Emberger's pluviothermal quotient characterize the Mediterranean climate and its nuances. Emberger (1930) reported that in Mediterranean regions, thermal amplitude is an important factor in the distribution of vegetation.

The quotient pluviothermique (Q_2) is calculated in order to determinate the climate of the study area and to locate it on the climmagram of Emberger. The pluviometric parameter considered the average number of days of precipitation per year by the average precipitation. For temperatures, it considers the average (m) of the minimum temperatures of the coldest month and the average (M) of the maximum temperatures of the hottest month. The quotient is expressed as follows:

$$Q_2 = 2000P/M^2 - m^2$$

- P: Annual precipitation in mm.
- M: Average temperatures of the maximums of the hottest month (kelvin).
- m: Average minimum temperatures for the coldest month (kelvin).
- Kelvin degree: (T ° K = T ° C +273.2).

By plotting the value of the Q_2 (43.06) on the Emberger climagram, it turned out that the region is located in a semi-arid bio-climatic stage with temperate winter (Figure I.6).

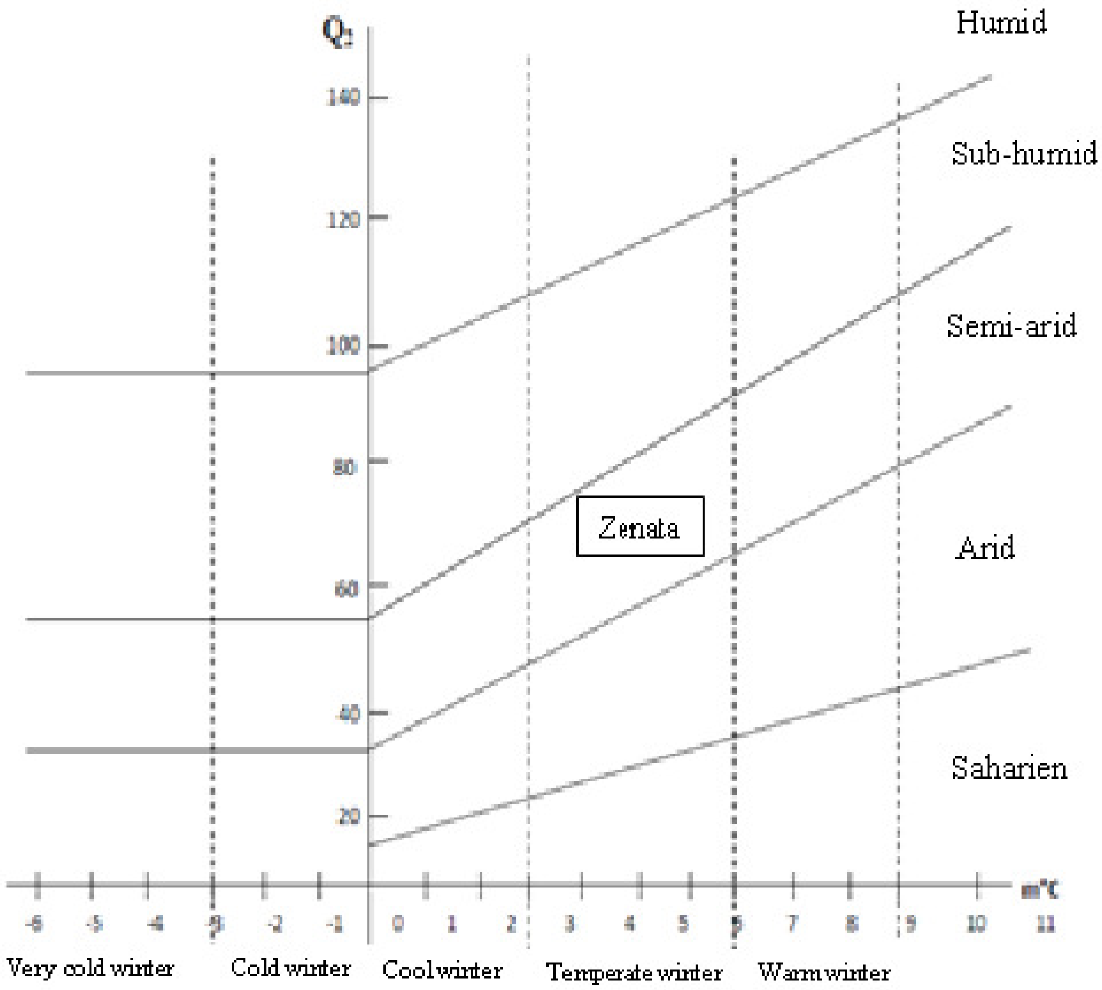


Figure I.9. Localization of the study region in Emberger's climagram.

9. Conclusion

The study region is characterized by a geomorphological variability, thus offering significant biodiversity, in addition to strong agricultural potential. Concerning the agricultural production, vegetable and cereals are the most cultivated in Tlemcen. While white meats, milk, eggs, and sheep meats are the most important in animal production.

As regards to the climatic characteristics, the region under study is part of the Mediterranean climate of the semi-arid type with moderate winter, and a dry period of 6 months. The irregularity in the distribution of rains marks this region, it is a seasonal and inter-annual irregularity. The seasonal distribution of precipitations is subject to a "WSpAS" regime. The average annual temperature is around 18.2 ° C. The coldest month corresponds to January while the warmest month coincides with August.

Chapter II

Weed flora and weed management

Literature Review

Chapter II

Weed flora and weeds management

Contents

1. Introduction

Each cultivated field is characterized by a flora commonly designated by weeds. In citrus orchards, weeds compete specially with young trees for limited resources (nutrients, water, light, and space), and harbor insects and rodents that aggress citrus trees. The competition frequently causes reductions of tree growth, leaf nitrogen level, water potential, fruit yield and fruit quality (Nasr et *al*. 2013). In citrus orchards, nitrogen uptake by weed vegetation can reach 38.6% (Takaqi et *al*., 1985). Weeds are adapted to the same edaphic and climatic conditions as cultivated plants. Therefore, practices that promote crops also promote weeds.

Reduction of soil and air temperature also occurs with weeds presence, increasing then the possibility of frost damage to citrus during cold seasons. Weeds also engender technical problems (for harvesting operations, water distribution patterns from irrigation systems emitters, disease control, and environmental conditions in the grove) (Nasr et *al*.2013).

In orchards, young trees are more sensitive to the presence of weeds, so a strict cleanliness plan should be considered until the age of five years, age at which the trees have a root system generally deep enough to no longer compete with weeds. Weeding at regular interval to reduce the competition of weeds with trees and to avoid harboring of dangerous fungal and insect pests, to which the citrus trees are highly prone, should be carried out. Some creeping plants suffocate the plant by physicochemical action, this problem occurs only for young plantations as for the adult trees, weeds form a screen which hinders photosynthesis while climbing on the tree (Kazi Tani, 2010).

In the literature, many authors have focused on weed flora since weeds are regarded as one of the main causes of loss in agricultural production. Knowledge, identification, and characterization of the weed flora is an important action to take for good management strategies.

In this part, a bibliographical overview on the weed flora in the North of Africa will be given. In addition to the weed flora of citrus orchards, as well as the methods used to manage this flora.

2. The concept of weeds

Flora and vegetation are two terms often used in agroecology. Flora represents the inventory of plant species in a region, while vegetation refers to all groups of plants from a physiognomic point of view.

The flora of the cultivated fields has often been neglected by botanists in favor of natural flora which has received more attention (Barralis (1976). Kazi Tani (2010) and Jauzein (1995) added that these environments are little prospected, nonetheless they harbor a rich and spectacular flora.

The cultivated fields are characterized by a flora which is generally designated by “weeds” in English, “mauvaises herbes” or “adventices” in French. The AFNOR in its terminological directory (second revision of December 2003) has defined “mauvaises herbes” as herbaceous or woody plants that are undesirable in the place where they are found. Whereas the Oxford Dictionary simply defines weeds as plants growing where they are not wanted.

In French, the concept of “mauvaises herbes” appeared as a pejorative qualification of a plant element not sown or not planted by the farmer and which has been replaced by the term “adventices” by botanists. The word “adventices” indicates all species introduced on cultivated plots (ethomologically *adventicium*: adding to or coming from elsewhere). The term “adventices” has also been decried by ecologists because it has only solved half of the problem, which is the "mauvaises" status that these plants carry. The word “herbes” means annual plant with a green and soft stem (annuals or grasses). However, all weeds are not necessarily grasses, they can be woody, such are frequently the apophytes. In English, the term “weeds” is also subjective but remains the term usually used to designate these plant species.

Weeds are generally considered as the most important overarching constraints in agricultural production. The weed flora evolves to form particular or more or less invasive plant groups (Abdelkrim, 1995). Generally, the problems linked with the presence of these plants are due to the evolution of the weed flora. Problematic weeds in agro-ecosystems are those that combine a high frequency with a high submergence tolerance and ecological plasticity, persistent root structures and broad-leaved species with high propagation rates (Touré et *al*., 2014).

Besides the competition of weeds with cultivated plants for light, mineral elements and water resources, several positive aspects are linked to their presence such as the maintenance of soil moisture, the protection of the soil from erosion, the enrichment of the soil by organic matter (OM), limit soil compaction, increase in biodiversity and indicator plants. Professionally managed, weeds can improve soil fertility as well as crop yields (Elong et *al*., 2018). Moreover, they represent a potential source of secondary metabolites and natural antioxidant, also they possess a good nutritive value.

3. A review on crops weed flora of North Africa

According to the African Development Bank Group and the African Union, the countries of North Africa are represented from west to east by Western Sahara, Morocco, Algeria, Tunisia, Libya, Egypt, and Sudan (Figure II.1). Herein we will give a review regarding the weed flora for all the previous cited countries except for Algeria which will be given with details next. Many studies were already conducted and published on the weed flora, from which we will cite the most relevant.

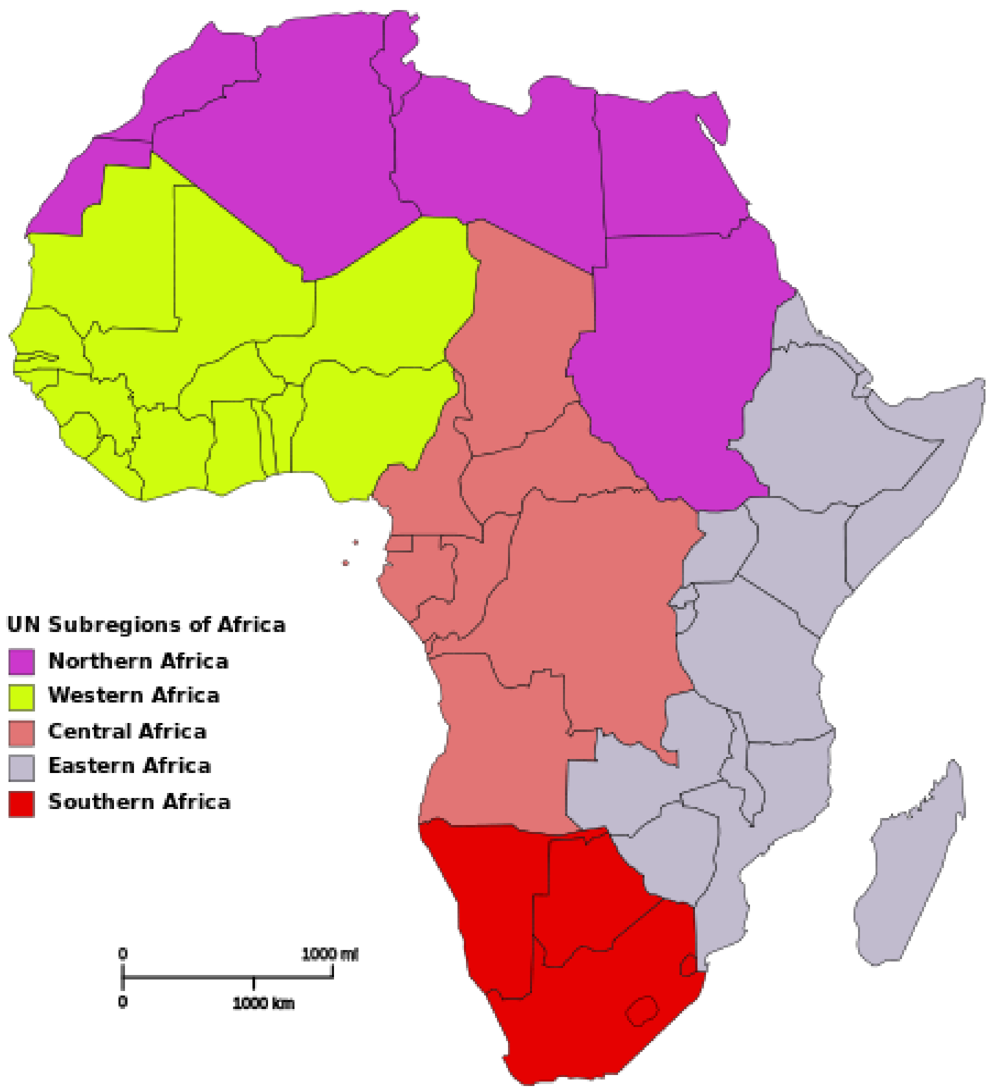


Figure II.1. United Nations Subregions of Africa arranged by the African Development Bank Group and the African Union (University of Pittsburgh, 2020). https://pitt.libguides.com/c.php?g=12378&p=65815

3.1. Crops weed flora of Western Saharan

In the literature, there was no data published on the weed flora of cultivated crops in Western Saharan.

3.2. Crops weed flora of Morocco

For morocco, we have found studies that cover west and east of Morocco as well as the important cultivated crops.

Rimani et *al*. (2019) conducted a floristic and ecological study on the weed flora in the most regions producing saffron in Morocco. From the inventory 210 weeds belonging to 34 botanical families (85.8% were dicotyledonous) were listed. Asteraceae, Poaceae, Fabaceae, Brassicaceae and Caryophyllaceae ranked first (represent jointly 56.8% of the total flora). For the ethological spectrum, annuals dominated with 147 species (70.3% of the total flora). As for the biogeographic distribution, Mediterranean taxa dominated the flora with a rate of 62.3%. The results considered twelve weeds as problematics among which *Hordeum murinum* (L.) Pers., *Malva parviflora* L., *Silene rubella* L., *Convolvulus arvensis* L., *Bromus rubens* L. and *Lolium perenne* L.

Chafik et *al*. (2014) updated the vineyards weed flora in the irrigated perimeter of Moulouya (North-East of Morocco) in comparison to that of 20 years ago. The obtained results showed important changes and a specific significative regression that affected the floristic, bio-ecologic, and agronomic aspects of this flora. Some perennials (*Cynodon dactylon* (L.) Pers., *Convolvulus arvensis* L., *Cyperus rotundus* L. and *Sorghum halepense* (L.) Pers.) advanced more in crop rows comparing with inter-rows. As well as some species (*Conyza canadensis* L., *Lolium rigidum* Gaud., *Galium aparine* L., *Raphanus raphanistrum* L.) developed a resistance to herbicides.

Continuing with the same investigation, for the floristic exploration, Asteraceae, Poaceae, Fabaceae, Brassicaceae, Apiaceae and Cayophyllaceae were the dominant families. Also, the dominance of therophytes was registered (70%). The Mediterranean species maintained their first position however cosmopolites, European and Ibero-Mauritanian taxa augmented. Nevertheless, Algerian-Moroccan endemics were in decline, chiefly in rows. Three groups of weeds were distinguished according to their noxious. In general, three perennial weeds (*Convolvulus arvensis* L., *Cynodon dactylon* (L.) Pers. and *Cyperus rotundus* L.) occupied the first ranks that were also declared problematic in similar studies (Chafik et *al*., 2014).

Another study was conducted in oriental Morocco by Chafik et *al*. (2013b) on the weed flora of agro-systems of the Moulouya perimeter. The results recorded 527 weeds belonging to 58 families. The most predominant families were Asteraceae, Poaceae, Fabaceae, Brassicaceae and Caryophylaceae (54.27%). This flora was characterized by the dominance of Dicotyledonous (83.30%), therophytes (85.08%) and the Mediterranean species (59.01%). Weeds were distinguished according to their noxiousness into five groups. From 93 noxious weeds, some geophytes (*Convolvulus arvensis* L., *Cynodon dactylon* (L.) Pers., *Cyperus rotundus* L. and *Cardaria draba* (L.) Desv.) and therophytes (*Sisymbrium irio* L., *Chenopodium album* L. *Sinapis arvensis* L. *Lolium rigidum* Gaud. and *Avena sterilis* L.) were the most abundant.

In contrast, in western Morocco, Zidane et *al*. (2010) studied the weed communities in the principal cereal areas strictly infested by the sterile oats. Three hundred twenty-four species were inventoried (from which three species are habitually cultivated, *Lens culinaris* Medikus., *Medicago sativa* L. and *Beta vulgaris* L.). The 324 inventoried species were attached to 190 genus and 47 families. Dicotyledons contained 284 species (87.65 %) while monocotyledons had 40 species (12.35 %). Six botanical families dominated the flora (Asteraceae, Fabaceae, Poaceae, Brassicaceae, Caryophyllaceae and Apiaceae). Therophytes were well presented with the rate of 80%, whereas the Mediterranean elements represented 62.3%.

The same authors classified the identified weeds in three groups basing on the partial index of noxious which revealed the existence of 27 species that could be harmful and aggressive for cereal crops. Annual Poaceae (*Avena sterilis* L., *Phalaris paradoxa* L., *Phalaris brachystachys* Link., *Scolymus maculatus* L., *Lolium multiflorum* Lam. and *Lolium rigidum* Gaud.) were the species causing serious problems in cereal crops in occidental Morocco. In their part, the most noxious perennials were represented by *Convolvulus arvensis* L., *Cynodon dactylon* (L.) Pers. and *Rumex crispus* L. (Zidane et *al*., 2010).

Tanji (2001) investigated weeds in irrigated fava bean in the Settat province (Morocco). The weed flora was composed by 191 weed species. Dicotyledons were the most represented with 169 species (88%). Moreover, annuals were dominants with 166 species (87%). The obtained results categorized weeds into three groups, parasitic weeds (*Orobanche crenata* Forssk. was the most noxious in this group), non-parasitic broadleaves and monocotyledonous species. *Sinapis arvensis* L., *Papaver rhoeas* L. and *Chrysanthemum coronarium* L. remained the three major annual broadleaf species. While the major perennial broadleaf species were *Convolvulus* *althaeoides* L. and *Convolvulus arvensis* L. As regarding monocotyledonous species, *Lolium rigidum* Gaud., *Avena sterilis* L. and *Phalaris brachystachys* were the three most important annual Poaceae, whereas *Cynodon dactylon* (L.) Pers. was the most important perennial Poaceae. The same author concluded that any program to control weed in the fava bean crop must take into consideration the predominance of the weeds cited previously.

In the literature, the ancient data found on crops weed flora in Morocco were those effectuated by Bouhache et *al*. (1994) and Taleb and Maillet (1994).

Taleb and Maillet (1994) analyzed the floristic composition of weed flora of cereals in Chaonia (Morocco). This flora registered a total of 315 species related to 45 families. Asteraceae, Fabaceae, Poaceae and Brassicaceae were the most dominant families. Dicotyledons (87%), therophytes (77%) and Meditteranean elements (64%) were predominant. The endemic species recorded a rate of 5%. They added that the dominant weeds and considered as noxious regrouped a large number of perennials (*Convolvulus althaeoides* L., *Convolvulus arvensis* L., *Silene vulgaris* (Moench) Garcke., *Arisarum vulgare* Targ.Tozz. and *Cynodon dactylon* (L.) Pers.).

Bouhache et *al*. (1994) reported that the weed flora of cereals, legumes, and sugar beet, in the Loukkos region (Northwest Morocco) was composed by 244 weed species, attached to 37 families. Three families (Asteraceae, Fabaceae and Poaceae) dominated with a rate of 41.8%. The weed flora was characterized by predominance of dicotyledonous (82.3%), annuals (82.8%) and the Mediterranean species (63.9%). The study distinguished 45 species causing considerable problems to the crops surveyed.

3.3. Crops weed flora in Tunisia

For Tunisia, we found only a few fragmentary studies that described the floristic composition of crops weed flora.

El-Waer et *al*. (2018) reported 44 weed species associated to rapeseed crop where the most abundant were *Arum italicum* Mill., *Lolium rigidum* Gaud., *Medicago ciliaris* (L.) Krock., *Glebionis coronaria* (L.) Cass. ex Spach, *Sonchus arvensis* L., *Fumaria parviflora* Lam., *Anagallis arvensis* L., *Diplotaxis erucoides* (L.) DC., *Papaver hybridum* L., *Papaver rhoeas* L., *Avena sterilis* L. and *Bupleurum lancifolium* Hornem.

Omezine and Teicheira da Silva (2012) studied the floristic composition of weed communities in relation to conventional and organic farming. The weed surveys were done in different crops, tomato in the field, tomato in greenhouses, pepper in greenhouses, potatoes in the field, artichoke in the field, lettuce in the field, cucumber in greenhouses and squash in greenhouses. The results showed that organic agricultural methods increased species richness of weeds comparing to conventional methods, confirmed by higher species richness and Shannon's diversity index. In conventionally managed fields 575 weeds, related to 19 families were inventoried (570 broad-leaved and 5 grasses). Whereas in organically managed field 823 weeds, belonged to 26 were found (818 broad-leaved and 5 grasses). Dicotyledonous and therophytes were the most represented in both conventional and organic management.

Other study was published by Omezine (2011) on the introduction of some weeds in crop fields in Tunisia, which identified eleven exotic species which could be invasive, *Eleusine indica* (L.) Gaertn, *Bidens tripartita* L., *Galinsoga parviflora* Cav., *Cyperus bulbosus* L., *Ipomaea stolonifera* (Cyr.) J.F. Gmelin, *Cuscuta australis* R. Br., *Oxalis floribunda* Lehm. ex Lindl., *Solanum elaeagnifolium* Cavi., *Salpichroa origanifolia* (Lam.) Baill., *Datura ferox* L. and *Nothoscordum gracile* (Aiton) Stearn.

3.4. Crops weed flora in Libya

Alhasi and El-Barasi (2017) investigated the weed flora of some crop fields (cucumber, pepper, tomato, lettuce, and onion crops) in Al- Oylia region, Al-Marj (Libya). The floristic inventory was composed by 41 weed species, 16 angiosperm families were found as ruderal weeds. Poaceae (11 species), Brassicaceae (6 species) and Asteraceae (4 species) were the dominant families. According to the frequency, three weeds (*Aegilopos ventricose* Tausch, *Allium roseum* L. and *Sinapis alba* L.) dominated. Globally, the results revealed dominance of the ruderal over the agrestal weeds that supposed to be due to ploughing and cleaning of agricultural soil.

Elhassi et *al*. (2017) studied the weed flora of barley and wheat crops in Al-Marj Plain (Libya). A total of 170 species attached to 117 genera and 46 families were registered. Forty-eight species were considered as noxious particularly the case of *Oxalis articulate* Savign., that caused serious losses in barley production. Therophytes presented 71.4 % in the cultivated farms and 45.5 % in neglected farms. Cryptophytes were highly present in the cultivated farm (14.3 %), while chamaephytes were mainly present in neglected farms (54.5 %).

3.5. Crops weed flora in Egypt

In Egypt, several studies were already conducted on the weed flora of many crops, such as cereal crops, Solanaceous crops (tomato and pepper), mango orchards and medicinal plants.

Alsherif (2020) investigated the weed flora of cereal crops in comparison to other crops in middle Egypt and their distribution. The inventory of all studied crops (cereals and other crops) was composed of 92 weed species belonged to 67 genera and 20 families. Regarding the chorology, cosmopolitan elements dominated the total weed flora. Egyptian clover presented the highest numbers for both weed species and genera, succeeded by wheat, while the lowest richness was recorded in Solanaceous crops (tomato and pepper). Among cereals, wheat recorded the highest richness in species and genera followed by maize crop whereas barley crops were the less rich.

The same author reported that the most frequent species in winter cereals were *Chenopodium murale* (L.) S. Fuentes, Uotila & Borsch, *Cynodon dactylon* (L.) Pers., *Convolvulus arvensis* L. and *Malva parviflora* L. As for summer cereals, *Echinochloa colona* (L.) Link and *Portulaca oleraceae* L. were the most recurrent.

Weed flora of medicinal plants also got attention by scientists. Hendawy et *al*. (2020) investigated winter weeds and their control in the medicinal plants in Egypt. Asteraceae, Poaceae, Chenopodiaceae and Fabaceae were the most representative families. The results showed that the dominant weeds in medicinal crops were *Malva parviflora* L., *Chenopodium album* L., *Medicago intertexta* (L.) Mill., *Anagallis arvensis* L., *Sonchus oleraceus* L., *Beta vulgaris* L., *Brassica kaber* L., *Cichorium pumilum* Jacq., *Melilotus indica* (L.) All., *Euphorbia geniculate* L., *Senecio desfontainei* Druce., *Emex spinosus* L., *Solanium nigrum* L., and *Conyza linifolia* Phil. Adding that the most problematic weed was *Senecio desfontainei* Druce. Narrow-leaf weeds were composed by *Lolium multiflorum* Lam., *Avena fatua* L., *Phalaris minor* Retz., and *Polypogon monspeliensis* (L.) Desf., while perennial broad-leaf and narrow-leaf weeds were *Convolvulus arvensis* L. and *Cyperus rotundus* L.

Elbous et *al*. (2018) realized a study on the weed flora in mango orchards of Ismailia Governorate (Egypt). In total 102 weed species (79 annuals, 21 perennials and 2 biennials) attached to 85 genera and 30 families were inventoried. Dicotyledons (79 species) dominated this flora comparing with monocotyledons (23 species). The most abundant families were Poaceae, Asteraceae, Brasicaceae and Fabaceae (representing together a rate of 50%). Therophytes and Mediterranean elements mainly dominated the total flora of the studied area.

Al-Sherif et *al*. (2018) studied weed flora of Fayoum (Egypt), recognized as being one of the oldest agricultural regions in the world and having the most fertile agricultural land in Egypt. A total of 175 species of vascular plants related to 124 genera and 35 families dispersed in eight habitats were recorded. Poaceae, Asteraceae and Fabaceae were the richest families whereas *Euphorbia*, *Amaranthus* and *Cyperus* were genera with the highest species number. Therophytes were the dominant ethological type. In the cited study, *Cynodon dactylon* (L.) Pers. and *Alhagi graecorum* Boise. were reported as the species present and recorded at all habitats. The highest species number was recorded in old, cultivated lands, followed by orchards. In contrast, lowest species number was reached in roadsides and wasteland habitats.

Salama et *al*. (2016) studied the weed flora of three common crops (wheat as a winter crop, millet as summer crop and alfa-alfa as a perennial crop) of desert reclaimed arable lands in southern Egypt. The floristic composition was composed of 169 species (105 annuals and 64 perennials) related to 121 genera and 39 families. Poaceae, Asteraceae, Fabaceae, Brassicaceae, Chenopodiaceae and Euphorbiaceae were the most species-rich families while the best represented life form were therophytes. With a wide ecological range of distribution, twenty-six species were categorized as dominants, they incorporated the most common weeds of arable lands in Egypt such as *Sonchus oleraceus* L., *Chenopodium murale* (L.) S. Fuentes, Uotila & Borsch, *Convolvulus arvensis* L., *Melilotus indicus* (L.) All., *Cynodon dactylon* (L.) Pers., and *Portulaca oleracea* L.

Serag et *al*. (2015) established an ecological study on weed flora in most named habitat types of new Damietta (orchards, sandy fertile cultivated lands, banks of irrigation canals, reclaimed lands, and waste lands). The weed flora contained 60 weeds related to 23 families. Poaceae (13 species) and Asteraceae (10 species) were the most dominant families and annuals were the most abundant life form. Mediterranean weeds extending into the Euro-Siberian land reached higher occurrence in the study area comparing to weeds with Saharo-Indian extension. The same authors reported that from the 60 weeds inventoried, 22 species are used to treat common diseases, 24 species are used as food, 19 species used to feed animals and 34 species used in industry, ornamental and furniture in the studied areas.

Abd El-Ghani et *al*. (2013) studied the weed flora in the reclaimed lands along the northern sector of the Nile Valley (Egypt). A total of 150 species distributed within 33 families were recorded. Poaceae (31), Asteraceae (23), Brassicaceae (13), Chenopodiaceae (12) and Fabaceae (12) were the most represented families. For the chorological analysis, the widely distributed species belonged to cosmopolitan, palaeotropical and pantropical chorotypes that represented about 39.3% of the total flora. Mediterranean species *sensu stricto* were less represented, whereas biregional and triregional Mediterranean chorotypes presented 28%. Saharo-Arabian elements constituted 32%. As for ubiquitous species (with extensive amplitude) were represented by *Cynodon dactylon* (L.) Pers. and *Sonchus oleraceus* L.

Ancient studies were also found, Fahmy (1997) evaluated the weed flora of Egypt from Predynastic to Graeco-Roman times. The study recorded 112 weeds from 61 archaeological sites dating from Predynastic times (4500 B.C.) up to the Graeco-Roman period (A.D. 395). The 112 species include 24 taxa from Predynastic Hierakonpolis (3800-3500 B.C.) identified for the first time. The highest number of species (63) was recorded from the Pharaonic period. The Predynastic era was represented by 46 species and the Graeco-Roman period by 34. Floristic analysis showed that 57 species were introduced in crop fields from the Middle East and 40 belonged to the native vegetation of the Nile valley.

3.6. Crops weed flora in Sudan

Abdelmageed et *al*. (2020) realized an eco-taxonomic study on weeds of Um Algura Area-Western Gezira State (Sudan). The inventory contained 82 species related to 30 families. Poaceae was the most dominant family with 19 genera and 22 species. The most important terrestrial species were *Ipomoea kordofan* L., *Xanthium brasilicum* L., *Bidens repens* D. Don, *Cynodon dactylon* (L.) Pers., *Cyperus Spp*, *Acacia nilotica* (L.) Del., *Cenchrus biflorus* Roxb. and *Setaria verticillate* (L.) P.Beauv. The most dominant emergent weeds included *Typhaan gustata* Pers., *Typha latifolia* L. and *Ipomoea kordofan* L. While the most important floating weeds were *Eichhornia crassipes* Mart., *Salvinia molesta* D. Mitch. and *Pistia stratiotes* L. In the mentioned study new species were introduced *Xanthium brasilicum* Vell., Eragrostis namaquensis Hochst. Ex Steud., *Ruellia Gondwana* L. and *Peristrophe bicalyculata* Nees.

Eltayep Ahmed (2016) conducted a study on weeds associated with potato plant in Khartoum area (Sudan). *Cyperus rotundus* L., *Echchino chloacolona* (L.) Link, *Gynandropsis gynandra* L., *Cynodon dactylon* (L.) Pers., *Sorghum* and *Portulaca oleracea* L. occurred at high relative abundance.

Abdel Aziz Mohamed et *al*. (2015) reported that the weed flora associated to garlic (*Allium sativum* L.) in Dongola (northern State, Sudan) consisted of grassy and broad-leaved weeds. The dominant weed species recorded were *Tribulus terrestris* L., *Malva palviflora* L., *Eruca sativa* Mill., *Echium rauwolfia* Del., *Amaranthus viridis* L., *Chenopodium album* L., *Euphorbia aegyptiaca* Boiss. *Cynodon dactylon* (L.) Pers., *Cyperus rotundus* L., *Echinochloa colona* (L.) Link., *Trigonella hamosa* L., *Convolvulus arvensis* L. and *Rhynchosia memnonia* (Del.) DC.

Abdel Aziz Mohamed (2012) realized a weed survey on wheat in the northern state of Sudan. The weed flora was composed by 29 species belonged to 15 families. Poaceae, Euphorbiaceae, Solanaceae and Cruciferae were the most dominant families. Dicotyledonous dominated the flora. Weeds that occurred at high relative abundance were *Sinapis arvensis* L., *Cynodon dactylon* (L.) Pers., *Malva palviflora* L., *Convolvulus arvensis* L., *Chenopodium album* L. and *Sorghum arundinaceum* (Desv.) Stapf.

Idris *et al*. (2011) reported in their study that the dominant weed species of common bean crop in Sudan were *Beta vulgaris* L., *Sinapis arvensis* L., *Ipomoea sinensis* (Desr.) Choisy, *Malva parviflora* L., *Portulaca oleracea* L., *Chrozophora plicata* (Vahl.) A. Juss. Ex Spreng and *Brachiaria eruciformis* (Sm) Grieseb.

4. A review on crops weed flora in Algeria

The flora of Algeria counts about 3139 species, where 653 species are endemic, represented by approximatively 150 families. The endemic flora represents 12.6% of the total flora from which more than 25% of the species are related with cultivated crops (Quézel and Santa, 1962-1963).

In Algeria, several studies were achieved on the floristic composition, distribution, and changes of the weed flora for some cultivated crops. Across the country, studies were carried out in the Northeast of the country (Melakhessou et *al*., 2020, Hannachi, 2019, Karkour and Fenni, 2016, Diab and Deghiche, 2013, Hannachi and Fenni, 2013, Fenni, 1993), in the North central of the country (Abdelkrim, 2004), in the Northwest (Fertout-Mouri, 2018, Hanitet, 2012, Kazi Tani et *al*., 2010) and, in the South of Algeria (Eddoud et *al*., 2018, Sayed et *al*., 2014).

In the Northeast of Algeria, Melakhessou et *al*. (2020) studied the weed flora of the Aurès in the North-East of Algeria. The results revealed the presence of 77 weeds (basically dicotyledonous and monocotyledonous), belonged to 23 botanical families. Dicotyledons with a rate of 82% (63 species) and therophytes with 69% (53 species) were the majoritarian. Asteraceae, Poaceae, Apiaceae and Brassicaceae were the most dominant families (58% of total flora). Also, the results identified 36 species that can be potentially noxious towards wheat crop where seven weed species (*Avena sterilis* L., *Malva sylvestris* L., *Lolium multiflorum* Lam., *Hordeum murinum* (L.) Pers., *Bromus rubens* L., *Anacyclus clavatus* (Desf.) Pers. and *Lolium rigidum* Gaud.) were considered as the most problematic in the Aurès zone.

Hannachi and Fenni (2013) performed a floristic and ecological study on different weed crops in the region of Batna. The weed flora of this area contained 120 weed species. Broadleaf weeds were dominant with a rate of 81.66% (98 species), monocotyledonous include 22 species (18.33%). The identified species belong to 95 genera and 30 botanical families. The biological type revealed the dominance of annuals 69.16% (83 species), perennials present 19.16% (23 species) and biennials 11.66% (14 species). As regards to the biogeographic origin, mono-regionals were the most present (78 species) while pluri-regionals were represented by 25 species. The species of hot regions counted 11 and the cosmopolites with 6 species.

In the same investigation, the authors classified weeds in three classes according to their frequency. Class IV (60 - 80 % of frequency), which was represented only by one species (*Veronica opaca* Fries), class II (20 - 40 % of frequency) that regrouped ten species where *Chrysanthemum segetum* L., *Medicago arabica* (L.) Hudson and *Sinapis arvensis* L. occupied the first ranks and class I (- 20 % of frequency) which contained the rest of species (109 species representing 90.80 % of the total flora), in this class three Poaceae rank first (*Cynodon dactylon* (L.) Pers., *Bromus madritens*is L. and *Lolium multiflorum* Lam.). As according to the relation between relative frequency of weeds and their average abondance-dominance, they identified five types of species which reflected their potential noxious. In the cited study *Sinapis arvensis* L. and *Malva sylvestris* L. were the most abundant weeds (Hannachi and Fenni, 2013).

Karkour and Fenni (2016) reported the floristic composition of the weed flora in cultivated lands in semi-arid region (Sétif, Algeria). A total of 178 weed species, attached to 132 genera and 35 botanical families were listed. Asteraceae, Poaceae and Brassicaceae were the most representative families. Dicotyledonous (84, 27%) and therophytes (75 %) dominated. In addition, the results allowed categorizing four groups of weeds according to their frequency. Three species (*Bromus rigidus* Roth, *Vicia sativa* L. and *Papaver rhoeas* L.) had a frequency between 60 and 80% (class IV) which turned out to be the most harmful species throughout the total crops.

In another study, Diab and Deghiche (2013) mentioned that the weed flora in the oasis of Ziban (Biskra, Algeria) contained 281 species, related to 47 botanical families with the dominance of broadleaf weeds (86%).

Fenni (1993) investigated the weed flora of cereal crops in Sétif (Northwest of Algeria). The floristic inventory was composed by 215 species, belonging to 143 genera and 31 families. Asteraceae (49 species), Fabaceae (23 species), Poaceae (21 species) and Brassicaceae (16 species) were the most common families. Also, therophytes dominated with a rate of 73% of the total flora. The results showed that the most abundant weed species were *Sinapis arvensis* L., *Vicia sativa* L., *Avena sterilis* L., *Papaver rhoeas* L., *Vaccaria hispanica* (Mill.) Rauschert, *Fumaria parviflora* Lam., *Bifora testiculata* (L.) Spreng., *Bromus rigidus* Roth and *Veronica hederaefolia* L.

In the central northern of Algeria, Abdelkrim (2004) conducted a study in the surroundings of Algiers (area characterized by a gradient from a humid to a semi-arid Mediterranean climate) where, the crop weed communities were determined. For fallow land in the sub-humid bioclimate and on heavy soils, the spontaneous vegetation was categorized into the *Ammio visnagae-Capnophylletum peregrini*, the *Senecio delphinifoliae-Fedietum cornucopiae ass. nov*., and the *Tetragonologo purpureae-Cerinthetum majoris*. Noting that All the previous communities belong to the alliance *Convolvulion tricoloris* (*Chrysanthemetalia segetum* L.). The weed vegetation in winter cereals was distinguished according to the climatic conditions: The *Bunio incrassatae-Galietum tricornis* colonizes arable land under subhumid, the *Calendulo algeriensis-Psychinetum stylosae* under semi-arid conditions. In the last association occur transgressive from steppe vegetation (*Thero-Brachypodietalia, Lygeo-Stipetea*). While perennial cultures such as vineyards and other root cultures were characterized by the *Ormeni-Silenetum fuscatae* (*Oxalido cernuae-Fumarion capreolatae, Chenopodietalia, Stellarietea mediae*).

In the Northwest of Algeria, Fertout-Mouri (2018) realized a phytoecological study of the weed flora of cereal agrosystems of the Tessala region (Northwestern Algeria). In total, 83 weed species, attached to 24 families and 57 genera were identified. Dicotyledons (86.75%) dominated clearly the flora listed. Therophytes (75.90%) and Mediterranean elements (40.97%) were the most representative.

Hanitet (2012) also established the different groups of weeds in crops in the Oran region. In this study different weed groups were divided according to their association to the different cultures. The identified weed flora included 80 species divided into 26 families and 65 genera. Asteraceae, Poaceae, Brassicaceae, and Fabaceae were the most important families.

Kazi Tani et *al*. (2010) investigated the floristic aspects of weed flora of the Oranian phytogeographic territory (Northwest Algeria). The weed flora contained 425 species belonging to 51 botanical families and 217 genera. The most dominant families were Asteraceae, Fabaceae and Poaceae (39%, jointly). Also, the weed flora was characterized by the dominance of dicotyledonous (354 species), annuals (76%) and Mediterranean floristic elements (57.64 %).

Kazi Tani (2010) highlighted the most problematic species in each crop. On the whole, it is particularly about *Convolvulus arvensis* L., which is ranked among the fifteen most problematic global "weeds" of spring and summer crops, as well as *Sinapis alba* L., a rather ruderal and xerophilic species which has become, these years, more infesting because of the prolonged drought of the last three decades. These two species are developed both in root crops and in field crops. Cereals still faces the problem of managing infesting grasses due to the lack of a diversified cropping system (diversification of herbicides) and the absence of effective mechanical control (precision agriculture).

Moving to the Southeast of Algeria, Eddoud et *al*. (2018) published an article on the changes in weed species composition in irrigated agriculture realized on weed monitoring over the 20 years which revealed that weed composition changed overtime, chiefly in the palm plantations and the cereal plots. As for the floristic inventory, 162 species belonging to 40 families were registered. The dominant families were Poaceae, Asteraceae, Brassicaceae and Amaranthaceae (45.1% of the total species). The weed communities were composed by species from various origins (biregional, triregional, and multiregional origins). Saharo-Arabian and Mediterranean species were most represented. As for the most common weeds in the study region, eight therophytes were mentioned, *Chenopodium murale* (L.) S. Fuentes, Uotila & Borsch, *Lolium multiflorum* Lam., *Melilotus indicus* (L.) All., *Anagallis arvensis* L., *Sonchus oleraceus* L., *Setaria verticillate* (L.) P.Beauv., *Polypogon monspeliensis* (L.) Desf., and *Erigeron canadensis* L.

Sayed et *al*. (2014) reported that the floristic inventory of the flora in cereal areas of the Ouargla region (Hassi Ben Abdellah) was composed by 52 weed species spread over 14 botanical families and 43 genera. The results obtained highlighted the dominance of Asteraceae (19.23%), Poaceae (17.30%) and Brassicaceae (13.46%). Therophytes were the most present. Finally, the introduced species predominated over spontaneous ones.

5. A literature review on citrus weed flora

In the literature, several studies were realized on crops weed flora but also some studies were conducted across the globe, particularly on the weed flora of citrus orchards.

Hannachi (2019) characterized the grouping and distribution of groups of citrus weeds in the Skikda region (Algeria). The results distinguished two main groups. Group 1, which contained several species (*Oxalis latifolia* kunt., *Bromus tectorium* L., *Coleostephus myconis* L., *Coronilla scorpioide* (L.) Koch, *Chinopodium ficifolium* JE., *Silene gallica* L., *Aegilops geniculata* Roth, *Geranium columbinum* L, *Cirisum arvense* L, *Festuca arunadinacea* Schreb., *Torilis nodosa* (L.)

Gaertn., *Chenopodium glaucum* L.) grouped according to agronomic, hydrological, climatic, and geomorphological parameters. And group 2, that consisted of species (*Chrysosplenium segetum* L., *Cheopodium mural* L., *Gladiolus italicus* Mill., *Vicia peregrina* L., *Vicia bithynica* (L), *Papaver rhoeas* L., *Medicago arabica* L., *Coleostephus myconis* L., *Borago officinale* L., *Verbena officinalis* L., and *Knuntia arvensis* (L.) Coult.) encountered on the mountainous area and hills.

In another study conducted in Kingdom of Saudi Arabia, 36 of weed species belong to 20 families were found (Al-Qahtani, 2019). In the same region, Gomaa (2017) accomplished a similar study where a total of 33 weed species were identified in citrus orchards, belonging to 15 families and 30 genera. The main families were Asteraceae and Poaceae, followed by Fabaceae, Chenopodiaceae and Polygonaceae. Whereas the most abundant life forms were therophytes and geophytes (Gomaa, 2017). The same author classified the weed vegetation of the study area into four vegetation groups dominated by *Plantago lagopus* L., *Cynodon dactylon* (L.) Pers., *Imperata cylindrica* (L.) P.Beauv., and *Convolvulus arvensis* L.

According to the study carried out in Turkey by Onen et *al*. (2018), sixty-eight weed species belonging to 30 families were inventoried. Moreover, they recorded a higher number of weed species in spring season compared with autumn. While therophytes were the most dominant life forms.

In Northwestern India, another study was conducted by Shweta et *al*. (2018) in sweet orange orchards, in which the results revealed the main presence of five weeds (*Cyperus rotundus* L., *Cynodon dactylon* (L.) Pers., *Chenopodium album* L., *Amaranthus spp*., and *Parthanium hyterophorus* L.). Broad leaf weeds were the most representative (55.29%).

In another study, Cristaudo et *al*. (2015) mentioned that weed flora of citrus grove in Italy was composed of 63 taxa belonging to 25 botanical families, from which 42% are included in the families Poaceae, Asteraceae and Brassicaceae. In addition, therophytes and species with a wide distribution were dominant which was related to the high degree of artificiality occurred by human pressure.

Le Bellec (2015) mentioned that the inventories have identified 154 plant species that compose the weed flora of the citrus orchards studied (Reunion Island). Seven species (*Oxalis corniculata* L., *Pterocypsela indica* (L.) C. Shih, *Bidens pilosa* L., *Conyza sumatrensis* (Retz.) E. Walker, *Lantana camara* L., *Ageratum conyzoides* L. and *Cynodon dactylon* (L.) Pers.) were declared as the most frequent.

In the Northeastern Morocco, Chafic et *al*. (2013a) reported in their study that the citrus fruits weed flora was composed by a total of 331 species belonging to 50 botanical families. Recording that Asteraceae, Poaceae, Fabaceae, Brassicaceae and Caryophyllaceae were the most dominant families (more than 50% of species). Also, they mentioned that the most abundant life forms were dicotyledonous with a rate of 80.9%, annuals (69.25%) and the elements with Mediterranean repartition (66.77%). Moreover, they précised that he most common species were *Cynodon dactylon* (L.) Pers., *Cyperus rotundus* L. and *Convolvulus arvensis* L. and *Oxalis pes-caprae* L. which are geophytes and that required a special control.

Mashaly and Awad (2013) studied the weed flora of orchards in Egypt reporting a total of 169 weed species in all the studied orchards where the highest number of weeds (115 species) was found in citrus orchards thus representing 68% of total species.

In Iran, another study was also realized by Nasr et *al*. (2013) which highlighted the presence of 130 weed species belonging to 42 families. Among these species, Poaceae, Asteraceae and Brassicaceae were the most dominant. The flora registered the abundance of perennial and biennial weeds than annuals. Additionally, broadleaf weeds were relatively abundant than the grass ones. In the studied citrus orchard, *Cynodon dactylon* (L.) Pers., *Digitaria sanguinalis* (L.) Scop., *Setaria glauca (L.) Beauv.*, *Dactylis glomerata* L. and *Acalypha australis* L. were found the most abundant.

In addition, Singh and Rana (n.d.) reported that the predominant weeds of citrus fields were *Cynodon dactylon* (L.) Pers., *Cyperus* spp., *Digitaria marginate* (Retz.) Koel, *Eleusine indica* (L.) Gaertn, *Setaria* spp., *Imperata cylindra* (L.) P.Beauv., *Amaranthus caturus* Roxb., *Biden Pilosa* L., *Lagasca mollis* Cav., *Acanthospermum hispidium* D.C., *Euphorbia* Spp., *Borreria articularis* (Linn. f.) F. N. Williams., and *Evolulus alsinoides* (Linn.) Linn.

Hanitet (2012) identified 45 weed species frequent in citrus orchards. The characteristic species of this culture were *Fumaria parviflora* Lam., *Inula viscosa* L., and *Mililotus sulcatus* Desf. that have not been found in the other cultures. The installation of these species was favored by the humidity of soil due to the regular irrigation. *Cynodon dactylon* (L.) Pers. and *Hordeum murinum* (L.) Pers. were also present in citrus orchards.

Alongside with the other studies, flora surveys were conducted by Antunes et *al*. (2005), in lemon orchards in the West of Portugal during two periods (spring-summer and autumn-winter). The results obtained identified 62 species with relative frequency higher than 15% (Forty-four species in autumn-winter while Forty-two species spring-summer). The Asteraceae, Poaceae and Fabaceae were the most representative families in autumn-winter, whereas, in spring-summer, Asteraceae, Poaceae, Polygonaceae and Chenopodiaceae occupied the first ranks.

In the neighboring country, a similar study was also carried out by Mas and Verdú (2005) in the western Mediterranean region (Southwest of Catalonia, Spain), on the weed flora in mandarin crops according to soil management at inter-rows. In total, 112 species belonging to 30 botanical families were cataloged, from which, Poaceae and Asteraceae were the most dominant, contributing together with 34% of the total species. Additionally, the results showed that the number of species present was greater in the plots whose streets or inter-rows were mowed, compared to those that suffered applications of chemical herbicides. Thus, confirming that the method of weed management has a significant effect on the floristic composition of the weed flora of orchards and even its distribution inside the orchard.

6. Weed management in citrus orchards

Weed control is a major component in citrus groves alongside with the other crops and has been achieved using different methods, including hand weeding, mechanical and chemical methods, mulching, and cover cropping/intercropping. In addition to chemical and mechanical weeding, other methods were discussed in the literature. Nonetheless, the identification of weed species is necessary and primordial before starting any weed management program (Nirmaljit and Rattanpal, 2017).

Emre Kitiş et *al*. (2017) investigated mulch textile comparing with conventional polyethylene mulch nylon and mechanical and chemical control in citrus orchards (mandarin orchards). The experiment was conducted in a three-year-period using two different thickness of polyethylene mulch, three different thickness of mulch textiles, mowing and herbicide (glyphosate) in recently established mandarin orchard. The results obtained showed that mulch textiles-controlled weeds at 100% than weedy control, followed by polyethylene mulch (99.6%), then chemical control (88.4%) and finally mowing (23.4%). Furthermore, soil temperature and moisture were preserved by mulch applications.

To replace chemical and mechanical weeding, cover plants can be facilely introduced in orchards and could be effective in weed control, in addition to other functions. Based on this supposition, Jannoyer et *al*. (2011) elaborated a specific approach for the selection of adequate cover plants in single crop orchards (case of citrus orchards) to manage weeds and furnish additional ecological services. The experiment was conducted in two regions (Guadeloupe and Martinique, France). Optimum cover crop functional groups were determined according to agrosystem and associated goals. In Guadeloupe, a participatory approach led initially to the selection of *Neonotonia wightii* (Wight & Arn.) J.A. Lackey, a species attached to Fabaceae and distinguished by high auxiliaries hosting services. While in Martinique, the necessity for a high covering index related with a low production led to the selection of *Urochloa mozambicensis* (Hack.) Dandy and three *Paspalum* species (grasses). They concluded that the concept of an optimal single cover plant remained complicated to attain and the performance of a multi-specific cover system is commonly required to get the desired efficiency.

Verdú and Mas (2007) discussed the mulching as an alternative technique for weed management in mandarin orchard tree rows. The objective of this study was to evaluate the effect of mulching applied to mandarin tree rows for weed control in comparison with chemical weed control (with glyphosate). Three mulches were tested (rice straw, almond husk, and black geotextile). The results showed that black geotextile and almond husk controlled the presence of weeds as well as or better than the applications of glyphosate at least during the first year after their introduction. Also, they cited that there were no significant differences the mean weed cover of black geotextile, almond husk, and herbicide plots, thus indicating that mulching is one weed management strategy in mandarin orchards that presents other benefits (soil protection and avoiding herbicide pollution) in terms of sustainable agriculture.

Sharma and Singh (2006) exanimated the bio-efficacity of Gramoxone Inteon (Paraquat) formulation in comparison with Gramoxone Max and Touchdown Hitech (Glyphosate) on weeds in citrus groves. Treatments were applied alone (Gramoxone Inteon 2SL and Gramoxone Max 3SL at 840 and 1120 g a.i./ha, Touchdown at 880 g a.i./ha) and as a tank mixture with Solicam (2688 g a.i./ha). The non-ionic surfactant X-77 TT (0.25%) was tank added to all treatments in order to increase spread. Gramoxone Inteon and Max recorded approximatively complete control of grasses and broadleaf even at low concentration. The maximum control was reached one week after treatment whereas after four weeks, control of all weeds was similar. In brief, the results of this study showed that both formulations of Gramoxone had equally better weed control in citrus groves than Touchdown. Adding that some broadleaf developed a slight tolerance to Touchdown. More clearly, weed control with paraquat was better than with glyphosate.

Protopapadakis (1985) studied the changes in the weed flora of citrus orchards in relation to the widespread use of glyphosate (chemical weeding) by comparison with traditional weed control systems. The results obtained after ten years of application revealed that weed control methods affected the flora that exists in citrus orchards. Mainly, the use of glyphosate allowed colonization by new species, which are resistant to glyphosate (*Parietaria judaica* L., *Equisetum telmateia* Ehrh, *Equisetum ramosissimum* Desf, *Hedera helix* L., etc.).

7. Conclusion

This bibliographic section allowed us to accomplish a brief overview on the floristic composition of the weed flora of North Africa on the large scale, Algeria in the second place and the flora of citrus groves on the small scale. As well as the weed management in citrus orchards.

According to the data reported in the literature, the weed flora of North Africa is rich in species, independent of the type of culture. This flora is clearly dominated by dicotyledons. For the most widespread ethological type, the therophytes continually occupy the first positions. While the Mediterranean taxa remain the most representative overall. Asteraceae, Poaceae, Fabaceae and Brassicaceae were the most predominant botanical families.

Basing on the global studies realized on the weed flora of crops in North Africa, the most abundant weeds are *Cynodon dactylon* (L.) Pers., *Convolvulus arvensis* L., *Cyperus rotundus* L., *Malva parviflora* L., *Avena sterilis* L., *Chenopodium album* L., *Sinapis arvensis* L., *Lolium rigidum* Gaud., *Papaver rhoeas* L., *Convolvulus althaeoides* L., *Portulaca oleraceae* L., *Anagallis arvensis* L., *Sonchus oleraceus* L. and *Beta vulgaris* L.

The weed flora of Algeria is part of the North African flora, thus follows the same characters previously mentioned, which confirms its belonging to this set. The most common weed species between cultures at national scale are *Avena sterilis* L., *Malva sylvestris* L., *Lolium multiflorum* Lam., *Sinapis alba* L., *Sinapis arvensis* L., *Vicia sativa* L., *Papaver rhoeas* L. and *Bromus rigidus* Roth.

The citrus weed flora also was characterized by the dominance of dicotyledonous, therophytes and Mediterranean elements. Moreover, Asteraceae, Poaceae, Fabaceae and Brassicaceae represent the most present botanical families. *Cynodon dactylon* (L.) Pers., Cyperus *rotundus* L. and *Convolvulus arvensis* L. were reported as the most problematic weed species for citrus orchards.

In the literature some researchers discussed the mulching and cover plants as alternative methods to chemical and mechanical weeding in citrus groves, which has been shown to be effective for better weed control, thus avoiding the negative sides of the mechanical and chemical methods used to manage weeds.

Weeds compete with the cultivated plants for natural resources, causing losses in the yield. However, they do not present only negative effects, weeds may be useful for food, feed, medicine, fuel, or insulation, thus deserving more attention and divers' investigations on their potential uses. In the current thesis, the possible uses of weeds as a source of secondary metabolites and natural antioxidants, as well as a nutritional potential for animal feeding will be discussed.

Chapter III

Secondary metabolites and antioxidant activity

Literature Review

Chapter III

Secondary metabolites and antioxidant activity

Contents

1. Introduction

Plants produce a wide range of biochemicals with various structures such as primary and secondary metabolites. The primary metabolites (carbohydrates, proteins, lipids, and nucleic acids) are products derived directly from photoassimilates, which participate in the structure of the plant cell as well as in its basic functioning (Hopkins, 2003). Moreover, plants produce a large number of compounds which do not come directly from photosynthesis but result from subsequent chemical reactions; this group of compounds is named secondary metabolites (Kossel, 1891), these compounds intervene in the growth and development of plants but are not compulsory for the plant to survive (Pagare et *al*., 2015). Then, by definition, the term “secondary metabolite” designates any substance present in an organism, and which does not directly participate in the basic processes of the living cell (Kossel, 1891). But the difference between the primary and secondary metabolites remains ambiguous because some intermediaries of primary metabolism can overlap with those of secondary metabolism (Verpoorte et *al*., 2000).

These metabolites are distributed differently between botanical families. In general, there are over 2,140,000 secondary metabolites known. Plants remain the main source (Bérdy, 2005) of these compounds (80%), along with other sources (bacteria, fungi, algae, etc.). Microorganisms also have a significant capacity to provide several antibiotics and other drugs against bacterial and fungal infections, tumor, and cardiac diseases (Gokulan et *al*., 2014).

Secondary metabolites are produced in large numbers, (more than 200,000 defined structures) but are produced in small quantities with a limited distribution in the plant organism. They play different roles, including that of defense against external aggression and ecological adaptation (Jain et *al*., 2019, Pagare et *al*., 2015, Hartmann, 2007). In addition to other functions such as, fuel, structure, signaling, stimulatory and inhibitory effects on enzymes, cofactor to enzymes and interaction with other microorganisms (Jain et *al*, 2019). They are too agents which attractant pollinators and seed dispersing animals (Pagare et *al*., 2015). Also, these compounds were characterized for their capacity to affect the growth of other plants by the mechanism called allelopathy (Siqueira et *al*., 1991, Waller, 1989) and for their role as intermediaries in plant-microbe symbioses (Pagare et *al*., 2015).

In many cases, plant secondary metabolites are fundamental and present in the biological active forms of healthy plants, although, others are activated unreason to tissue damage or pathogen aggresses and appear as inactive precursors (Jain et *al*., 2018).

The secondary metabolites are recognized to have medicinal properties and nowadays it is possible to identify and separate with precision the molecules responsible for the different biological activities. Whereas in the past man has used plants in medical treatments and to heal their animals without knowing which are the molecules responsible of these therapeutic properties. Different biological activities (antioxidant, antimicrobial, antitumor, anti-inflammatory, etc.) are attributed to these molecules. The phytochemicals have antimicrobial activities with various mechanism of bacteria inhibition (Compean and Ynalvez, 2014).

Recently, the secondary metabolites have been the subject of several studies with the objective of finding natural antioxidants from plants in order to eliminate the use of synthetic antioxidants which presented a lot of side effects.

In this chapter, the classification of plant secondary metabolites, their biosynthetic pathways and their biological activities will be presented as well as their relationship with oxidative stress and antioxidant activity. As well as some important methods to evaluate the *in vitro* antioxidant activity will be described. In addition, a literature review on the nineteen studied weed species will be provided.

2. Biosynthesis of secondary metabolites

Secondary metabolites result from three important biosynthesis pathways (Figure III.1): the shikimate, mevalonate, and the pyruvate pathways (Verpoorte and Alfermann, 2000, Taiz and Zeiger, 1998). The most of secondary metabolites are derived from amino acids or acetyl Co-A (Ahmed et *al*., 2017).

Both primary and secondary metabolites derived from biosynthesis pathways. Furthermore, the primary and secondary metabolisms are integrated (Figure III.2), and as mentioned previously some intermediaries of primary metabolism can overlap with those of secondary metabolism (Verpoorte et *al*., 2000).

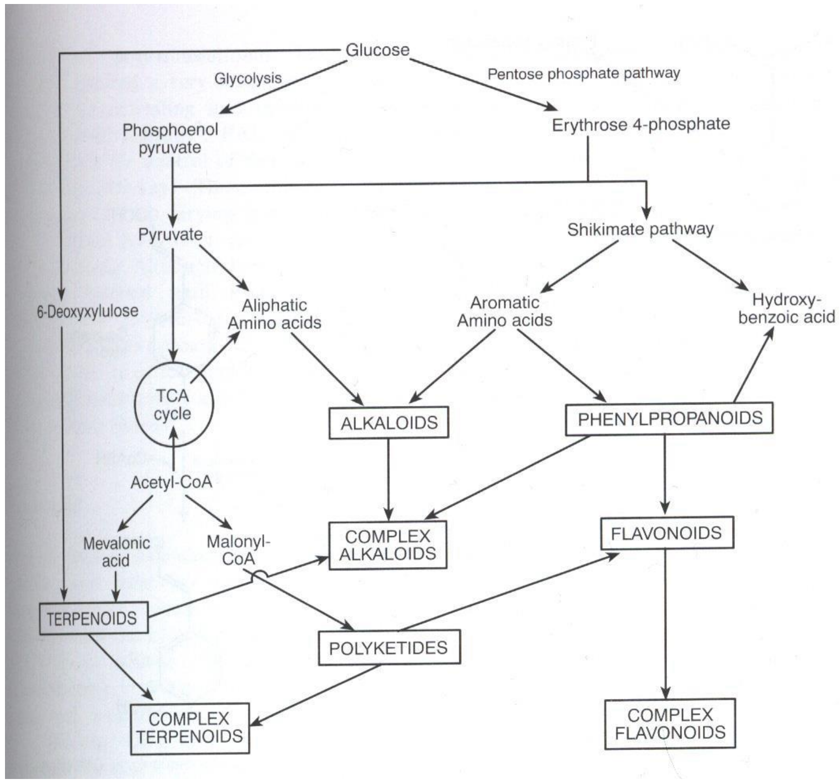


Figure III.1. Overview of biosynthetic pathway of secondary metabolites (Ghabru and Rana, 2019).

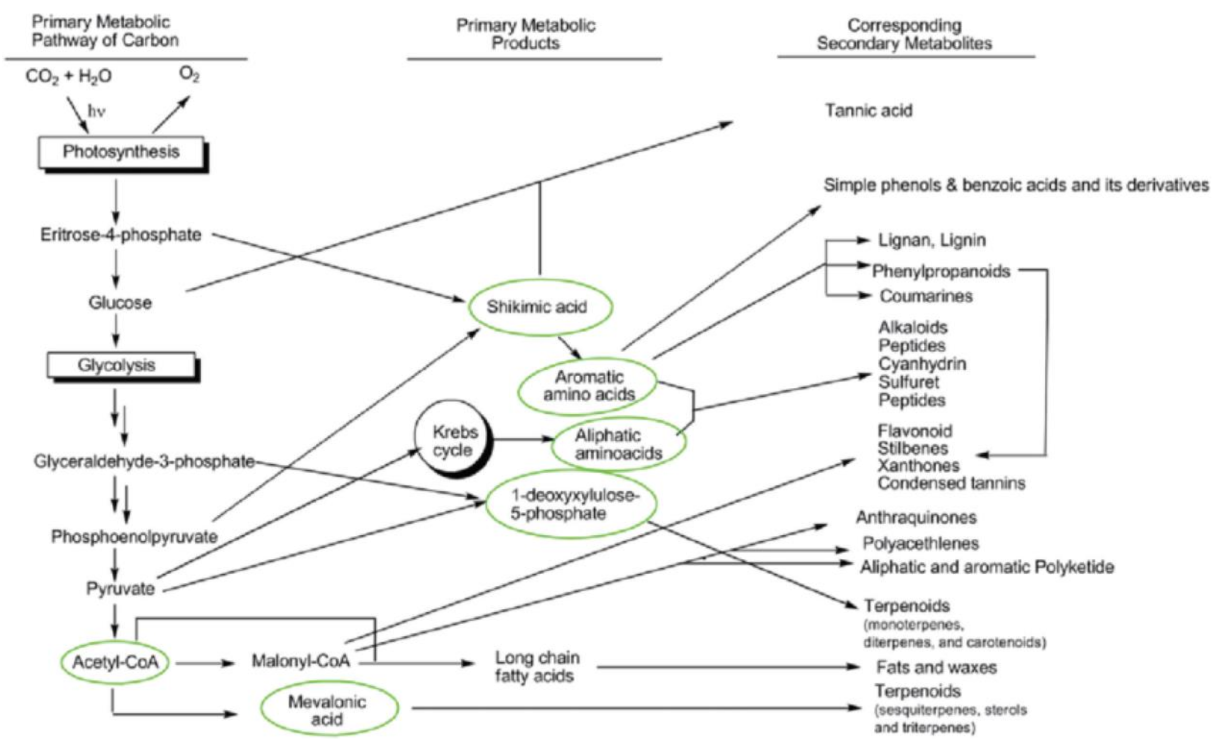


Figure III.2. Interaction Integration between primary and secondary metabolisms (Verpoorte et *al*., 2000).

3. Plant secondary metabolites classification

In the literature, different classifications of plant secondary metabolites have been reported by many scientists. Verpoorte (1998) and Kabera et *al*., 2014, mentioned that secondary metabolites are divided in three main classes: terpenoids, alkaloids and phenolics. While other authors separated them in four main classes: terpenoids, steroids, alkaloids and phenolics (Jain et *al*., 2019, Ghabru and Rana, 2019, Ahmed et *al*., 2017, Okunade et *al*., 2004).

According to Pagare et *al*. (2015), plants secondary metabolites are distinguished in three groups: Terpenes, Phenolics, N (Nitrogen) and S (Sulphur) containing compounds. Whereas five principal classes have been reported by Durairaj Thirumurugan (2018) and McMurry (2015), which are terpenoids and steroids, fatty acid-derived substances and polyketides, alkaloids, non-ribosomal polypeptides, and at the end enzyme cofactors.

In this bibliographic overview, we presented the classification reported by Muanda (2010), which was reported in detail.

3.1. Alkaloids

Alkaloids are one of the largest groups of secondary metabolites with almost 10,000 to 12,000 different structures (Stöckigt et *al*., 2002, Roberts and Wink, 1999). This group includes organic substances of plant origin, with an alkaline character, that contain a nitrogen atom in the structure. More than 12,000 composites of alkaloids are known (McMurry, 2015). The colchicine, atropine, tubocurarine, theine, cocaine, mescaline, lysergic acid and aconitine are the most known alkaloids. Around 1805, the first alkaloid (morphine) was isolated in opium and, in 1818, the strychnine was discovered (Muanda, 2010).

The alkaloids have complex molecular structures and even at low doses, they have important physiological properties (Zenk and Jueng, 2007). They are used in drugs, medicines, and poisons, they have a potent analgesics and antimalarial activities, also used for treatment of hypertension, mental disorders, and tumors (Hesse, 2002, Cordell 1981, Rueffer 1988, Harborne 1999)

3.2. Terpenes

Terpenes are derivatives of isoprene. They form a class of hydrocarbons, produced by many plants, especially conifers. These are major components of resin and the essence of turpentine produced from resin. The α-pinene, ß-pinene, δ-3-carene, limonene and carotene are cited among the most important terpenes (Muanda, 2010).

3.3. Sterols

Sterols are naturally present in the lipid fraction of plants. They are derivatives of phytosterols. They can only be provided by food because they are not synthesized by humans and animals (Muanda, 2010).

3.4. Steroids

Steroids are a group of lipids derived from triterpenoids (mainly squalene) and they are characterized by a hydrophobic cyclopentanophenanthrenic nucleus, partially or completely hydrogenated (Muanda, 2010).

3.5. Saponins

By definition, a saponin is a steroid or triterpene glycoside. They are divided into steroidal saponins and triterpene saponins (Muanda, 2010). They have hemolytic, antimicrobial, insecticide and molluscicide properties (Vincken et *al*., 2007).

3.6. Phenolic compounds

Phenolic compounds include a broad set of chemicals including at least one aromatic ring and one or more hydroxyl groups, in addition to other constituents (Bamforth, 2000). They can vary from simple molecules, such as phenolic acids to highly polymerized compounds, over 30,000 Dalton, such as tannins (Sarni-Manchado and Cheynier, 2006, Hagerman et *al*., 1998). The polyphenols have several phenolic groups, with or without other functions (alcoholic (OH), carboxylic (COOH), etc.). They are probably the most common natural compounds in nature. These compounds have a wide variety of structures, divided into non-flavonoids and flavonoids.

3.6.1. The non-flavonoids

This group includes phenolic acids, lignans, coumarins, stilbenes and xanthones.

3.6.1.1. Phenolic acids

The phenolic acids are universally encountered and distributed in plants (Dai and Mumper, 2010, Elzaawely et *al*., 2007). They contribute to the taste, flavor, and health-benefits presents in vegetables and fruits (Tomas-Barberan and Espin, 2001). These compounds are distinguished in two groups (Muanda, 2010, Bruneton, 2009, Andersen and Markham, 2006):

3.6.1.1.1. Hydroxybenzoic acids

These acids respond to a structural representation of type (C_6-C_1), of which the most answered are salicylic acid and gallic acid.

3.6.1.1.2. Hydroxycinnamic acids

Responding to a structural representation of type (C_6-C_3), the caffeic and coumaric acids are the most abundant in this group.

3.6.1.2. Stilbenes

The stilbenes have a structure of the C_6-C_2-C_6 type. They proceed as phytoalexans that are formed by plants when attacked by microorganisms and nematode pathogens (Rice-Evans and Packer, 2003). The resveratrol is part of this group of compounds, which is an anticancer present in certain medicinal plants (Andersen and Markham, 2006, Bruneton, 2009, Muanda, 2010).

3.6.1.3. Lignans and lignin

The lignans have a structural representation of type $(C_6\text{-}C_3)_2$. They are mainly found in oilseeds. Lignans are involved in the defense mechanisms of the plant (Stalikas, 2007). As for lignin, they have a structural representation of type $(C_6\text{-}C_3)_n$. They contribute to forming, with cellulose and hemicellulosic derivatives, the wall plant cells. Lignin dissuade herbivory by its physical toughness but also avoids microbial proliferation just after infection of wounds by the process of lignification (Maderand Amberg-Fisher, 1982, Gross, 1992, Gould, 1983). Lignin possess several biological activities, including fungal growth inhibition, fish toxicity, insecticidal functions (Ghabru and Rana, 2019).

3.6.1.4. Coumarins

Coumarins are odorous substances present in many plants with variable actions (Bruneton, 1999). They have an intense blue fluorescence in ultraviolet light. They are divided into simple and complex coumarins (furocoumarines, pyrannocoumarines) (Bruneton, 2009, Cseke et *al*., 2006). As reported by Ahmed et *al*. (2017), coumarins play an essential role in the plant defense system and the protection of plants from herbivores, pathogenic insects, bacteria and fungi (Murray et *al*., 1982) and they are characterized by a higher antimicrobial activity *vs.* bacteria and fungi. These compounds result from the Shikimic acid pathway (Serghini et *al*., 2001, Murray et *al*., 1982).

3.6.1.5. Xanthones

Xanthones are a tribe of polyphenolic compounds with a basic C_6-C_1-C_6 structure (Bruneton, 2009). They are usually found in superior plants.

3.6.2. The flavonoids

Flavonoids represent a secondary metabolites family generally distributed in the plant taxa. They are universal plants pigments and are the partial responsible of flower and fruit colors and sometimes leaves. They are commonly encountered in superior plants. More than 6000 flavonoids have been isolated and identified (Ghasemzadeh, 2011, Austin and Noel, 2003, Stöckigt et *al*., 2002, Tolonen et *al*., 2002). All the flavonoids have the same basic structure with fifteen carbon atoms composed of two aromatic units, two C_6 rings, linked by a C_3 chain (Mamyrbékova-Békro et *al*., 2008, Bruneton, 1999).

3.6.2.1. Flavonoids in the strict sense

Flavonoids are divided into different classes based on their skeleton (Edenharder and Grünhage, 2003, Havsteen et *al*., 2002): chalcones, aurones, flavones, flavans, flavanones, flavanols, flavonols and flavanonols.

3.6.2.2. Isoflavonoids

Isoflavonoids are sub-class of flavonoids. They derive from a 1,2-diphenylpropane structure (Schwarz et *al*., 2007). They are almost specific to the Fabaceae family, probably due to the existence in this family of the enzyme that rearrange the 2-phenylchromone (flavanone) to 3-phenylchromone (isoflavone). According to the degree of oxidation and the existence or not of additional heterocycles, they are classified into 3-arylcoumarins, coumaronochromones, coumestanes, isoflavans, isoflavenes, isoflavones, rotenoids, pterocarpans (Muanda, 2010).

3.6.3. Anthocyanins

Anthocyanins is a general term that includes anthocyanidols and their glycosylated derivatives (Guignard, 1996). This sub-class of flavonoids possess a charge on the oxygen of hetero rings C. They have a basic structure with a "flavone" nucleus generally glycosylated in position C_3 (Ribereau, 1968). These molecules are able to absorb the visible light and they are the responsible of the blue, red, mauve, pink or orange coloration of plants (Harbone, 1998, Brouillard, 1986).

3.6.4. Tannins

Tannins are polar polyphenols of plant origin, which exist in almost every part of the plant: bark, wood, leaves, fruit, and roots (Berthod et *al*., 1999). They are divided into two groups, hydrolysable tannins, and condensed tannins (Cowan, 1999, Clifford et *al*., 1997).

Tannins have toxic properties and process as deterrent for herbivores. They provide astringent sensation in combination with salivary proteins (Clifford et *al*., 1997).

4. Free radicals, oxidative stress, and antioxidants

Free radicals are chemical species containing one or more unpaired electrons called single electrons (Halliwell and Gutteribge, 1977), these radicals can be formed by mono-electronic transfers or by homolytic cleavage of covalent bond (Bonnefont-Rousselot et *al*., 2003). In normal daily circumstances, free radicals are produced continuously and in small quantities and this physiological production and perfectly controlled by defense systems, moreover, adaptive to the level of radicals' present. Under these normal circumstances, it is said that the antioxidant/pro-oxidant balance is in balance. If this is not the case, whether due to antioxidant deficiency or because of a huge overproduction of radicals, the excess of these radicals is called oxidative stress (Favier, 2003).

When the cell uses oxygen, a lot of oxidation reactions take place. The result is the production of energy, but also of various by-products called reactive oxygen species "ERO" (Pokorny et *al*., 2001).

Ghasemzadeh (2011) described oxidative stress as crucial in the induction and progress of multitude conditions and diseases present currently, counting (Lukyanova et *al*., 2007): inflammation, autoimmune diseases, cataract, cancer, Parkinson's disease, arteriosclerosis, and aging. According to Astley (2003) and Zima et *al*. (2001), the oxidative stress is also involved in heart diseases neurodegenerative diseases, cancer and in the aging process.

As reported by Ghasemzadeh (2011), antioxidants are molecules or substances that greatly inhibit or prevent the oxidation of an oxidizable material albeit present in small concentrations in comparison to the material (Lucio et *al*., 2009). In recent years, phenols and flavonoids have reached a great interest for their antioxidant activity, revealed more efficient than that of Vitamin C, E, and carotenoids (Dai and Mumper, 2010).

5. Role and interest of secondary metabolites in the human organism

Plants are particularly important in the development of new medicines. They are an essential origin of bioactive molecules or of inspiration in pharmaceutical field. Indeed, secondary metabolites, used as a separate compound or as a combination, are medicines that may be efficient and secure even in case of synthetic drugs failed. They can alike potentially synergize the effects of other medicine compounds (Maria et *al*., 2019).

The role of secondary metabolites is widely shown in protection against certain diseases as natural antioxidants, anti-inflammatory, antidiabetic, antimicrobial, antifungal, antiproliferative, etc., in addition to other common uses. Some examples of secondary metabolites and their biological activities are listed in the Table III.1.

The secondary metabolites display diverse beneficial properties in pharmaceutical, cosmetic and food industries; however, some of them produce detrimental effects in humans and animals and damage some food types.

The secondary metabolites display certainly diverse beneficial properties in pharmaceutical, cosmetic and food industries, however some of them produce detrimental effects in humans and animals and also damage to some food types, for example we cited the toxins secreted by several pathogenic bacteria which contaminate food industry products (Gokulan et *al*., 2014).

Table III.1. Biological properties and uses of some secondary metabolites for the body.

Secondary metabolites	**Biological properties**	**References**
Coumarins	Analgesics, anti-inflammatory, vasculoprotective, antiparasitic, anti-edematous, anti-tumor, hemorrhoid, and varicose vein care, neurosedative, diuretic, stomachic and carminative	Smyth et *al*. (2009), Ito *et al*. (2005) and Hennebelle et *al*. (2004).
Alkaloids	Treatment of cardiovascular, nervous, gastrointestinal diseases for humans and animals. Antitumor, antipyretics and antimalarial properties and used as protective substances *vs.* animal or insect attacks.	Bruneton (2009), Reyburn et *al*. (2009) and Jordan and Leslie (2004).
Phenolic acids	Antiparasitic, antifungal, antioxidant, antibacterial and anti-ulcer.	Kim et *al*. (2010) and Flores et *al*. (2009).
Xanthones	Antimicrobials and cytotoxicity especially inhibition of monoamine oxidase.	Panglossi (2006).
Anthocyanins	Diuretics, capillary-venous protectants, and antioxidants.	Bruneton (2009) and Hennebelle et *al*. (2004).
Terpenes	Fragrance and flavoring agents.	Ohloff (1990).
Flavonoids	Antispasmodic, antitumor, anticarcinogenic, antiparasitic, antiviral, antimicrobial, antioxidant, anti-inflammatory, antiallergic, antithrombotic, antiatherogenic, hypotensive, analgesic, osteogenic, diuretic, and anti-allergic.	Batovska et *al*. (2009), Kssouri et *al*. (2008), Cushnie et *al*. (2007), Friedman et *al*. (2006), Lim et *al*. (2006), Hennebelle et *al*. (2004), Middleton et *al*. (2000) and Mills and Bone (2000).
Condensed tannins	Stabilizing effects on collagen, antioxidant, anti-tumor, antifungal and anti-inflammatory activities.	Zhou et *al*. (2011) and Masquelier et *al*. (1979).
Lignans	Healing, anti-diarrheal, anti-inflammatory, and analgesic.	Kim et *al*. (2009) and Hennebelle et *al*. (2004).
Gallic and catechic tannins	Antioxidants, antimicrobials, antivirals, anti-inflammatory and hypoglycemic agents.	Kubata et *al*. (2005) and Okamura et *al*. (1993).

6. The methods used to assess the antioxidant activity

The antioxidant activity of samples could be evaluated *in vivo* and *in vitro*. In this investigation, we are interested by the *in vitro* evaluation. In view of there is not an approved and standardized method for determining the exact antioxidant capacity of a material, the performance of more than one test is therefore necessary. There are two principal ways to evaluate the *in vitro* antioxidant activities of samples: the inhibition of lipid oxidation in diverse systems and the capacity to scavenge free radicals (Aazza et *al*., 2011b).

There are several methods and tests to assess the *in vitro* antioxidant activity of a material, we can cite the followings: DPPH scavenging activity (Tefiani et *al.,* 2016), ABTS scavenging ability (Aazza et *al*., 2011a), hydroxyl radical scavenging activity (Chung et *al*., 1997), superoxide anion scavenging activity (Non-enzymatic method) (Soares, 1996), nitric oxide scavenging capacity (Ho et *al*., 2010), chelating metal ions ability (Wang et *al.,* 2004), reducing power determination (Oyaizu, 1986), thiobarbituric acid reactive species (TBARS) (Dorman et *al*., 1995), liposomes (Bakchiche et *al*., 2013), etc.

In this section, we will describe the three methods (DPPH and ABTS scavenging activities and chelating metal ions ability), that we used in this thesis to evaluate the *in vitro* antioxidant activity of the nineteen selected weed species. The ability to scavenge ABTS and DPPH free radicals can be criticized because they are non-physiological free radicals. Nonetheless, they are considered as reference methods where they are used basically in all studies evaluating the antioxidant activity of samples because of their simplicity and ease (Bahloul et *al*., 2016).

6.1. DPPH assay

The method of 1, 1-diphenyl-2-picrylhydrazine free radical scavenging ($DPPH^{\bullet}$) was first described by Blois (1958). At ambient temperature and in alcoholic solution, this free radical presents an intense violet coloration that disappears once in contact with a proton-donating substrate (Figure III.3). The antioxidant ability is manifested by this discoloration, which indicates the capacity of the sample to scavenge this radical, which results in a decrease in absorbance at 517 nm (Masuda et *al*., 1999).

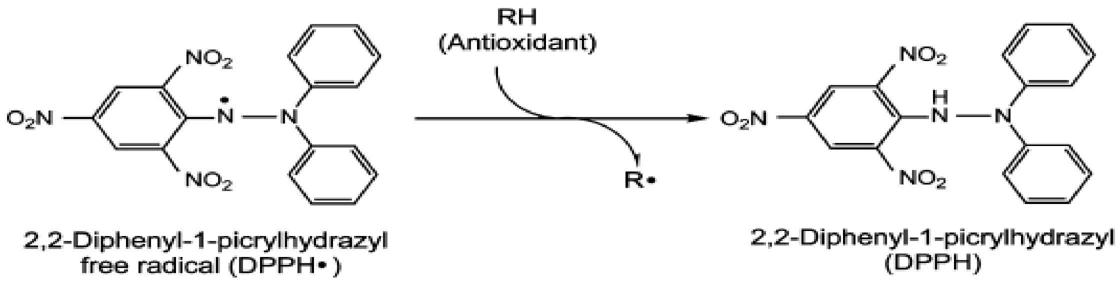


Figure III.3. Reduced form of the DPPH $^{\bullet +}$ radical (Moon and Shibamoto, 2009).

6.2. ABTS assay

As regards to the ABTS assay, the ammonium salt of 2,2'-azinobis- (3-ethylbenzothiazoline- 6-sulfonic acid (ABTS) free radical is obtained (Miller and Rice Evans, 1997) from contact of ABTS with a peroxidation enzyme (metmyoglobin peroxidase) in the presence of H_2O_2 or an oxidant, manganese dioxide (Benavente-Garcia et *al*., 2000, Miller et *al*, 1996), or potassium persulfate (Moon and Shibamoto, 2009). The generation of the ABTS free radical by potassium persulfate is illustrated in the Figure III.4.

The appearance of an intense blue green coloration indicates the formation of this free radical (ABTS $^{\bullet +}$). In the presence of a hydrogen donor (antioxidant agent), the disappearance of this coloration, at 734 nm is accompanied by the passage from the ABTS $^{\bullet +}$ radical to the non-radical form which express the ability of the sample to scavenge the ABTS free radical (Lien et *al*., 1999, Re et *al*., 1999).

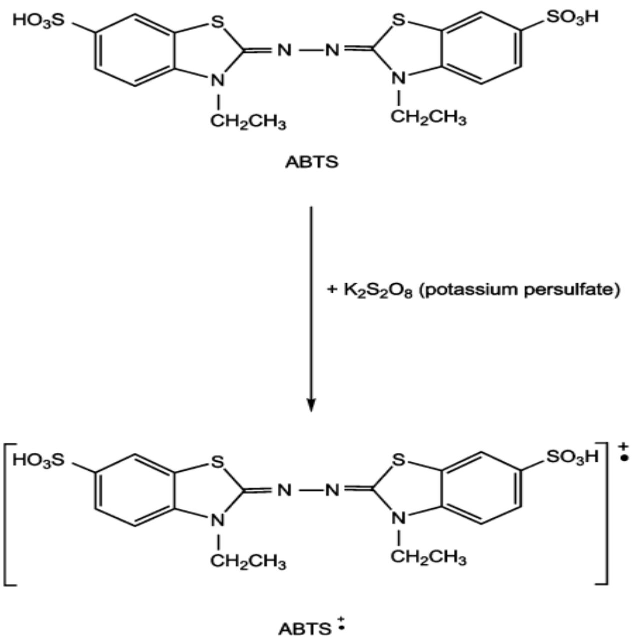


Figure III.4. Formation of ABTS $^{\bullet +}$ by a potassium persulfate oxidant (Moon and Shibamoto, 2009).

6.3. Chelating metal ion assay

Chelating metal ions is part of the mechanisms of antioxidant activity. The chelation of the metal ion transition stimulates lipid peroxidation and thereby, takes part in the acceleration of this peroxidation, in lipid hydro-peroxide in other compounds capable of removing hydrogen and also by perpetuating peroxidation lipids (Miguel, 2010). This activity consists of chelating trace metal ions involved in the formation of free radicals (Cotelle, 2001).

Ferrozine is the most used stabilizing compound (Figure III.5) to assess the chelating ferrous ions activity of a sample (Gulcin, 2012). In a reaction medium, the free iron present forms with ferrozine a ferrozine-Fe^{2+} complex of an intense purple color. The disappearance of this color measured by spectrophotometry at 562 nm expresses the capacity of the sample to chelate iron (Zhao et *al.*, 2006).

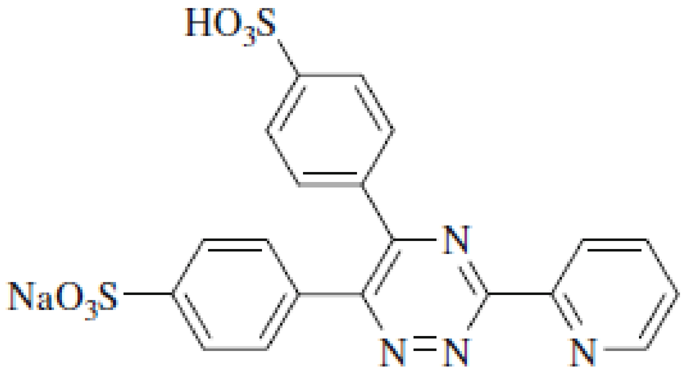


Figure III.5. Chemical structure of ferrozine (Gulcin, 2012).

7. A literature review on the nineteen investigated weed species

Nineteen weed species were chosen to be investigated from the total inventory realized in the first experimental part of this thesis. These species represent 20% of the total flora.

The selected species have been used from antiquity in the folk medicine and also have been investigated by several scientists, for the phytochemical composition of their extracts, their essential oil and hydrosols, for their antioxidant activities and also for others biological activities. A brief literature review regarding the folk uses, secondary metabolites and biological activities of the studied weed species is provided in Table III.2.

Table III.2. Folk uses, plant secondary metabolites and biological activities reported in the literature for the nineteen weed species under study.

Weed species	Folk uses	References	Plant secondary metabolites	References	Biological activities	References
Anagallis arvensis L.	Respiratory infections, infected spots, diaphoretic, diuretic, expectorant for the treatment of rheumatism, dropsy, and hepatic and renal complaints.	Gulshan et *al*. (2012). Sundaram et *al*. (2010) and López et *al*. (2008).	Saponins, tannins, flavonoids, glycoside, alkaloids, phenols, and resin.	Sharifi-Rad et *al*. (2016), Tawaha et *al*. (2007) and Apak et *al*. (2006).	Antioxidant, antifungal, antibacterial, allelopathy and antitumor activities.	Akhtar et *al*. (2018), Shakoor et *al*. (2018) and Rebaz et *al*. (2001).
Aristolochia baetica L.	To treat constipation, wounds, ringworm, burns and tumor, arthritis prevention, emmenagogue and stimulant.	Achenbach and Fischer (1997), Bellakhdar (1997) and Millspaugh (1887).	Phenols, alkaloids, flavonoids, saponins, tannins and terpenoids.	Bourhia et *al*. (2019) and Wu et *al*. (2004).	Anti-proliferative, cytotoxic, antioxidant, insecticidal and larvicidal activities.	Bourhia et *al*. (2019), Bnouham (2012) and Jbilou et *al*. (2008).
Calendula arvensis L.	Treatment of wounds, burns, skin complaints, conjunctivitis, low eyesight, menstrual irregularities, varicose, veins, hemorrhoids, and duodenal ulcers. Vegetable, spice, tea, tincture, ingredient, dye, diaphoretic, and sedative.	Passalacqua et *al*. (2007) and De Clavijo (2005).	Phenols, flavonoids, terpenoids, tannins, saponins and alkaloids.	Akhtar et *al*. (2018)	Antioxidant, antifungal, antibacterial, insecticidal, anticandidal, anti-cancer, cytotoxic, phytotoxic and acetylcholinesterase activities.	Abudunia et *al*. (2016), Ercetin et *al*. (2012) and Ullah et *al*. (2012).

Chrysanthemum coronarium L.	Consumed as vegetable and used for decoration, plasters against dermal diseases, vermifuge, expectorant and stomachic. Treatment of gonorrhea, syphilis, chronic constipation, headaches, eye diseases, ringing in the ears, swamp fever, alcoholism, kidney stone disease, radiation sickness, cardio-vascular diseases, rheumatism, and hypertension.	Bar-Eyal et *al*. (2006), Song et *al*. (2003) and Oran and Al Eisawi (1998).	Phenols, tannins, flavonoids and saponins.	Hosni et *al*. (2013)	Antiproliferative, antimicrobial, antioxidant, acetylcholinesterase, antifungal and phytotoxic activities.	Bordoloi et *al*. (2016), Bardaweel et *al*. (2015) and Hosni et *al*. (2013).
Chrysanthemum segetum L.	-	-	Phenols, flavonoids and, coumarins.	Derouiche et *al*. (2018), Kennouche et *al*. (2016) and Ochocka et *al*. (1995).	Antioxidant, antimicrobial, nematocidal and insecticidal activities.	Derouiche et *al*. (2018), Kennouche et *al*. (2016), Haouas et *al*. (2010), and Péreza et *al*. (2003).
Convolvulus althaeoides L.	-	-	Phenols, flavonoids, saponins, tannins and alkaloids.	Aly Ahmed et *al*. (2014), Tawaha et *al*. (2007).	Antioxidant, antibacterial and cytotoxic activities.	Hassine et *al*. (2014) and Tawaha et *al*. (2007).

Echium vulgare L.	Used in diets. To treat colds, coughs, fevers, headaches, water retention, kidney stones, inflammation, pain, reddened skin and boils, melancholia, wounds, and ulcer.	Conforti et *al*. (2009), Özer et *al*. (2004) and Schmidt et *al*. (2003).	Phenols, flavonoids, tannins, and alkaloids.	Vukajlović et *al*. (2019), Eruygur et *al*. (2012) and El-Shazly et *al*. (1996).	Antioxidant, analgesic, antiproliferative and insecticidal activities.	Vukajlović et *al*. (2019), Eruygur et *al*. (2012) and Conforti et *al*. (2008).
Emex spinosa (L.) Campd.	Consumed as vegetable. To treat constipation, indigestion, jaundices liver problems, male sexual disorders, and stomach disorders. Used as forage, labor inducer, appetite stimulator, diuretic, and purgative.	Shahat et *al*. (2015), Abbas and Al-Saleh, (2002) and Watt and Breyer-Brandwjik (1962).	Phenols, flavonoids, triterpenoids, and alkaloids.	Makni et *al*. (2019) and Biosci et *al*. (2014).	Anti-inflammatory, antioxidant, cytotoxic, antispasmodial, phytotoxic and acetylcholinesterase activities.	Makni et *al*. (2019), Shahat et *al*. (2015) and Biosci et *al*. (2014).
Fumaria capreolata L.	To treat hepatobiliary dysfunction, fever, gastrointestinal disorders, infections, diarrhea, syphilis, and leprosy. Anthelmintic, antidyspeptic, blood purifier, diuretic, laxative, sedative, tonic, spasmolytic and depurative.	Paltinean et *al*. (2017), Bribi et *al*. (2015) and Gilani et *al*. (2005).	Alkaloids, phenols, and flavonoids.	Paltinean et *al*. (2017), Bribi et *al*. (2015) and Maiza-Benabdesselam et *al*. (2007).	Antioxidant, diuretic, acetylcholineserase, analgesic, antinociceptive and anti-inflammatory activities.	Bribi et *al*. (2015), Erdogan et *al*. (2012) and Mukherjee et *al*. (2007).

Hedera helix L.	Ornamental plant. Used for treatment of wounds, burns, dermatitis, gastrointestinal irritation, bloody, gout, diarrhea, post-partum disorders, rheumatism, respiratory tract inflammation, parasites, homeopathy, and hyperthyroidism.	Miser-Salihoglu et *al*. (2013), Blumenthal (2000) and Gruenwald et *al*. (2000).	Phenols, flavonoids, saponins, coumarins, anthocyanin, tannins, sterols, alkaloids and terpenoids.	Galip Akaydin et *al*. (2013), Uddin et *al*. (2011) and Lutsenko et *al*. (2010).	Antioxidant, antibacterial, antifungal, anti-mycobacterial, antitumor, and antihypertensive activities.	Salma et *al*. (2020), Deliorman Orhan et *al*. (2012) and Lutsenko et *al*. (2010).
Inula viscosa L.	For treatment of skin diseases, diabetes, tuberculosis, anemia, inflammation and as antiseptic.	Zeggwagh et *al*. (2006), Al-Dissi et *al*. (2001) and Alarcón de la Lastra et *al*. (1993).	Flavonoids, steroids, alkaloids, tannins, phenols, sterols, and carotenoids.	Mahmoudi et *al*. (2016) and Nikolakaki and Christodoulakis (2004).	Antioxidant, antiproliferative, antibacterial, antifungal, allelopathic, insecticidal anticholinesterase and anti-cancer activities.	Bar-Shalom et *al*. (2019), Ozkan et *al*. (2019) and Omezzine et *al*. (2011).
Malva sylvestris L.	Used in diets. For treatment of inflammations, chest pain reliever for children and cough. Emollient, laxative and antihemorrhoidal agent.	Conforti et *al*. (2008), Couplan (2003) and Classen et *al*. (2001).	Phenols, flavonoids, flavonols, tannins, saponins, anthocyanin and carotenoids.	Beghdad et *al*. (2014), Tabarki et *al*. (2012) and Barros et *al*. (2010).	Anti-inflammatory, antioxidant, allelopathic, acetylcholinesterase, antimicrobial and cytotoxic activities.	Majda et *al*. (2017), Ozsoy et *al*. (2017) and Burcin Akgunlu et *al*. (2016).
Medicago rugosa Desr.	-	-	-	-	Allelopathic.	Fujii (2001).

Oxalis pes-caprae L.	Diuretic. Culinary herb used as a salad and as ingredient. To treat swelling and cognitive disorder and to fight against parasites (tapeworm and other worms).	Edrah et *al*. (2018) and Watt and Breyer-Brandwijk (1962).	Phenols, terpenes, alkaloids, saponins, flavonoids, glycosides and coumarins.	Edrah et *al*. (2018) and Gaspar et *al*. (2018).	Anti-inflammatory, antioxidant, neuroprotective, antibacterial, and acetylcholinesterase activities.	Edrah et *al*. (2018), Gaspar et *al*. (2018) and Ali-Shtayeh et *al*. (2014).
Reichardia tingitana L.	Ornamental plant. For treatment of inflammations and diabetes. Used as tea, a source of vitamins and minerals, insecticidal and anti-feeding.	The Local Food-Nutraceuticals Consortium, (2005), Stalińska et *al*. (2005) and Daniewski et *al*. (1988).	Phenols, esters, flavonoids, tannins, saponins, alkaloids, sterols, aldehydes, ketones, volatile oils, coumarins and triterpenes.	El Alfy et *al*. (2015) and El-Gawad et *al*. (2015).	Antioxidant, allelopathic, cytotoxic, antitumor, antimicrobial, and insecticidal activities.	Chandel and Singh (2017), El-Gawad et *al*. (2015) and El Alfy et *al*. (2015).
Rubia peregrina L.	Used as a natural dye. To heal wounds, inflammation, and skin infections.	Meena (2010) and Usai (2004).	Anthocyanins.	Longo et *al*. (2007).	Antioxidant and antimicrobial activities.	Özgen et *al*. (2003).
Sideritis montana L.	Used as antispasmodic, carminative, anti-inflammatory, anti-ulcer, cytostatic, antimicrobial, flu vaccine, stimulant circulatory agent, tea, and flavoring agent.	Emre et *al*. (2011) and Basile et *al*. (2006).	Phenols and flavonoids.	Balkan et *al*. (2019), Radojević et *al*. (2012), and Emre et *al*. (2011).	Antioxidant, cytotoxic antifungal, and antiproliferative activities.	Balkan et *al*. (2019), Tóth et *al*. (2017) and Venditti et *al*. (2016).

Sinapis arvensis L.	Used as vegetable, boiled, salads, seasoning, savory or rustic cake.	Disciglio et *al*. (2017) and Sarikurkcu et *al*. (2017).	Phenols, flavonoids, saponins, flavonols and carotenoids.	De Oliveira et *al*. (2019) and Sarikurkcu et *al*. (2017).	Antioxidant, antimicrobial, enzyme inhibitory and antifungal activities.	Torun et *al*. (2018), Sarikurkcu et *al*. (2017) and Rad et *al*. (2013).
Withania frutescens Pauquy.	Treatment of dysentery, used against poisoning and gastric ulceration.	Bellakhdar (1997).	Phenols and flavonoids.	El Moussaoui et *al*. (2019), El Bouzidi et *al*. (2017) and Rached et *al*. (2010).	Antioxidant, antimicrobial, antifungal and antiplasmodial activities.	El Moussaoui et *al*. (2019), El Bouzidi et *al*. (2017), El Bouzidi et *al*. (2011) and Rached et *al*. (2010).

- : Not found.

8. Conclusion

Secondary metabolites are organic substances released from various plants that act in the defense system, chemical signal compounds between plant *vs.* plant, plant *vs.* animal/insects and plant *vs.* microorganism interactions. In addition to their protective role, several medicinal properties and biological activities are attributed to them. Their antioxidant activities and their contribution in the prevention of oxidative stress have been reported briefly in this chapter. As well, some *in vitro* method used to assess the antioxidant activity of a materiel.

Plants as well as weeds produce a vast range of bioactive substances, with diverse structures. Since plants have reached a great interest for their secondary metabolites and biological activities, weeds may be a new axis of research and a promising area of investigation. Weeds are very abundant in nature and in cultivated fields, belonging to different botanical families and to different genera, which might be studied and exploited instead of eliminating them, thus, providing a new source of incoming for farmers and furnishing natural bioactive compounds.

In the corresponding experimental trial part, we studied nineteen weed species (selected from the floristic inventory established in the first experimental part), for their phenol and flavonoid contents, DPPH and ABTS scavenging ability and their chelating metal ions capacity. This experimental part aimed to the valorization of weeds that composed the citrus weed flora to be used as antioxidants in pharmaceutical, food and cosmetic industries.

Chapter IV

Minerals and nutritive value

Literature Review

Chapter IV

Minerals and nutritive value

Contents

1. Introduction

Many livestock species use forages (either harvested mechanically or by grazing) as their primary source of nutrition. Thus, it is important to provide animals with the best quality forage available. Good quality forage ensures healthy animals and minimize the costs of purchasing concentrate feeds. The stage of maturity at harvest plays a major role in determining the quality of a forage. Early in the growing season, forage plants get into their vegetative stage, marked by leafy growth containing high concentrations of starches, sugars, proteins, and minerals.

During the grazing season, vegetative forage generally provides the nutritional needs of livestock. however, outside of the grazing season, livestock depend on the producer to provide for their nutritional needs. It is during this period, when animals are fed conserved forage, in which, the nutritional management program plays an important part in maintaining the growth and condition of livestock. In order to provide a ration that meets the nutritional needs of livestock, it is priority to know the nutritive value of the forage (Ron Hathaway and Gene Pirelli, 2004). In addition, forage must be available at a cost that will permit profitable livestock production (Hag, 1951).

As alternative source, weeds can be a significant basis for feeding ruminants. In this context, studies about nutritive value of weeds have increased due to their potential for animal feeding uses. Weeds have high nutritional constituents consisting of essential minerals (major and trace elements), fatty acids, proteins, dietary fibers, and vitamins (Gutiérrez et *al.*, 2008, Nashiki et *al.*, 2005, Marten and Andersen, 1975). Rather than, they are culturally important for local farmers as a vegetable source presenting a major part of their diet and culinary tradition (Cruz-Garcia and Price 2012).

In this part, the different parameters that constitute and determine a forage nutritive value (or plant nutritive value in general) and the principal nutrients for ruminants will be provided. Functions, deficiencies symptoms, and excess disorders of the different nutrients will be also highlighted. In addition to a literature review on the 19 weed species selected in the present study to be valorised from a forage value point of view.

2. Forages nutritive value

Nutrients supply for livestock is provided generally by forages, which are the main diet component (Newman et *al.* 2009). In general, forages are the vegetative parts of grasses containing a high proportion of fiber (more than 30% of neutral detergent fiber). They are required in the diet in a coarse physical form (particles larger than 1 or 2 mm in length).

Forages usually occur at the farm, directly grazed, and harvested and preserved as silage or hay. From a nutritional viewpoint, forages may range from very good feeds (lush young grass, legumes at a vegetative stage) to very poor (straw and roughage). Generally, the feeding value of a forage reaches its maximum during vegetative growth stage and the lowest during seed formation stage. As maturity advances, the concentration of CP, energy, calcium, phosphorus, and digestible dry matter in the plant is decreased; while, increasing the amount of NDF. By increasing NDF, the lignin content augment that make carbohydrates less available to the rumen microbes. Therefore, the energy value of the forage decreases (Ramirez Lozano, 2015).

Mostly, forage quality and forage nutritive value terms are used interchangeably but a distinction could be made between them. Forage nutritive value usually refers to concentration of available energy (total digestible nutrients, TDN) and concentration of crude protein. Whereas forage quality is a broader term that not only includes nutritive value but also forage intake. In practice, animal performance of grazing animals reflects forage quality (Newman et *al.*, 2009).

Forage/plant material can be separated, from a nutritional point of view (Table IV.1) to the cell contents fraction (organic acids, soluble carbohydrates, crude protein, fats and soluble ash) and the cell wall fraction (hemicellulose, cellulose, lignin, cutin and silica). Nothing that the digestibility (Table IV.1) varies between the different components (Van Soest, 1982).

Maturity and weather conditions are the main factors that affect forage quality. Maturity or stage of growth is the principal factor responsible for decreasing forage nutritive value. With advancing maturity, lignin that accumulates mostly at maturity acts as a barrier to fiber degradation by rumen microbes. Moreover, as the forage ages, digestibility, and protein decline while fiber increases. Weather conditions also affect forage quality, in addition to poor storage and harvest conditions that lead to losses of sugars (Newman et *al.*, 2009).

Table IV.1. Classification of forage fraction (Ramirez Lozano, 2015).

Forage fraction	Components included	Digestibility by ruminant
Cell contents	-Sugars, starch, and pectin. -Soluble carbohydrates. -Protein, nonprotein. -Nitrogen, lipids (fats). -Water soluble vitamins and minerals.	-Complete -Complete -Higher -Higher -
Cell wall (NDF)	-Hemicellulose -Cellulose -Heat damaged protein -Lignin -Silica	-Partial -Partial -Indigestible -Indigestible -Indigestible

3. Nutrient requirements for ruminants

Nutrient requirements (energy, proteins, lipids, carbohydrates, major minerals, and trace minerals) vary among animals and depends on the age, weight, stage of production, rate of growth, environmental conditions, breed, gender, and other factors. The National Research Council published estimates of nutrient requirements for livestock, which represent reference values to achieve good nutrition for livestock, as well as to discuss the results of scientific research.

3.1. Energy

Newman et *al.* (2009) reported that the main sources of energy for ruminants are the products of carbohydrate fermentation in the rumen. As cited previously, forages contain two basic types of carbohydrates. Those associated with cell contents which are soluble, highly digestible and easily broken down by the rumen microbes, in contrast to those generally associated with the cell wall constituents, that are composed by fiber components, subject to partial degradation by rumen microbes (more resistant to degradation).

The concentration of available energy is indicated by the total digestible nutrients (TDN). this parameter is calculated as the sum of the digestible protein, digestible crude fiber, digestible nitrogen free extract, and 2.25 times the digestible fat. Total digestible nutrients vary with maturity (the older the forage the lower TDN value it will have and vice-versa) but also with forage type (Newman et *al.*, 2009).

3.2. Proteins

Regarding their functions, proteins are the principal constituents of the animal body and are constantly needed in the diet for cell repair and synthetic processes. This important nutrient possesses a vital element for animal maintenance, growth, reproduction, and milk production (NRC, 1981).

Proteins alongside with energy are the most important nutrients for ruminants because they support rumen microbes that in turn degrade forage. Forage protein amounts vary significantly depending on species, soil fertility, and plant maturity (Newman et *al*., 2009). According to their nutritional characteristics, proteins can be divided into soluble protein, rumen degradable protein, and rumen undegradable protein. For fresh forage, most of the protein is degraded in the rumen, with only an average of 25% that can pass unchanged into the small intestine (Minson, 1990). Dietary protein generally refers to crude protein (CP), which is measured indirectly by determining the amount of N in the forage plant and multiplying that value by 6.25. Ruminant CP requirement is influenced by the physiological state of the animal (Newman et *al*., 2009, NRC, 2001).

The CP content is related to the leafiness of the plant. According to Mangan (1982), plants CP are distinguished in three main groups, fraction I (composed of chloroplasts that contain approximatively 75% of the total leaf protein), fraction II (represent about 25% of total leaf protein, and is originates from chloroplasts and the cytoplasm) and the last fraction (III) (composed of chloroplast membrane proteins and contributes about 40% to the chloroplast protein). Cell walls contain a cell wall associated protein named extensin in relation to their role in fiber cross-linking. This protein is less soluble than leaf proteins which is recuperated in neutral detergent fiber.

As reported by NRC (1981), protein deficiencies exhaust stores in the blood, liver, and muscles, and predispose animals to a variety of serious and even fatal disorders. Feed intake will be reduced when the level of crude protein (CP) is below 6%. that leads to a combined deficiency of energy and protein (Platt et *al*., 1964, Perkins, 1957). Moreover, this deficiency reduces rumen function and lowers the efficiency of feed utilization. A retard fetal development, low birth weights, low kid growth, and depress milk production occur with a long-term protein deficiency (Singh and Sengar, 1970).

3.3. Fats

Lipids constitute the main energetic source for animals, and they have the highest caloric value among all the nutrients. The term fat (animal or vegetal) is used as a synonym for lipid in the human food as well as in the ingredients for animal nutrition (Baião and Lara, 2005).

Also, fat is frequently used as a generic term to describe compounds that present a high content of long-chain fatty acids (FAs) including triglycerides, phospholipids, nonesterified FAs, and salts of long-chain FAs. Long-chain FAs are the energy-rich moiety of fats. The dietary fat should not exceed 6-7 % of dietary DM (NRC, 2001).

Fats are present in small amounts in most natural feedstuffs available for animal feeding, except oilseeds. Nevertheless, supplemental fats are used in diets for nutritional or economic purposes in order to increase the level of fat in diets. Fat supplements may contribute to meeting the energy requirements of animals due to their high energy value. Moreover, it may be cheaper in some situations to provide energy as fat rather than carbohydrates (Doreau and Chilliard, 1997).

As reported by Palmquist (1976), Fat increased energetic efficiency in lactating cows by increasing total energy intake, by generating ATP more efficiently (ATP/unit energy expended) than volatile fatty acids or protein, by direct incorporation into product, and by promoting nutrient partition toward milk production.

The energetic value of fats depend on the length of the carbonic chain, the number of double bonds, the presence or absence of ester bonds (triglycerides or free fatty acids), the specific arrangements of the saturated and unsaturated fatty acids on the glycerol backbone and the composition of the free fatty acid (NRC, 2001).

Reduced DM intake can occur by feeding higher concentrations of fat (NRC, 2001). Lipid supplementation of diets frequently leads to a decrease in the extent of carbohydrate digestion (Doreau and Chilliard, 1997). In addition, providing supplemental fat to ruminants reduced digestibility of calcium, magnesium, or both in some studies (NRC, 2001).

3.4. Carbohydrates

Carbohydrates are the main source of energy in ruminants ‘diets. The major function of carbohydrates is to provide energy for rumen microbes and the host animal, in addition to another essential function that consists to maintain the health of the gastrointestinal tract (NRC, 2001). Microorganisms in the rumen permit the ruminant to obtain energy from fibrous carbohydrates (cellulose and hemicellulose) which are associated to the lignin in cell walls of the plant (Ramirez Lozano, 2015).

Carbohydrates account from 50 to 80% of the dry biomass of forages (Van Soest, 1982). They are divided in to structural (SC) or nonstructural (NSC) (Figure IV.1). Nonstructural carbohydrates are found inside the cells of plants and are generally more digestible than structural carbohydrates that are encountered in plant cell walls (NRC, 2001). The NSC fraction is composed by sugars, starches, organic acids, and other reserve carbohydrates (fructans, etc.) while the SC fraction contains crude fiber, acid detergent fiber (ADF), and neutral detergent fiber (NDF) (Figure IV.1).

Ruminants rapidly and completely ferment the nonstructural carbohydrates (starch and sugars) in the rumen. By improving energy supply and increasing microbial protein produced in the rumen. The content of nonstructural carbohydrates augments the energy density of the diet. Nevertheless, the rumination fermentation and saliva production are not stimulated by NSC. rather than they may inhibit fermentation of fiber when present in excess. Consequently, the balance between structural and nonstructural carbohydrates is necessary in ruminant feeding for optimal production (Ramirez Lozano, 2015).

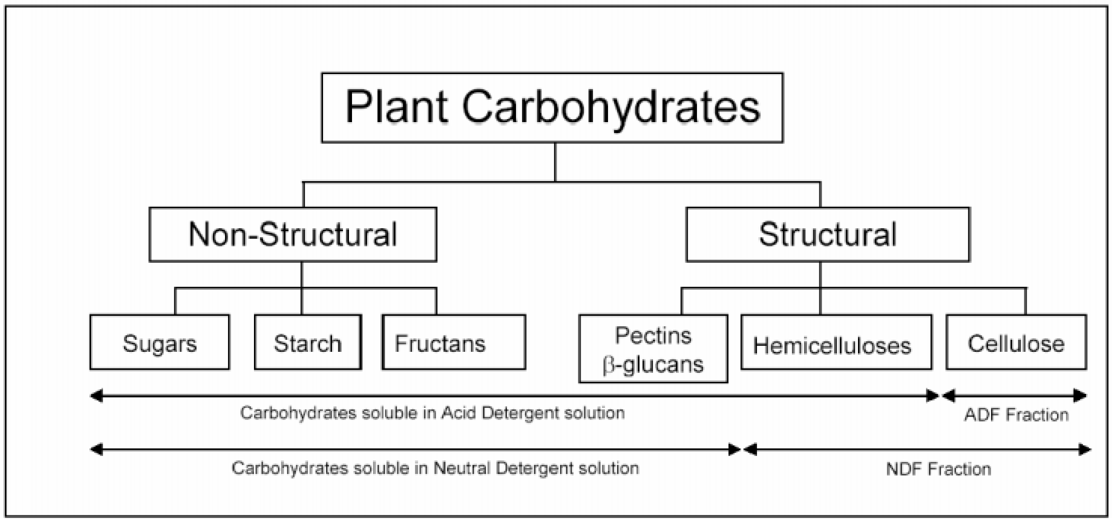


Figure IV.1. Simplified Diagram of Plant Carbohydrate Fractions.

Modified diagram, courtesy of Mary Beth Hall (University of Florida, now USDA) and reported in the technical note of Hill Laboratories, Number 8391 Version: 8.

3.4.1. Sugars

A chemical approach divides carbohydrates into three main groups, sugars, oligosaccharides (short-chain carbohydrates) and polysaccharides. Sugars are nonstructural carbohydrates found in the cell contents. They consist of monosaccharides, disaccharides, and polyols (sugar alcohols) (Cummings and Stephen, 2007). The most common simple sugars found in plants are glucose and fructose while the most abundant disaccharide is sucrose (molecule of glucose bonded to fructose). Lactose (glucose + galactose) is found in milk. Maltose is a disaccharide with the same glucose-to glucose alpha-linkage as starch (Zadeh and Kor, 2013).

The nutritional availability depends on the capacity to break the glycosidic linkages in the carbohydrates of plants and between carbohydrates and other compounds. They form an important portion of the food supply for animals and is the most abundant class of components found in plants. they play basic roles (intermediary metabolism, energy transfer, storage, and plant structure). Sugars are rapidly fermented in the rumen. The polysaccharides need to be degraded into simple sugars before being used. Nonstructural polysaccharides (starch and fructans) are rapidly and completely degraded in the rumen, while the degradability of structural polysaccharides (cellulose and hemicellulose) varies considerably (Ramirez Lozano, 2015).

3.4.2. Fiber

Fiber indicates the cell wall constituents of hemicelluloses, cellulose, and lignin. Fiber extraction in forages is realized with the detergent analyses system. Ramirez Lozano (2015) signaled that an extremely high fiber levels slow the rate of digestion and limit dry matter intake, but a certain amount of fiber is required to stimulate rumen activity. Diets with low fiber levels and high in concentrates generate in a low percentage of fat in the milk and causes digestive disorders (displaced abomasum and rumen acidosis).

3.4.2.1. Neutral Detergent Fiber (NDF)

The NDF values represent the total fiber fraction (cellulose, hemicellulose, and lignin) that compose cell walls (structural carbohydrates or sugars) of the forage tissue (Figure IV.2). It represents the insoluble portion of the forage. A high NDF amount indicates high overall fiber in forage. The lower the NDF value the better. In addition, the NDF is negatively correlated with dry-matter intake (Lardy, 2018, Newman et *al*., 2009).

3.4.2.2. Acid Detergent Fiber (ADF)

The ADF values represent cellulose, lignin, and silica (if present) (Figure IV.2). The ADF fraction of forages is relatively indigestible. High ADF values are related with decreased digestibility (as the ADF increases, the forage becomes less digestible), thus a low ADF is desired (Lardy, 2018, Newman et *al*., 2009).

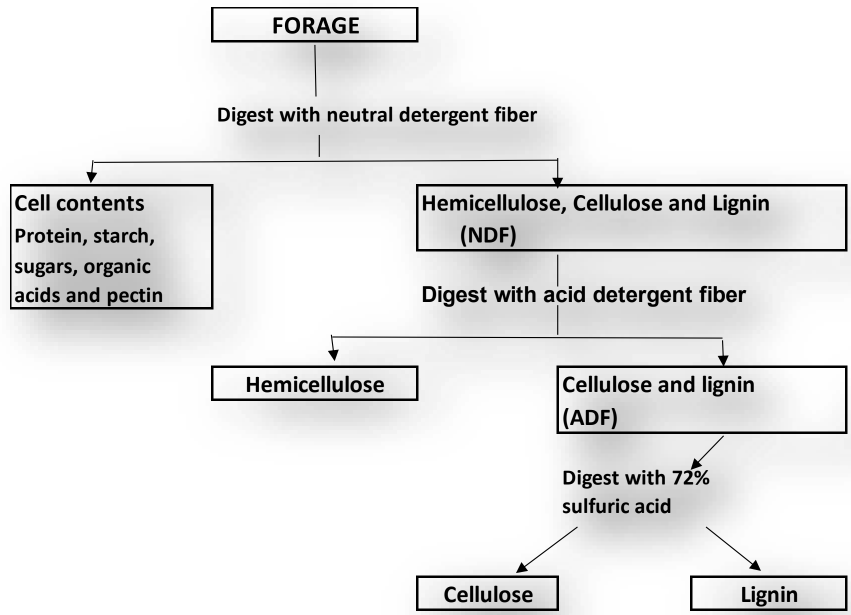


Figure IV.2. ADF and NDF fractions of forages.

3.4.3. NDF-Digestibility (NDFD)

The NDF concentration of forages differs from 30% to 80% of dry matter (DM) along with a large variation for its digestibility (Bender et *al.*, 2016), from minus than 40% for highly lignified mature legumes to more than 90% for unlignified immature grass (Goeser and Combs, 2009). The NDFD is more variable than the digestibility of any other feed component and can extremely affect intake and milk production.

The characteristics of the plant material (species, maturity at harvest, etc.) and the animal consuming the fiber affect the fiber digestion (Combs, 2016). The evaluation of NDFD is particularly important for the formulation of ruminant diets. It allows to assess forage quality, predict diet digestibility, and select plant genotypes for breeding.

Forage NDFD can be measured in situ and in vitro. The in-situ method consists of placing forages in small Dacron bags and then inserted into the rumen of a cow through a ruminal cannula. The amount of NDF before ruminal incubation is compared to the amount of NDF remaining after ruminal incubation and NDFD is calculated (Hoffman, 2001). Whereas the in vitro technique involves the use of ruminal inoculum (Lopes et *al.*, 2015, Hall and Mertens, 2008, Hoffman, 2001).

Fiber digestibility is usually determined in laboratory with rumen inoculum obtained from cannulated cows (Chiaravalli et *al.*, 2019). Nevertheless, in recent years, fresh feces from ruminants have been investigated as alternative inoculum to substitute rumen fluid (Ramin et *al.*, 2015, Hughes et *al.*, 2012, Mauricio et *al.*, 2001).

In this context, Chiaravalli et *al.* (2019) estimated the NDFD (48 h) of several feed ingredients using a rumen inoculum in comparison with fecal inoculum where the NDFD results obtained with rumen fluid were always higher than those obtained with fecal inoculum confirming a lower activity of the fecal inoculum compared with rumen fluid. But despite the differences obtained, results showed that fecal inoculum could replace rumen fluid at longer incubation times.

3.5. Minerals: Function's deficiencies and excess

Alongside to the elements in organic matter (oxygen, nitrogen, carbon, and hydrogen), seven major and nine minor minerals are considered dietary essentials for livestock. The major minerals required in relatively large amounts (calcium, phosphorus, sodium, chlorine, magnesium, potassium, and sulfur) and minor or trace minerals, that could be fed in small amounts (iron, iodine, copper, molybdenum, zinc, manganese, cobalt, selenium, and fluorine). The mineral contents of forages depend on a complex interrelationship of soil, climate, topography, plant species, its age and yield, pasture or forage management and cropping patterns (McDowell and Arthington, 2005).

Many times, the requirement of trace minerals is expressed as parts per million (ppm) or as milligrams per kilogram (mg kg^{-1}) of diet dry matter while requirements of major elements are expressed as percentage of dry matter (% DM).

Other elements are possibly essential but at extremely low levels (chromium, nickel, vanadium, silicon, tin, and arsenic). Most of these essential elements occur naturally in feedstuffs with amounts that do not constitute problems in nutrition. However, some major ones, could be sufficiently low to reduce productivity whereas trace minerals particularly can be present in toxic amounts (NRC, 1981).

Herbivores animals under natural grazing conditions obtain their minerals from forage plants. Inadequate mineral intake leads to reduced productivity (Ghazanfar *et al.*, 2011). Though, deficiencies, imbalances and toxicity of certain mineral elements may cause reproductive disorders as minerals play an important role in health and reproduction of the livestock (Sharma et *al.*, 2007).

3.5.1. Major minerals

3.5.1.1. Functions

The major minerals are required in gram quantities, they are important structural components of bone and other tissues and serve as important constituents of body fluids. They play vital roles in the maintenance of acid-base balance, osmotic pressure, membrane electric potential and nervous transmission (NRC,1981). Calcium, phosphorus, and common salt represent the most important minerals (Ghazanfar et *al*., 2011).

3.5.1.1.1. Calcium

The calcium is essential for formation of skeletal tissues, transmission of nervous tissue impulses, excitation of skeletal and cardiac muscle contraction, blood clotting, and as a component of milk. furthermore, it is involved in the activity of a wide array of enzymes and serves as an important second messenger conveying information from the surface of the cell to the interior of the cell.

Approximately 98 % of the calcium in the body is in the skeleton where calcium, along with phosphate anion, provide the structural strength and hardness to bone. The other 2 % of the calcium in the body is in the extracellular fluids of the body. When dietary calcium is insufficient to meet the requirements of the animal, calcium will be withdrawn from bone to maintain a normal concentration of extracellular calcium (NRC, 2001).

3.5.1.1.2. Phosphorus

Phosphorus has the most known biologic functions. Approximatively 80 % of phosphorus in the body is in bones and teeth. It is present in bone, along with calcium, principally as apatite salts, and as calcium phosphate. It is found in every cell of the body and almost all energy transactions involve formation or breaking of high-energy bonds that link oxides of phosphate to carbon or to carbon-nitrogen compounds (such as adenosine triphosphate, ATP) (NRC, 2001).

Phosphorus is intimately involved in acid-base buffer systems of blood and other bodily fluids, in cell differentiation, and is part of cell walls and cell contents as phospholipids, phosphoproteins, and nucleic acids (NRC, 2001). Phosphorus also is required by ruminal microorganisms for digestion of cellulose and synthesis of microbial protein (NRC, 2001). For livestock, it is required for normal milk production, growth, and efficient use of feed (Murugan et *al*., 2017). Also is declared to be one of significant element for normal sexual behavior (Sathish Kumar, 2003).

Phosphorus requirements for livestock are calculated by summing up the P retained by the animal, P lost in animal products (conceptus and milk) and endogenous P losses (feces and urine) (Cohen, 1976, NRC, 2001).

3.5.1.1.3. Potassium

Potassium is the third most abundant mineral element in the body. It must be supplied daily in the diet because there is little storage in the body and the animal's requirement for potassium is highest of all the mineral element cations (NRC, 2001).

Potassium is involved in osmotic pressure and acid-base regulation, water balance, nerve impulse transmission, muscle contraction, oxygen, and carbon dioxide transport; in phosphorylation of creatine, pyruvate kinase activity, as an activator or co-factor in many enzymatic reactions, in cellular uptake of amino acids and synthesis of protein, carbohydrate metabolism, and in maintenance of normal cardiac and renal tissue (NRC, 1980).

3.5.1.1.4. Magnesium

Magnesium is a necessary cofactor for enzymatic reactions vital to every major metabolic pathway but also is play a vital role to normal nerve conduction, muscle function, and bone mineral formation (NRC, 2001). Magnesium act at three biochemical levels, as a cofactor at the enzymatic level, at the structural level in assembly of ribosomes, and at the whole cell level as a stabilizing force in membranes (Fontenot et *al*., 1989).

Magnesium influences the activity of further than 300 cellular enzymes that are involved in energy metabolism, protein synthesis, cell growth and reproduction, synthesis of DNA and RNA and stabilization of mitochondrial membranes (Shills 1997, Ryan 1991, Aikawa 1981). In ruminants, the amount of Mg available for metabolism depends on Mg intake and the amount of Mg absorbed from the gastro-intestinal tract (Schonewille, 2013).

3.5.1.2. Deficiencies symptom

Mineral deficiencies that occur naturally may vary from relatively harmless disturbances to acute deficiencies that cause death (Haag, 1951). The major mineral deficiencies and symptoms for ruminants (beef cattle, dairy cattle, sheep, and goats) are presented in Table IV.2.

Table IV.2. Symptoms of major mineral deficiencies for livestock

Class of animal	Ca	Mg	K	P
Beef cattle	Weak, soft bones that may be easily fractured. Signs include swollen, tender joints, enlargement of the ends of bones, an arched back, stiffness of the legs, and development of beads on the ribs.	Excitability, anorexia, hyperemia, convulsions, frothing at the mouth, profuse salivation, and calcification of soft tissue.	Reduced feed intake and weight gain, pica, rough hair coat, and muscular weakness.	Reduced growth, feed efficiency and milk production, decreased appetite, impaired reproduction, weak, and fragile bones.
Dairy cattle	Affected ovarian function, increased incidence of dystocia, retention of placenta, prolapse of uterus and delaying of uterine involution process and impaired immune function.	-	Marked decline in feed and water intake, reduced body weight and milk yield, pica, loss of hair glossiness, decreased pliability of the hide, lower concentrations of potassium in plasma and milk, and higher blood hematocrit	Decreased reproductive performance, impairment of normal sexual behavior, delayed onset of puberty, delayed sexual maturity, silent or irregular estrus in heifers, failure of estrus, inactive ovaries, and low conception rates.
Sheep	May result in tetany or precipitate an outbreak of urinary calculi in intact or castrated male sheep. Hypocalcemia, osteomalacia in lambs and osteoporosis in ewes.	Hypomagnesemia tetany may fall on its side with its legs alternately rigidly extended and relaxed. Frothing at the mouth and profuse salivation are evident, and death may occur. loss of appetite, hyperemia, and calcification of soft tissues.	Decreased feed intake and decreased live weight gain. Listlessness, stiffness, impaired response to sudden disturbances, convulsions, and death have also been reported.	Slow growth, deprived appetite, unthrifty appearance, listlessness, low level of phosphorus in the blood and development of rickets.
Goats	Reduced milk production	Hypomagnesemia tetany (grass tetany), reduced urinary excretion and milk production.	-	Slowed growth, unthrifty appearance, and occasionally a depraved appetite.

-: not available. The information mentioned in this table has been reported by Balamurugan (2017), NRC (2001) and NRC (1981).

3.5.1.3. Excess disorders

Feeding excessive dietary calcium is generally not associated with any specific toxicity. In excess calcium could interfere with trace mineral absorption (especially zinc) and replaces energy or protein the animal might better utilize for increased production. Whereas, a long-term feeding of excess phosphorus can cause problems of calcium metabolism, inducing excessive bone resorption and urinary calculi, secondary to the elevated concentrations of phosphorus in blood. Most often, this toxicity is complicated with low dietary calcium (NRC, 2001).

The dietary concentration of potassium that leads to toxicity is not well defined (Ward, 1966) that is unlikely to occur under natural conditions but could occur because of excess supplementation. As for the negative effects of diets high in magnesium, they are generally restricted to causing a reduction in feed intake and/or inducing an osmotic diarrhea (NRC, 2001).

3.5.2. Trace minerals

3.5.2.1. Functions

Trace minerals are required in milligram or microgram amounts, they are present in body tissues in extremely low concentrations and often serve as components of metalloenzymes and enzyme cofactors, or as components of hormones of the endocrine system (NRC, 2001).

Microminerals should be provided to livestock in optimum concentrations and according to requirements which vary during the rapid growth and development of the animal and the production cycle. The term “requirements” for minerals is difficult to establish and most estimates are founded on the minimum level needed to overcome a deficiency symptom and not essentially to promote productivity (Lopez-Alonso, 2012). For trace minerals, the concentration of one can have a great effect on the requirement of another. For example, copper requirements are greatly affected by the concentrations of antagonists such as molybdenum, sulfur, iron, and zinc.

3.5.2.1.1. Iron

Iron principally functions as a component of heme found in hemoglobin and myoglobin. Enzymes of the electron transport chain, cytochrome oxidase, ferredoxin, myeloperoxidase, catalase, and cofactor for the cytochrome P-450 enzymes (NRC, 2001). It has been shown that animals cannot utilize the iron in their rations properly unless a trace of copper also is present. (Haag, 1951). Iron deficiency is still a major problem in several segments of the livestock industry.

3.5.2.1.2. Manganese

As reported in the article published by Balamurugan et *al.* (2017). Manganese possesses a significant role in reproduction. It is linked to the function of the corpus luteum regarding its role as an enzyme cofactor. Also, is involved in the synthesis of cholesterol and sex hormones (Suttle, 2010). The manganese deficiency produces poor fertility problem both for female and male (Wilson, 1966). For females, it is responsible for silent and decrease conception rate, birth of deformed claves and abortions. As for males, the Mn deficiency leads to absence of impairment testicular growth (Masters et *al.*,1988), libido, decreased spermatozoa motility and reduced number of spermatozoa in ejaculate (Satish Kumar, 2003).

3.5.2.1.3. Zinc

Zinc is a component of many metalloenzymes such as copper-zinc superoxide dismutase, carbonic anhydrase, alcohol dehydrogenase, carboxypeptidase, alkaline phosphatase, and RNA polymerase, which affects metabolism of carbohydrates, proteins, lipids, and nucleic acids. Zinc also regulates calmodulin, protein kinase C, thyroid hormone binding, and inositol phosphate synthesis. it is further a component of thymosin (a hormone produced by thymic cells that regulates cell mediated immunity). This mineral has a significant role in the repair and maintenance of the uterine lining following parturition, speeding return to normal reproductive function and early return of postpartum estrus (Green et *al.*, 1998). In addition to a major role in the immune system and certain reproductive hormones.

3.5.2.2. Deficiencies symptoms

Trace mineral deficiencies occur more frequently than recognized by most livestock producers. The micromineral deficiencies and symptoms for ruminants (beef cattle, dairy cattle, sheep, and goats) are presented in Table IV.3.

3.5.2.3. Excess disorders

In general, to avoid mineral excess disorders, the concentration of minerals should be below the Maximum Tolerable Level (MTL) for major and even trace elements. The MTL for each element is defined as the dietary level, when fed for a limited period, will not impair animal performance, and should not produce unsafe residues in human food derived from the animals.

Excessive dietary iron can interfere with the absorption of other minerals, primarily copper and zinc. If absorption of dietary iron exceeds the binding capacity of transferrin and lactoferrin in blood and tissues, free iron may increase in tissues. Free iron is very reactive and can cause generation of reactive oxygen species, lipid peroxidation, and free radical production leading to ‘‘oxidative stress’’ increasing antioxidant requirements of the animal. Free iron also is required by bacteria for their growth and excessive dietary iron can contribute to bacterial infection. Iron toxicity is associated with diarrhea, reduced feed intake, and weight gain.

Manganese toxicity in ruminants is unlikely to occur, the negative effects began to appear when dietary manganese exceeded 1000 mg kg^{-1}, they are limited to reduced feed intake and growth. While extremely high levels of zinc have a negative effect on absorption and metabolism of copper.

Table IV.3. Symptoms of trace mineral deficiencies.

Class of animal	Zn	Mn	Fe
Beef cattle	Parakeratosis, inflamed nose and mouth with sub-mucosal hemorrhages, unthrifty appearance, a roughened hair coat and joint stiffness.	Reproductive disorders, delayed estrus, reduced fertility, abortions, and deformed young. Weak, shortened bones, impaired spermatogenesis, testicular, epididymal degeneration, sex hormone inadequacy, and eventual sterility.	Anemia and decreased growth.
Dairy cattle	Lowered feed intake. skin parakeratosis, hair loss, unthrifty appearance, stiffness of joints, teeth gnashing, retarded testicular growth and excessive salivation. reduced reproductive performance	Impaired growth, skeletal abnormalities, disturbed or impaired reproduction, and abnormalities of the newborn, Deformed calves at birth and lower conception rate.	Anemia, reduced gain, listlessness, inability to withstand circulatory strain, labored breathing after mild exercise, reduced appetite, decreased resistance to infection, blanching of visible mucous membranes and a pale color of the muscle meat
Sheep	Lack of appetite, reduced growth, slipping of wool, swelling around the eyes and hooves, excess salivation, general listlessness, impaired growth of testes and cessation of spermatogenesis.	Depressed or delayed estrus and poor conception rates.	Anemia sometimes occurs with lambs raised on slotted, wooden floors.
Goats	Reduced feed intake, weight loss, parakeratosis, stiffness of joints, excessive salivation, swelling of the feet and horny overgrowth, small testicles, and low libido.	Reluctance to walk, deformity of the forelegs, reduced reproductive efficiency, tarsal joint excrescences, leg deformities, and ataxia.	-

-: not available. The information mentioned in the table has been reported by Balamurugan (2017), NRC (2001), NRC (1981) and Berger (1987).

4. Literature review on the nineteen investigated weed species

Nineteen weed species were selected to be valorised from a forage value point of view. The species were selected from the total inventory realized in the first experimental part of this thesis. These species represent 20% of the total flora. The weed species are *Anagallis arvensis* L., *Aristolochia baetica* L., *Calendula arvensis* L., *Chrysanthemum coronarium* L., *Chrysanthemum segetum* L., *Convolvulus althaoides* L., *Echium vulgare* L., *Emex spinosa* (L.) Campd., *Fumaria capreolata* L., *Hedera helix* L., *Inula viscosa* L., *Malva sylvestris* L., *Medicago rugosa* Desr., *Oxalis pes-caprae* L., *Rubia peregrina* L., *Sideritis montana* L., *Sinapis arvensis* L. and *Withania frutescens* Pauquy.

In the literature, some studies, investigating the mineral and nutritive value of the selected weed species were found. While regarding *Aristolochia baetica* L., *Calendula arvensis* L., *Chrysanthemum segetum* L., and *Rubia peregrina* L., there was no data available for their mineral composition and nutritive value.

Overall, there were no previous studies investigating the mineral and the nutritional composition of *Anagallis arvensis* L. Nevertheless, few studies have been conducted on forage mixture (Carpino et *al*., 2003), fodder flora (Badshah and Hussain, 2011) or plants community (El-Shesheny et *al*., 2014), including this species. As cited by Shaheen et *al*. (2014), *A. arvensis* is a common species. The whole plant is usually grazed by all animal's type with a high palatability. While El-Shesheny et *al*. (2014) mentioned this same species as unpalatable.

In another study, Badshah and Hussain (2011) reported that *A. arvensis* is economically used as fodder in Pakistan. Some studies reported the toxicity of *A. arvensis* (Schneider, 1978, Lander, 1944, Hurst,1942). According to the experimentation conducted by Sultan et *al*. (2003), this species has been considered as potentially nephrotic and highly toxic to rats inducting several changes in the animal tissues.

For *Ch. coronarium* L., Akrout et *al*. (2010) suggested its potential use as fodder for livestock due to its high content of minerals, mainly Ca and K, thus satisfy the mineral requirements for animal feeding. Nothing that it contained some secondary metabolites which may reduce its palatability. *Ch. coronarium* is considered as classical edible herb in India. It also contains Fe and Zn and not contains heavy metals (Cd and Pd) (Bordoloi et *al*., 2016). In another study, Wills et *al*. (1984) reported that *Ch. coronarium* represents a traditional Chinese vegetable where leaf and stem are the parts consumed however the lower stem part is inedible. As mentioned by the same authors, this species contains interesting levels of β-carotene, Fe, K, Ca, and dietary fiber.

Ch. coronarium is usually grazed by animals in Mediterranean pastures where it is spontaneous species, it is also a common weed in grain cereals and in the cultivated area generally (Valente et *al.*, 2003, Sulas et *al.*, 1999). *Ch. coronarium* can be used as conserved forage for the dry and winter periods due to their great amounts of dry matter produced in spring (Valente et *al.*, 2003). The same authors reported that this species is appropriate for conservation through ensiling. Therefore the *Ch. coronarium* forage might be considered as an alternative or complement to the traditional Mediterranean forage resources.

Additionally, the results obtained by Sulas et *al.* (1999) showed the interest of introducing *Ch. coronarium* in forage chains. In the same study, this species presented an extremely high level of digestible cell content, a high nitrogen level during the leafy stage and a moderate level during the flowering stage. Generally, it was marked by a high nutritive value and palatability. Therefore, for this purpose, it could be considered as interesting alternative to the legumes to complete successfully diets based on annual grasses. Moreover, in the literature there was no data reporting the toxicity of *Ch. coronarium*.

Convolvulus althaaoides L. is a perennial climbing shrub growing in north coast habitats (Aly Ahmed et *al.*, 2014, Hassine et *al.*, 2014). Few studies were conducted on its mineral and nutritional composition, finding only that conducted by Aly Ahmed et *al.* (2014).

For *C. althaoides*, the results obtained by Aly Ahmed et *al.* (2014) indicated a higher moisture, organic matter, total and insoluble carbohydrates, and total lipid contents during the winter period comparing with summer. Also, it registered a higher amount of total nitrogen, protein, and lipid. While the inorganic matter (ash), acid soluble ash, acid insoluble ash, water soluble ash, water insoluble ash and crude fibers content recorded their maximum in stem and their minimum in leaf. In addition, the amount of soluble carbohydrates, total nitrogen and total protein was in maximum in flower and minimum in leaf.

On the whole, few studies have been found on the forage quality of *E. vulgare* L. In the study conducted by Ivana et *al.* (2018), this species was characterized by a relatively large content of K, Ca, and Mg. In the same investigation, the ethanol, ethyl acetate, chloroform, petroleum ether and acetone extracts of *E. vulgare* contain major elements (Na, Mg, K and Ca) and microelements

(Cr, Mn, Fe, Co, Ni, Cu, Zn, Se and Sn) at different concentrations among the extracts. Also *E. vulgare* possess a high phytosterol content (Nogala-Kalucka et *al.*, 2010).

Regarding the toxicity of this species, Simmonds et *al.* (2000) mentioned a moderate toxic risk for goats by *E. vulgare*. In addition to a toxicity to horses, cattle and to a lesser extent sheep. Specifying that the poisonous principle are pyrrolizidine alkaloids causing sometimes chronic weight loss, acute jaundice with or without photosensitization, or sudden death with no other signs. The same reference specified that goats and sheep can tolerate many months of ingestion, however the plant risk to be poisoned when eaten in significant amounts for more than one season.

For *Emex spinosa* (L.) Campd., Shaltout et *al.* (2008) reported that *E. spinosa* recorded a high amount of ash and contains lignin.

Regarding *Fumaria capreolata* L., Baillie et *al.* (2018) mentioned its composition of major elements (Ca, k, Mg, N, P and S) and microelements (Al, B, Cr, Cu, Fe, Mn, Mo, Ni, Pb and Zn), that turned out to be rich in minerals.

Malva sylvestris L., known as common mallow, is native to Europe, North Africa, and Asia (Gasparetto et *al.*, 2010). Mallow is used as medicinal or ornamental plant or as fodder (Ahmad et *al.*, 2010). *M. sylvestris* has been investigated by several scientists.

Tabarki et *al.* (2012) studied *M. sylvestris* for its proximate composition, fatty acids composition and mineral content. This study revealed that this species contained four major fatty acids (linolenic, linoleic, palmitic, and oleic acids) represented more than 82% of total fatty acids in leaves and petioles. These results suggested that *M. sylvestris* represent a potential resource for food and/or feed.

Leaves and immature fruits of *M. sylvestris* contain major elements (Na, K, Ca, Mg, P, and S) and microelements (Fe, Zn, Mn, and Cu). It had a rich chemical composition with high Na, Ca, and Mn levels (Romojaro et *al.*, 2013). Tunçtürk et *al.* (2018) also analyzed *M. sylvestris* for minerals and heavy metals constituents. Leaves, flowers, and seeds of *M. sylvestris* are used boiled, roasted, as salads or flavoring salads or as soups and stews (Civelek and Balkaya, 2013, Romojaro et *al.*, 2013, Bianco and Santamaria, 1998).

M. sylvestris has been also studied by Hiçsönmez et *al.* (2009) for major and minor elements where the high Ca and Mg concentrations were detected in their leaves. The same authors reported that the mineral contents (major and trace) of *M. sylvestris* were between the international safety limits both for human and animal consumption. Civelek and Balkaya, (2013) mentioned that *M. sylvestris* and others wild species have higher nutrient contents comparing with cultivated leafy vegetables reported in previous studies.

As reported by Barros et *al*. (2010), mallow flowers contained a high content in carbohydrates (particularly, sugars such as fructose and glucose) while the immature fruits had a low nutraceutical potential with high levels of total fats and saturated fatty acids. the high moisture content was detected in leafy flowered stems while the protein was obtained in low levels. As for fats, they were the less abundant macronutrients.

Alongside with others wild edible plants, *M. sylvestris* was considered as a good source of vitamin C and carotenes that could be used for nutritional purposes (Guil et *al*., 1997). Also, it contains tocopherols (vitamin E) (Gasparetto et *al*., 2010).

A good forage quality of *M. rugosa* Desr. have been reported by Porqueddu (2001). Beside others annual medics, *M. rugosa* represents an important feed resource as green forage during the growing season but also in summer as stubbles and pods. The P, Ca and S contents and the nutritive value (ash, CP, EE, CP, and N) were cited by Dougall and Bogdan, (1966). In addition, Porqueddu (2001) mentioned the CP, ADF and NDF contents.

O. pes-caprae L. known as cape sorrel, is a culinary herb (Van Wyk, 2011). Their tubers are consumed boiled, as salads or as flavoring salads (Bianco and Santamaria, 1998). The microelements (Ca, k, Mg, N, P and S) and microelements (Al, B, Cr, cu, Fe, Mn, Mo, Ni, Pb and Zn) were determined by Baillie et *al*. (2018). *O. pes-caprae* is rich in minerals and contains some heavy metals (Edrah et *al*., 2018). Romojaro et *al*. (2013) also analyzed cape sorrel for its mineral composition (K, Ca, Mg, Na, Fe and Zn) which was rich than some cultivated vegetables. However, they recommended it use in small quantities regarding its high oxalic acid content.

The toxicity of *O. pes-caprae* remained due to its high oxalate content. But the oxalate poisoning is a complex and unwell understood phenomenon. Symptoms and signs of poisoning differ with the animal species and the chemical form of the oxalate (James, 1987). For horses, Herbert and Dittmer (2017) reported a toxicity of *O. pes-caprae* which is associated with the acute and chronic hypocalcemia. While for ruminants, they possess mechanisms to degrade oxalates into relatively harmless substances such as formic acid and carbon dioxide (Allison and Cook, 1981). As for goats, there is a low toxic risk (Simmonds et *al*., 2000).

Sayed (1993) investigated *Reichardia tingitana* L., in addition to other species (Asteraceae and Fabaceae) for their nutritional value, which presented variable average amounts of water, dry matter, and nutrients. Gamal (2017) also analyzed the mineral and nutritive value composition of this species which remains good.

For *Sideritis montana* L., there was no data about its mineral nutritive value composition. The only study found reported its fatty acids composition, where palmitic acid, oleic acid and α-linolenic acid were the dominants while it contains the low levels of lipid-soluble vitamins (Emre et *al*., 2011).

Sinapis arvensis L., known also as wild mustard also has been investigated by several scientists. Lenzi et *al*. (2019) described the mineral composition (Ca, Mg, P, Fe, Cu, Zn and Mn) of *S. arvensis*. The different parts (leaves, flowers, stems, fruits and seeds) of wild mustards were investigated separately for their ion composition (F, Cl, Na, K, Mg, Ca, NH_4, PO_4, SO_4) by Başyiğit et al. (2020). The results obtained revealed its rich composition.

Leaves and flowers of *S. arvensis* are culinary used boiled, as salads, seasoning, savory or rustic cakes (Disciglio et *al*., 2017, Bianco and Santamaria, 1998). Disciglio et *al*. (2017) analyzed some wild edible plant species in addition to *S. arvensis* which recorded good amounts of minerals.

Wild mustard is considered as a non-conventional vegetable, usually prepared by family farmers in rural communities. De Olivieira et *al*. (2019) analyzed the nutritive value (moisture, ash, protein, lipids, and carbohydrates) and mineral composition (P, K, Ca, Mg, Cu, Fe, Zn and Mn) of this species raw and after cooking. It recorded excellent nutritional value with a high content of Fe, P, Mn, and Mg. In addition, it was highlighted that the lipids amount, carotenoids, vitamin E and phenolic compounds increased after cooking.

In another study, Kamalak et *al*. (2005) determined the nutritive value of *S. arvensis* at different maturity stages. It presented a high forage quality for ruminants, where it was recommended to be harvested at early flowering stage when it contains a higher amount of protein but low fiber content resulting in higher digestibility and metabolize energy to obtain higher quality forage.

5. Conclusion

Forages, either harvested mechanically or by grazing, are the basal dietary ingredients for beef cattle, dairy cattle, sheep, and goats. The nutritive value of forages depends on different factors (species, edaphic and climatic conditions, stage of maturity, etc.).

Many livestock species use forages (either harvested mechanically or by grazing) as their primary source of nutrition. For ruminants, forages represent the primary source of nutrition.

Nutrient requirements differ from one species to another, but they all need adequate amounts of each nutrient for healthy bodily functions.

The previous investigations reported in the literature revealed the potential interest of weeds, which presented a good nutritional value and therefore require more research on their mineral composition and nutritive value. Weeds can be used to feed animals (fresh or stored) as alternative source.

Additionally, given their chemical composition, which is rich in mineral elements, the well-managed weeds could be used in biofertilization by their incorporation into the soil. This improves the organic matter rate, the activity of the pedo-fauna, the quality of the soil and at the same time the production. Moreover, reducing the chemical inputs, their resulting costs and their impact on the environment and the health.

In the corresponding experimental trial part, we studied 19 weed species (selected from the floristic inventory established in the first experimental part), for their mineral composition and nutritive value. This experimental part aimed to the valorization of weeds that composed the citrus weed flora to be used to feed animals (ruminants).

Section 2

Experimental Trials

<u>Chapter V</u>

Impact of mechanical and chemical weeding on the floristic diversity of citrus orchards in Tlemcen (Northwestern Algeria)

One longs for a weed here and there, for variety….

"A Fable for Critics" (Lowell, 1848)

Experimental Trials

Chapter V

Impact of mechanical and chemical weeding on the floristic diversity of citrus orchards in Tlemcen (Northwestern Algeria)

Contents

▪ **Abstract:** This study was conducted to evaluate the impact of weeding (chemical, mechanical and control) on the floristic composition of citrus orchards in the region of Tlemcen (Northwestern Algeria). A comparative approach between two methods of weeding (mechanical *vs.* chemical) compared to a control (without weeding) was carried out at 3 stations of 100 m^2 inside the citrus orchard studied. The floristic surveys were performed in the stations at different times (before and after each weeding). In the floristic inventory, 168 surveys were carried out and a total of 88 species were identified belonging to 71 genera and 30 botanical families. This adventitious flora was dominated by the Mediterranean elements (64%), the therophytes (51%) and dicotyledons (64%). Statistical elaboration of floristic data by means of multifactorial ANOVA revealed the existence of a significant spatial-temporal difference in the mean species richness between the stations (control *vs.* chemical and mechanical). In addition, the species richness before weeding tended to be higher than that after it. Weeding methods practiced by crop growers need to be reconsidered. The benefits that weeds can provide should be considered as well as the damage caused by the different weeding techniques.
Keywords: Biodiversity, weeds, weeding, citrus, Algeria.
▪ **Résumé :** Cette étude a été réalisée afin d'évaluer l'impact du mode de désherbage (chimique, mécanique *vs.* contrôle) sur la composition floristique des vergers d'agrumes dans la région de Tlemcen (Nord-ouest de l'Algérie). Une approche comparative entre deux méthodes de désherbage (mécanique *vs.* chimique) par rapport à un témoin (sans désherbage) a été réalisée dans 3 stations de 100 m^2 à l'intérieur du verger d'agrumes d'étude. Les relevés floristiques ont été effectués dans les stations dans deux temps (avant et après chaque désherbage). Un total de 168 relevés a été effectué. La flore adventice du verger d'étude se compose de 88 espèces, appartenant à 71 genres et 30 familles botaniques. Cette flore adventice était dominée par les éléments méditerranéens (64%), les thérophytes (51%) et les dicotylédones (64%). Une analyse statistique multifactorielle des données par ANOVA a révélé l'existence d'une différence spatio-temporelle significative de richesse moyenne en espèces entre les différentes stations (contrôle, chimique et mécanique). En addition la richesse moyenne en espèces avant le désherbage avait tendance à être plus élevé que celui après désherbage. Les méthodes de désherbage pratiquées par les agriculteurs doivent être reconsidérées. Les avantages que les adventices peuvent apporter doivent être pris en considération ainsi que les dégâts causés par les différentes techniques de désherbage.
Mots-clés : Biodiversité, adventices, désherbage, agrumes, Algérie.
▪ **ملخص :**أجريت هذه الدراسة لتقييم تأثير طريقة إزالة الأعشاب الضارة (الكيميائية والميكانيكية مقارنة بشاهد) على التنوع النباتي لبساتين الحمضيات في منطقة تلمسان (شمال غرب الجزائر). أقيمت مقارنة بين طريقتين لإزالة الأعشاب الضارة (الميكانيكية مقابل الكيميائية) مقارنة بشاهد (بدون أي طريقة لإزالة الأعشاب الضارة) في ثلاث رقعات بمساحة 100 متر مربع داخل بستان الحمضيات المدروس. تم إجراء المسوحات داخل البستان في أوقات مختلفة (قبل و بعد إزالة الأعشاب الضارة) ؛ حيث تم إجراء 188 مسحا وتم تحديد 88 نوعا نباتيا ينتمي إلى 71 جنسا و 30 عائلة نباتية. النباتات ذات التوزيع الجغرافي من البحر الأبيض المتوسط (64 %) و النباتات الحولية (51%) و ثنائيات الفلقة (64%) كانت هي السائدة. كشف التحليل الإحصائي المتعدد العوامل عن طريق تحليل التباين، عن وجود اختلاف مكاني زماني كبير في متوسط ثراء الأنواع بين المحطات (الكيميائي مقابل الميكانيكي) بالإضافة إلي ذالك كان ثراء الأنواع قبل إزالة الأعشاب الضارة يميل إلى أن يكون أعلى من ذلك بعده . يجب إعادة النظر في طرق إزالة الأعشاب الضارة التي يقوم بها الفلاحون ، و لا سيما الفوائد التي يمكن أن توفرها الأعشاب الضارة، يجب أن تؤخذ في عين الإعتبار و كذلك الأضرار التي تخلفها مختلف تقنيات إزالة الأعشاب الضارة.
الكلمات المفتاحية : التنوع البيولوجي، الأعشاب الضارة ، طرق إزالة الأعشاب الضارة ، الحمضيات ، الجزائر.

1. Introduction

The presence of weeds in cultivated crops can affect the production and quality of the crop by competing for light, moisture, nutrients, and space (Ashenafi and Dalga, 2014), in addition to the technical problems associated with their presence (Labreuche et *al*., 2014. The problems caused by weeds are also related to the evolution of the adventitious flora under the pressure of cultural techniques (Maillet, 1981).

High crop productivity requires control and proper use of weeds (Siyahpoosh et *al.*, 2012). Mechanical weeding is the classic method to control weeds but in modern agriculture, chemical weeding of major crops has become a routine part of cropping techniques. Various socio-economic and environmental factors require an increased rationalization of chemical weed control to avoid reliance on unproductive treatments. At the plot scale, the criteria for any weed control program should be the risk of weed damage to crop plants and potential damage to the harvest. Any weed control program must be part of a technical farming itinerary and be planned in economic terms, whereas the weed nuisance is evaluated using experimental data measured in population biology in an artificially created environment.

A farmer is advised to manage weeds for useful purposes as service plants (Siyahpoosh et *al*., 2012). The repeated use of chemical weeding and of mechanical weeding provokes resistance in the weeds and considerably disturbs the role of the pedo-fauna in biofertilization (Labreuche et *al*., 2014). Thus, weed control in recent decades has led to the intensive use of herbicides that cause serious pollution problems and damage to biodiversity (Zhang et *al*., 2016). Chemical fertilizers are the main factors in maintaining soil fertility, but greater application with inadequate management reduces the availability of soil organic matter (Galal and Shehata, 2015) and reduces the number of weeds (Franke et *al*., 2009). In the face of this degradation of the soil and plant species, many developing countries need to rethink their management methods. They must restrict the use of herbicides and promote agroecosystems (Olesen et *al*., 2004).

Good management of the adventitious flora certainly depends on good knowledge of the flora and its member species. For this purpose, several studies have been conducted in different regions in Algeria (Abdelkrim, 2004, Hannachi and Fenni, 2013 and Kazi Tani et *al*., 2010). In general, the presence of a weed has both an ecological (soil and climate) and an agronomic (cultural practices) dimension. It is through the change of this environment that it is possible to quantify the impacts on agriculture (Fried et *al*., 2008). Changes on agricultural practices influence the composition and evolution of weed communities (Chafik et *al*., 2013a, Fried and Reboud, 2008). The impact of humans on the weed communities causes some characteristic species to disappear from poor environments (oligo-trophiles) in favor of nitrophilic species. Specialist species also disappear in relation to generalist species and the massive decline in species and species diversity affects even common species (Fried, 2010).

Several management strategies have been developed. These include that of Bertrand and Doré (2008), who discussed the control of weed flora through an integrated production system; Chauvel et *al*. (2011), who also studied the integrated management of weed flora, which greatly limited the use of herbicide treatments, thus contributing to the control of the seed stock of weeds; and Quennesson and Oste (2017) who have worked on weed management through soil cover by exploiting allelopathy and plant competition relationships for resources.

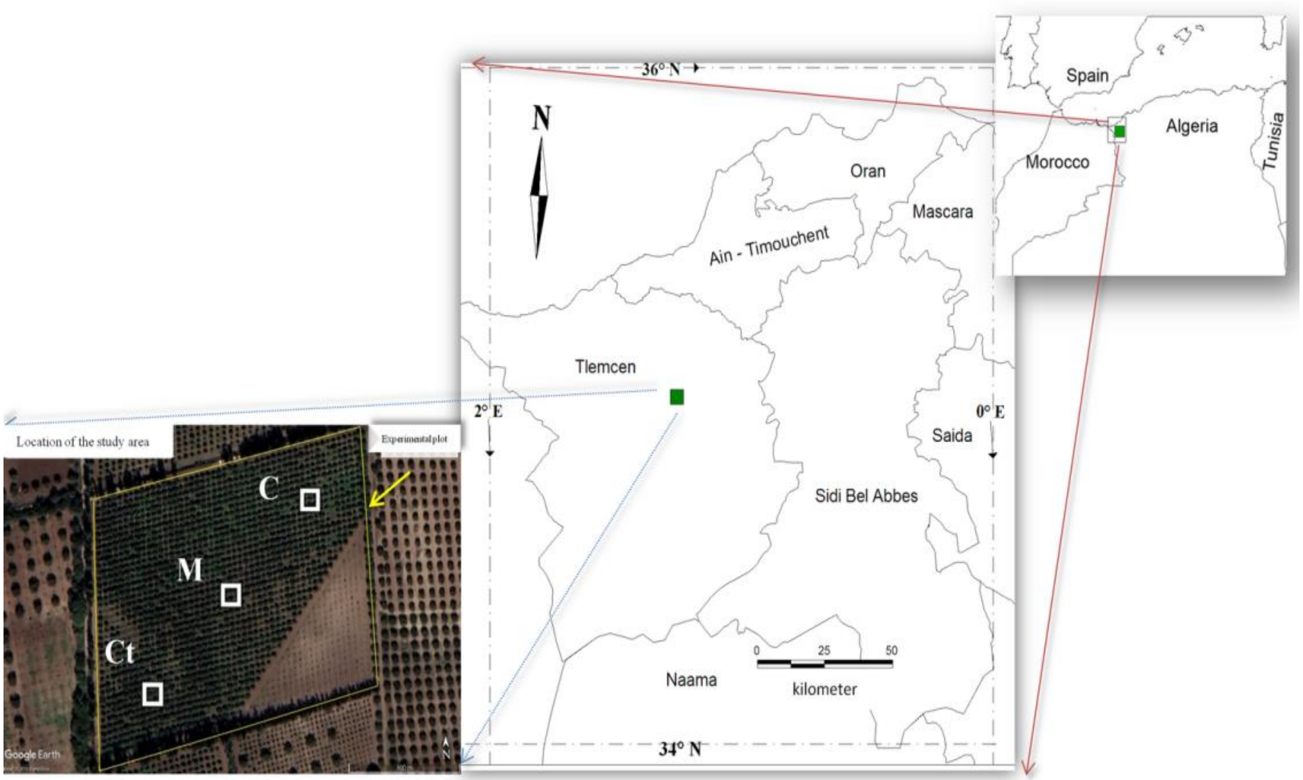
In contrast, many studies indicate that weed diversity can have positive impacts on the functioning of agroecosystems (Franke et *al*., 2009, Albrecht, 2003, Norris and Kogan, 2005). In addition, weeds can have beneficial properties on the biodiversity index and can also be a host to auxiliaries (Colbach et *al*., 2014). Chemical and mechanical weeding can affect the floristic diversity and they can decrease the species richness after each weeding. The point is not only that mechanical weeding favors therophytes and chemical favors geophytes.

The objective of this investigation was to study the adventitious flora associated with citrus orchards by comparing the impacts of mechanical and chemical weeding on their floristic composition compared to a control without weeding.

2. Materials and methods

2.1. Presentation of the study area

Located at latitude 34°58′19′′N, longitude 0°20′36′′W and altitude 364 m (Figure V.1), the area under study is part of the plain of Hennaya in the agricultural basin of the region of Tlemcen (Northwestern Algeria) where citrus cultivation predominates. The climate of this region is Mediterranean, semi-arid with moderate winter under a semi-continental influence and with a seasonal regime of precipitation of the WSPAS type (Winter, spring, autumn and summer).



Legend: (C: Chemical weeding, M: Mechanical weeding, Ct: Control).

Figure V.1. Location of the experimental area.

2.2. Presentation of the orchard studied

Table V.1 describes the studied orchard.

Table V.1. Description of the studied orchard (Hennaya, Tlemcen-Algeria).

Statue of Land holding	Private family farm	**Irrigation mode**	Drip irrigation
Age of the orchard	14 years	**Origin of irrigation water**	Oued Sekkak
Area of the orchard	4 hectares	**Types of fertilizing**	Mineral and Organic
Density of plantation	400 trees/hectare	**Phytosanitary treatments**	Insecticide, Fungicide, and acaricide
Row spacing	5 meters	**Weeding mode**	Mechanical weeding
Altitude	400 meters	**Product destination**	Regional market
Cultivated variety	Thomson	**Soil texture**	Loamy balanced

2.2.1. Soil analyzes

Soil sampling was realized in each plot (Ct, C and M) (Figure V.2) and then transported to the laboratory for analyses. The color of the soil was determined using the MUNSELL code. The granulometric composition was determined using the method cited by Casagrande (1934). As for the stature, the texture triangle was used. The pH was measured by an electrode pH meter. For the determination of the limestone, the calcimeter of Bernard was used, which allows to measure the volume of CO_2 released by the action of hydrochloric acid (HCL) on the calcium carbonate $CaCO_3$ of a sample.

Organic carbon assay was done using the Tjurin method which consists in knowing the quantity of potassium dichromate which will oxidize the carbon of the organic matter in sulfuric acid medium. The electrical conductivity (EC) was measured on an aqueous extraction solution using a conductivity meter. The results were expressed in millisiemens per centimeter ($mS.cm^{-1}$).

(Mechanical plot) (Chemical plot)

Figure V.2. Soil sampling.

2.3. Experimental protocol

The experimental approach was designed to compare two weed control methods against a control without weeding (Figure V.3). According to the minimal area recommended by Sorensen (1948) to study perennial crops, three stations of 100 m^2 were chosen inside the experimental plot located in a citrus orchard (Figure V.1). Stations were subjected to mechanical weeding (M = y: 34.972006°, x: -1.343233°), chemical weeding (C = y: 34.972580°N, x: 1.342573°W) and control without weeding (Ct = y: 34.971189ºN, x: 1.343983ºW), respectively.

Figure V.3. Experimental plots inside the citrus orchard.

The surveys were carried out in each station according to the method described by Braun-Blanquet (1951) and Guinochet (1973), seasonally (autumn and spring). The quadrat technique consists in placing a square of 1 m^2 (Figure V.4). All the species present inside this square were sampled to be identified and each species encountered was assigned a presence-absence index.

Figure V.4. Quadrat technique (square of 1 m^2).

A total of 168 surveys were conducted during the period from August 2016 to September 2017. Mechanical weeding was done by a mechanical weeder (Figure V.5) (an agricultural machine used for removing weeds from an area). For chemical weeding, the product used is a total, systemic, and non-selective herbicide (Figure V.6) in the form of a water-soluble concentrated liquid, in which the active substance is glyphosate™ as isopropyl amine salt (IPA) 48% w/v. The treatment was carried out using a knapsack sprayer. The treatments were carried out in two periods, the first in autumn (October) and the second in spring (April).

Figure V.5. Machine used for the mechanical weeding

(Total herbicide: glyphosate™) (Knapsack sprayer)

Figure V.6. Product and material used for the chemical weeding

2.4. Systematic approach

New flora of Algeria and southern desert regions (Quézel and Santa, 1963), the "portative flora" of France, Switzerland, and Belgium (Bonnier and De, 1948), flora and vegetation of the Sahara (Ozenda, 1991) and the flora of cultivated fields (Jauzein, 2011) were used for the identification (keys identification), chorology determination and biological spectrum of inventoried species.

2.5.Analyze of floristic similarity (Jaccard's Index)

In order to compare species composition of the three plots (Ct, M and C) of the citrus orchard studied, floristic similarities based on binary (presence-absence) data between pairs of plots were calculated by using Jaccard's similarity index. Jaccard's Index (JI) take into consideration the presence or absence of species. It allows to show similarities or differences that exhibit the biggest effect on the repartition of species between different stations.

This coefficient is the ratio expressed as a percentage between the species common to the two stations, and the total number of species present in each station. It is expressed as follows:

$J (\%) = [(a/ (a + b + c)) \times 100]$, where:

- a: represent the number of species common between two habitats.
- b: represent the number of species unique for the habitat 1.

- c: represent the number of species unique for the habitat 2.

2.6. Frequency of occurrence of species

The frequency of occurrence of the weed species that composed the floristic inventory of the studied orchard was determined. Bigot and Bodot (1971) distinguished four categories of species according to their Constance:

- Constant species, present in 50% or more in the realized surveys.
- Accessory species, present between 25 à 49% in the surveys.
- Accidental species with occurrence frequency varying between 12.5 to 24%.
- Very accidental or sporadic species with a frequency inferior to 12.5%.

2.7. Statistical analysis

In order to evaluate the impact of the method of weeding on the weed flora, the collected data were statistically processed (ANOVA) using the R software by considering two factors: treatments (plots) (n = 3; chemical, mechanical *vs.* control) and time of the treatment (n = 2; before *vs.* after weeding) and the interactions between factors, using the following model: $Y_{ijk}=\mu+\alpha_i+\beta_j+(\alpha\beta)_{ij}+\varepsilon_{ijk}$, where μ=average, αi= time of the treatment (n = 2), β_j= treatments (n = 3), $(\alpha\beta)_{ij}$= interactions between factors. Each species was codified by the first three letters of the genus, the first two letters of the species, and a number was assigned to each floristic survey.

3. Results and discussion

3.1. Soil analysis results

Table V.2 provides results of the soil analyses. The dominant soils were brown, with a loamy to sandy-loamy texture, balanced, low in organic matter, and slightly alkaline with a low P_2O_5 reserve.

Table V.2. Soil analyses of the studied orchard.

Stational characteristics	Sample 1	Sample 2	Sample 3
Geographic location	Sekkak (Hennaya)	Sekkak (Hennaya)	Sekkak (Hennaya)
Altitude (m)	400	400	400
Exposition	Null	Null	Null
Slope	Null	Null	Null
Depth of horizons (cm)	0-30	0-30	0-30
Color according to MUNSELL	5 Y/R à 7,5 Y/R 6/2	10 Y/R 5/4	10 Y/R 5/3
Color	Gray-beige to beige-gray	Yellow brown	Brown
Granulometry			
Sand	40	39	44
Loam	32	38	34
Clays	28	23	22
Classification	Loamy	Loamy	Sandy-loamy
Type of texture	Balanced	Balanced	Balanced
Organic matter			
Cox (%)	1.23	1.43	1.52
Humus	0.89	1.05	1.11
Quantity	Low	Medium	Medium
Structuration factor	20.5	29.3	31.1
Solution of soil			
pH to H_2O	7.85	7.79	7.83
Classification	Little alkaline	Little alkaline	Little alkaline
Carbonate ($Ca CO_3$) (%)	22.9	20.1	22.3
Quantity	Medium	Medium	Medium
Salinity and electric conductivity			
EC ($mS.cm^{-1}$)	0.14	0.12	0.09
Dry residue of dry soil (%)	0.10	0.15	0.16
Degree of salinity	Unsalted	Unsalted	Unsalted
Phosphor			
P_2O_5 (mg 100 g^{-1})	0,87	0,79	0,63
Reserve in P_2O_5	Low	Low	Low

Cox: oxidized carbon.

3.2. Floristic composition

From the 168 floristic surveys conducted in citrus orchards, 88 species have been inventoried. All the species encountered are angiosperms, including 8 families, 23 genera and 32 monocotyledonous species. There are 22 families, 48 genera and 56 dicotyledonous species (Figure V.7).

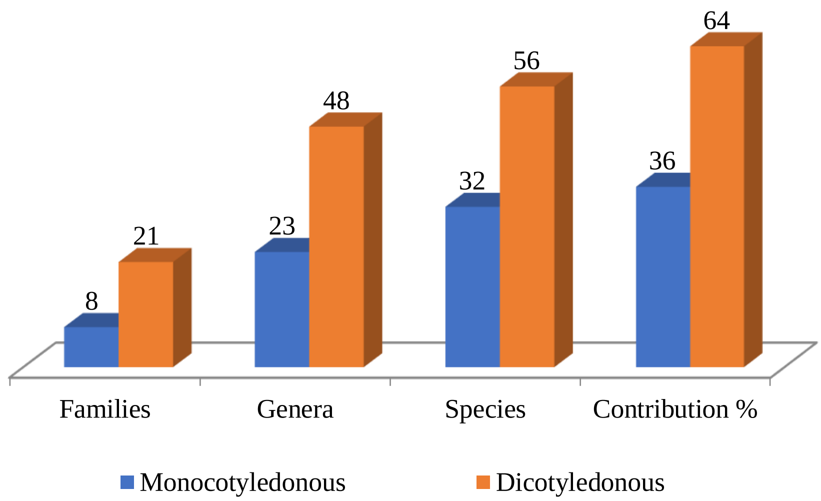


Figure V.7. Floristic composition of the experimental area.

The 88 species registered in this inventory belong to 71 genera and 30 botanical families. These families account for 73% of the weed flora of phytogeographic crops in Oranais (Kazi Tani et *al*., 2010) and 76% of the adventitious agro-systems flora of eastern Morocco (Chafik et *al*., 2013b). Poaceae, Asteraceae, Apiaceae and Fabaceae were the dominant families (Table V.3). These four botanical families alone represented 55% of all the species encountered. Some families were accidentally encountered such as Palmaceae, Oleaceae, Moraceae and Orchideae (Table V.3).

3.3. Ethological spectrum

Raunkiaer (1905, 1934) proposed the bases for a biological classification of plants. The criterion of bud position during the most unfavorable season to the plant remains in the case of weeds the basic criterion of a biological classification (Jauzein, 2011). Figure V.8 shows the ethological spectrum established for all species. Therophytes are the majority and represent 51% of the inventoried flora. This contribution is lower than that previously established at 69% for citrus flora in eastern Morocco (Chafik et *al*., 2013a) and for that reported by Kazi Tani et *al*., (2010) at 75%, respectively. Despite the importance of therophytes, hemicryptophytes occupy an important place (22%) and geophytes rank third with a contribution of 17% of the total inventoried species. The phanerophytes, chamaephytes and nanophanerophytes represent 5%, 3% and 2%, respectively (Figure V.8).

Table V.3. Total distribution of botanical families according to their specific contribution.

Botanical families	Genera	Species	Contribution (%)
Poaceae	12	19	21.59
Asteraceae	10	14	15.9
Apiaceae	9	9	10.22
Fabaceae	6	6	6.81
Liliaceae	4	4	4.54
Chenopodiaceae	2	3	3.4
Cyperaceae	1	3	3.4
Convolvulaceae	1	2	2.27
Malvaceae	1	2	2.27
Orchideae *	2	2	2.27
Polygonaceae	2	2	2.27
Anacardiaceae	1	2	2.27
Labiateae	2	2	2.27
Solanaceae	2	2	2.27
Amaranthaceae	1	1	1.13
Primulaceae	1	1	1.13
Araceae	1	1	1.13
Aristolochiaceae	1	1	1.13
Boraginaceae	1	1	1.13
Geraniaceae	1	1	1.13
Moraceae *	1	1	1.13
Fumariaceae	1	1	1.13
Iridaceae	1	1	1.13
Araliaceae	1	1	1.13
Oleaceae *	1	1	1.13
Oxalidaceae	1	1	1.13
Portulacaceae	1	1	1.13
Rubiaceae	1	1	1.13
Brassicaceae	1	1	1.13
Palmaceae *	1	1	1.13

*: Accidental families.

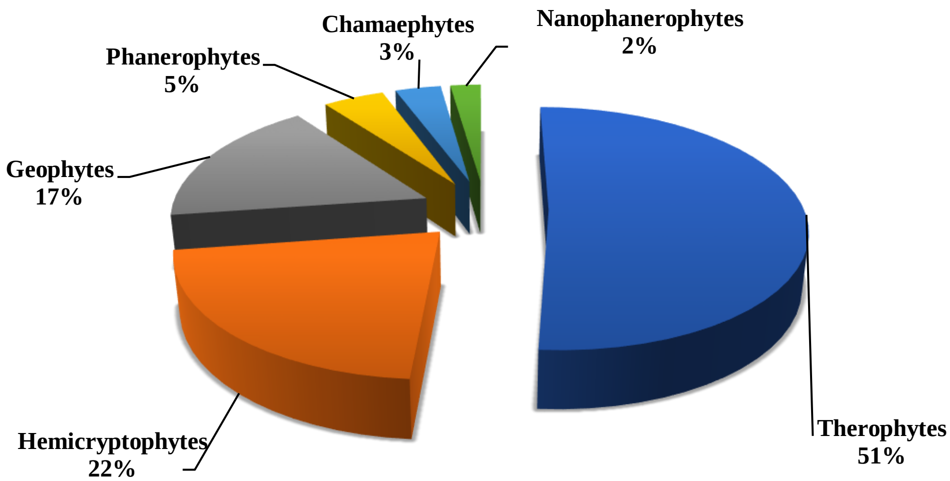


Figure V.8. Global ethological spectrum of weeds.

In the same context, the perturbation index calculated by Loisel and Gamila (1993) allows the researcher to quantify the therophytization of a medium by the following formula:

PI= [(Number of Chamaephytes + Number of Therophytes)/Total number of species]

For the experimental area, the perturbation index is of the order of 54.54%. Therefore, the experimental area is almost half conserved, tending to a medium still in equilibrium with the biotic and abiotic conditions of the environment.

3.4. Biogeographic distribution

The phytogeographic study is an essential basis for any attempt to conserve biodiversity (Quézel et *al*., 1991). It represents a true model of interpretation of the phenomena of regressions (Olivier et *al*., 1995). Regarding the chorology, the weed flora of Tlemcen citrus groves consists of heterogeneous elements of various origins: Mediterranean, northern, and southern (Table V.4).

Species with Mediterranean distribution *sensulato* (including bi-regional and tri-regional) have a contribution of 64% (Table V.4). This rate is close to that obtained in a previous study for weed communities of phytogeographic crops Oranais in which 58% of the taxa are Mediterranean (Kazi Tani et *al*., 2010), 52% for perennial crops in the Mitidja plain (Kheddam and Adane, 1996), 59% for the weeds of eastern Morocco (Chafik et *al*., 2013b), and 62% for the weed flora of western Morocco agrosystems (Zidane et *al*., 2010). As for the endemic elements of North Africa, they are represented by a single species

(*Pistacia atlantica* Desf.), which accounts 1.13% of the total flora. However, for Algerian endemics, there is also a single species (*Hedysarum naudinianum* Coss.).

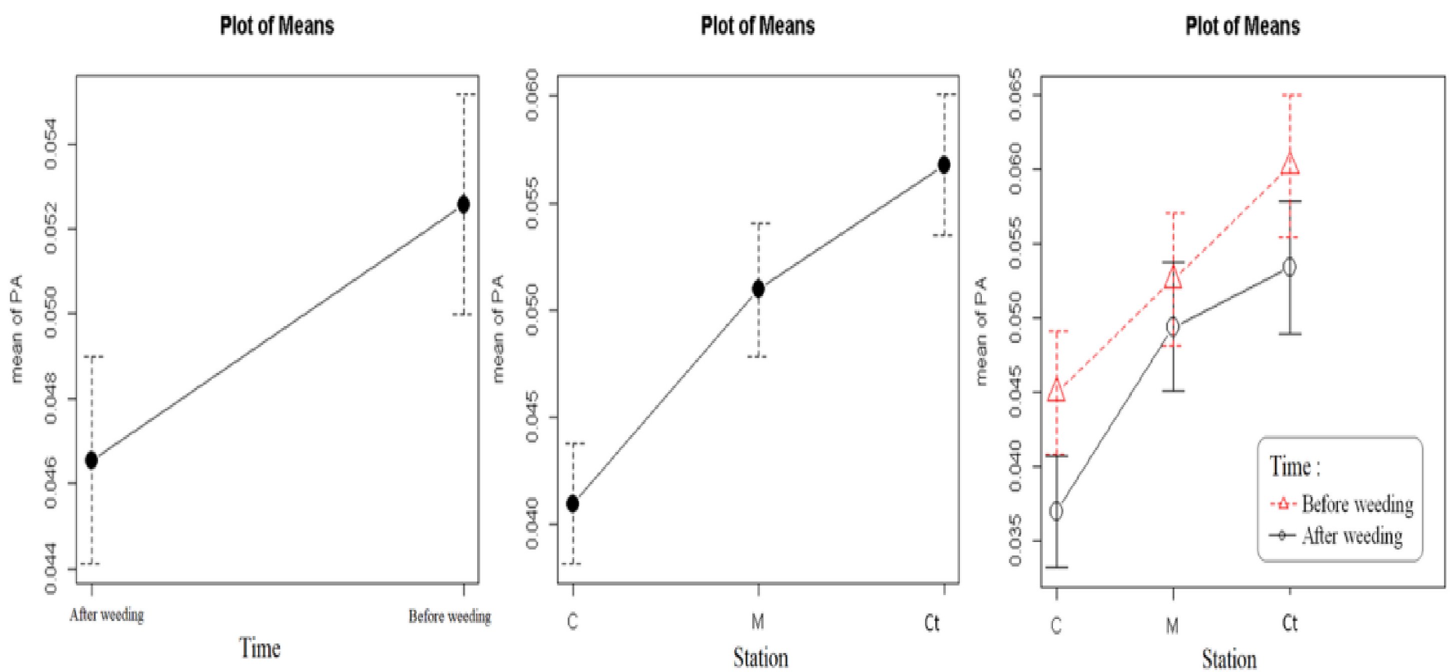
Table V.4. Biogeographic origin of weeds and their distribution.

Geographic origin	Number of species	Contribution (%)
Mediterranean	30	34.09
Cosmopolitan	6	6.81
European-Mediterranean	5	5.68
Mediterranean-Iranian-Turanian	4	4.54
Atlantic-Mediterranean	4	4.54
Sub-cosmopolitan	3	3.40
Eurasian	3	3.40
Paleo-Subtropical	3	3.40
Paleo-Temperate	3	3.40
Circum- Mediterranean	2	2.27
Ibero-Moroccan	2	2.27
Eastern Mediterranean	2	2.27
Western Mediterranean	2	2.27
Thermo cosmopolitan	2	2.27
Temperate-Subtropical	2	2.27
Macaronesian-Mediterranean	1	1.13
Mediterranean-Asian	1	1.13
Sub-Mediterranean	1	1.13
Spain-North Africa	1	1.13
Eurasiatic- Mediterranean	1	1.13
Tropical-Subtropical	1	1.13
Endemic	1	1.13
Circumboreal	1	1.13
West Mediterranean-Sub-Atlantic	1	1.13
South African	1	1.13
Endemic. North African	1	1.13
European-Siberian-Mediterranean	1	1.13
European-Asian-North American	1	1.13
Macaronesian-Mediterranean-Iranian-Turanian	1	1.13
North tropical	1	1.13

3.5. Spatio-temporal evolution

The results obtained by analyzing the method of weeding (chemical, mechanical and control) for the three stations (plots) showed that the richness of species differs between plots according to the weeding applied: C and M with respect to the Ct (Figure V.9). In fact, 81% of the inventoried species were present in the control station (Ct), 56% in the station with mechanical weeding (M) and 43% in the chemical (C) weeding station. Due to the level of significance of the F-test is less than 0.001 (Table V.5) for plots, this indicates that there are very significant differences (p = 0.000) between the different weeding methods. The significant difference for the typology of the three stations reveals the impact of the weeding mode on the floristic composition of the weed flora of citrus groves.

Considering the time of weeding, the species richness before weeding tended to be higher (p = 0.06) to that after weeding for the Ct and the two methods of weeding. A graphical representation using the R software is added to identify the spatio-temporal change in mean species richness (Figure V.9). At the level of the chemical weeding station, it has been recorded the presence of perennial weeds in favors of annuals. Most perennials are found in the watering basin at the foot of trees. Paradoxically, annual weeds dominate in the station subjected to mechanical weeding. Therophytes and geophytes represent the most advanced groups in the Raunkiaer classification (Jauzein, 1995).



Legend (C: Chemical weeding, M: Mechanical weeding, Ct: Control, PA: Presence/Absence).

Figure V.9. Spatio-temporal evolution of species richness according to treatment.

From the agronomic point of view, the geophytes and species with vegetative propagation are clearly abundant in a soil not constantly worked (Delpech, 1980). The tillage operations favor the annual appearance of therophytes, however, through the redistribution of the seed stock. This biological type is perfectly adapted to productive and disturbed habitats (Grime, 1977). In the study area, farmers rarely use chemical weeding (herbicides). Mechanical weeding is the norm (Kazi Tani, 2010).

The species-plot interactions confirm the dissimilarity of the species richness and nature of the species recorded in the three stations, including their frequency of occurrence (Table V.5). The species mainly present in all floristic surveys were: *Oxalis pes-capreae* L., *Convolvulus arvensis* L., *Cynodon dactylon* (L.) Pers. and *Cyperus rotundus* L. Otherwise several species were indifferent to the method of weeding and were present in the three stations (C, M and Ct) of the experimental plot (*Solanum nigrum* L., *Malva sylvetris* L., *Avena sterilis* L., *Sinapis arvensis* L., *Chenopodium album* L., *Daucus carota* L., *Fumaria capreolata* L., *Portulaca oleracea* L. and *Setaria viridis* (L.) P. B.).

As for the species-plot interaction and time, it turns out that the difference is highly significant with a p-value (<0.001) (Table V.5), the method of weeding resulted to be an important factor that influence on the floristic diversity of the three plots under study and condition results at weeding.

Table V.5. Multi-factor ANOVA (Response : Presence / Absence).

	SumSq	DF	F value	P value	Code
Species	90.5	87	26.1716	$< 2.2e^{-16}$	***
Plot	0.64	2	8.0620	0.0003167	***
Time	0.14	1	3.4075	0.0649198	NS
Species: Plot	12.25	174	1.7708	0.000000002172	***
Species: Time	16.67	87	4.8204	$< 2.2e^{-16}$	***
Plot: Time	0.02	2	0.1969	0.8212916	NS
Species: Plot: Time	10.79	174	1.5599	0.000003800582	***

NS = Not significant; * = Significant ** = Highly significant; *** = Very highly significant at 5% probability level, 1% and 1 ‰, respectively.
DF: Degrees of Freedom, SumSq: sum of squares.

3.6. Floristic similarity (Jaccard's index)

The results of similarity are presented in table V.6. According to De Bello (2007), if the JI augment, an important number of species are encountered in the two habitats, thus evocating that the biodiversity inter-habitat is low (similar environmental conditions between habitats).

JI showed that different plots vary according to the presence/absence of species (Table V.6). C_B showed moderate similarity to Ct_B (38.46%) but showed low similarity to M_B (34.61%), while M_B (54.09%) had strong similarity to Ct_B, however M_A (40.62%) and Ct_A (44.28%) revealed a moderate similarity to Ct_B. Finally, C_A (28.12%) showed the least similarity to Ct_B.

Table V.6. Coefficient of similarity (%) between the three plots before and after treatments (Ct, C and M) of the studied citrus orchards using Jaccard's Index.

	Ct_B	C_B	M_B	T_A	C_A	M_A
Ct_B	1					
C_B	38.46	1				
M_B	54.09	34.61	1			
Ct_A	44.28	33.89	54.09	1		
C_A	28.12	45.65	35.84	33.89	1	
M_A	40.62	25.92	47.16	47.45	25.92	1

Ct_B: Control before treatment, C_B: Chemical plot before treatment, M_B: Mechanical plot before treatment, Ct_A: Control after treatment, C_A: Chemical plot after treatment, M_A: Mechanical plot after treatment.

3.7. Frequency of occurrence of species

The results of the frequency occurrence of species in the citrus orchard studied are presented in Table V.7. Three constant species were recorded in the Ct plot and two constant species in the M plot before treatment, whereas after treatment one species for each plot (C and M) was characterized as constant. Ct plot contained the highest number of sporadic species, followed by M and then C plots. Moreover, for accessory and accidental species Ct plot contained the highest number of species, succeeded by M and then C plots. The degree of consistency is different compared to the different protocols; it takes a descending line.

Table V.7. Occurrence frequency of species for the three plots before and after treatments.

	Plots	Constant	Accessory	Accidental	Sporadic
Before	Ct	3	6	7	36
	C	0	3	6	27
	M	2	4	5	31
After	Ct	0	4	6	39
	C	1	3	3	22
	M	1	5	3	28

Ct: Control, C: Chemical, M: Mechanical.

4. Conclusion

The objective of this study was to evaluate the impact of mechanical and chemical weeding on the floristic diversity of citrus orchards *vs.* a control (without weeding) at different weeding times (before and after). The floristic surveys carried out during 2016-2017 have identified 88 species belonging to different botanical families. The results showed that the agricultural practices influenced the weed flora of the citrus orchards. This was mainly due to the method of weeding, which affected the weed flora’s composition and the frequency of species composing it.

In general, four species occupied the first places; they were the most common perennial weeds present in Algeria: *Oxalis pes-caprae* L., *Convolvulus arvensis* L., *Cynodon dactylon* (L.), and *Cyperus rotundus* L. The notion of biological type is also a point to consider because, in agronomy, it is closely related to the cultural context. Superficial and irregular weeding favors perennial weeds. The therophytes are adapted to productive and disturbed habitats because they are the only ones capable of growing between two weeding operations. Thus, farming techniques affect the composition, evolution, and dynamics of the weed flora. The impact of the weed control method on the floristic diversity of citrus orchards cannot be the only factor governing the floristic composition and the dynamics of the adventitious flora. Other agricultural practices may have an effect and may need to be studied.

Consequently, this study requires other more precise studies to illuminate the questions studied. We recommend continuing the study for at least three years to see if there is reproducibility, expand the study area and complete the present study with an inquiry into the current situation of citrus growing in Algeria on all plans, and more specifically on the use of chemical inputs. Weeds can be useful for many purposes. The biological properties the chemical composition of weeds may make some species useful for the pharmaceutical and food industries, but also for animal nutrition and production.

5. Acknowledgements

The authors thank the owner of the citrus orchard studied and his employee for their technical assistance.

Chapter VI

Total phenol and flavonoid contents and the *in vitro* antioxidant activities of nineteen weed species from Northwest of Algeria

"Weed is a plant whose virtues have not yet been discovered"

Ralph Waldo Emerson

Experimental Trials

Chapter VI

Total phenol and flavonoid contents and the *in vitro* antioxidant activities of nineteen weed species from Northwest of Algeria

Contents

▪ **Abstract:** Weeds are usually considered undesirable plants for agricultural systems world-wide. However, research is needed to explore the antioxidant activity of them related to their content in plant secondary metabolites (PSM) for valorizing its use at farm level instead of destroying them. The link between the total phenol and flavonoid content and the *in vitro* antioxidant activity (AA) of the aqueous extract got from fifteen weed species [*Aristolochia baetica* L., *Calendula arvensis* L., *Chrysanthemum coronarium* L., *Echium vulgare* L., *Emex spinosa* (L.) Campd., *Fumaria capreolata* L., *Hedera helix* L., *Inula viscosa* L., *Malva sylvestris* L., *Medicago rugosa* Desr., *Oxalis pes-caprae* L., *Reichardia tingitana* L., *Rubia peregrina* L., *Sideritis montana* L. and *Sinapis arvensis* L.] was under study in the current experiment. All weeds were collected from a citrus orchard located in Tlemcen (Northwest of Algeria). Phenols were determined by Folin-Ciocalteu reagent and flavonoids by aluminum chloride colorimetric assay. The antioxidant activities were assessed by the DPPH (2, 2- diphenyl-1-picrylhydrazyl), ABTS (2, 2'-Azino-bis (3-ethylbenzothiazoline-6-sulphonic acid) scavenging activities and chelating metal activity. Statistical analysis was conducted by one-way ANOVA with a single factor (species) considered. Significant differences among species were declared at p-value lower than 0.05 by a comparison Tukey test. Pearson correlation was performed between PSM and AA. Also, a principal component analysis (PCA), boxplot graphic and stacked chart were carried out. According to results, weeds differed significantly ($p<0.001$) into their richness in phenols, flavonoids, DPPH and ABTS scavenging abilities, and the chelating metal ion's ability. The lowest phenolic content ($p<0.001$) was found in *C. arvensis* L. and *Ch. coronarium* while the highest was reached in *I. viscosa*. *S. montana* showed the greatest flavonoids content. However, these compounds have not been detected in *S. arvensis* L. extract. The most effective species in scavenging DPPH free radicals was *I. viscosa* while S. *arvensis* was the least effective. Interestingly, *I. viscosa* also showed the best ABTS scavenging ability while *C. arvensis* was the least effective to scavenge ABTS. For the chelating metal ion's ability, *S. arvensis* was the least effective while *M. sylvestris*, *M. rugosa*, *R. tingitana* and *E. spinosa* were the highest ones. A very strong positive correlation was found between phenols and flavonoids and between phenols and DPPH scavenging ability while a moderate correlation was observed between phenols and ABTS scavenging ability and between flavonoids and DPPH scavenging ability. To sum up, four species (*I. viscosa*, *S. montana*, *R. peregrina* and *R. tingitana*) were characterized by their richness in phenols, flavonoids, and as good DPPH scavengers. In addition, three other species (*M. sylvestris*, *E. spinosa* and *M. rugosa*) were highlighted as good chelating agents. These results suggest the potential capacity of those weed extracts to be used as source of bioactive compounds that might be applied in cosmetic, food and pharmaceutical industries.
Keywords: Valorization, weeds, citrus orchards, DPPH, ABTS, metal chelating, Tlemcen.

▪ **Résumé :** Les adventices ont généralement toujours été indésirables dans les systèmes agricoles du monde entier. Cependant, des recherches ont prouvé qu'elles peuvent nous être très utiles et nous rendre d'énormes services, d'où la nécessité de leur valorisation au lieu de leur destruction. Parmi les avantages, on peut distinguer leurs propriétés antioxydantes liées à leurs teneurs en métabolites secondaires (PSM). Le lien entre la teneur totale en phénol et flavonoïdes et l'activité antioxydante in vitro (AA) des extraits aqueux de quinze espèces d'adventices [*Aristolochia baetica* L., *Calendula arvensis* L., *Chrysanthemum coronarium* L., *Echium vulgare* L., *Emex spinosa* (L.) Campd., *Fumaria capreolata* L., *Hedera helix* L., *Inula viscosa* L., *Malva sylvestris* L., *Medicago rugosa* Desr., *Oxalis pes-caprae* L., *Reichardia tingitana* L., *Rubia peregrina* L., *Sideritis montana* L. et *Sinapis arvensis* L.] a été étudié dans cette expérience. Les adventices ont été récoltées dans un verger d'agrumes situé à Tlemcen (Nord-ouest de l'Algérie). Les phénols ont été déterminés par le réactif Folin-Ciocalteu et les flavonoïdes par un dosage colorimétrique au chlorure d'aluminium. Les activités antioxydantes ont été évaluées par les activités de piégeage du DPPH (2, 2-diphényl-1-picrylhydrazyl), ABTS (2, 2'-azino-bis (acide 3-éthylbenzothiazoline-6-sulfonique) et l'activité de chélation des ions métalliques. Une analyse ANOVA a un seul facteur (espèce) considéré était réalisée. Des différences significatives entre les espèces ont été déclarées à une valeur p inférieure à 0,05 par un test de comparaison de Tukey. Une corrélation de Pearson a été réalisée entre PSM et AA. De plus, une analyse en composantes principales (ACP), un graphique des boites a moustaches et un graphique empilé ont été réalisés. Selon les résultats, les adventices différaient significativement ($p <0,001$) par leur

richesse en phénols, flavonoïdes, leurs capacités de piégeage de DPPH et ABTS et leur capacité de chélation des ions métalliques. La plus faible teneur phénolique a été trouvé dans *C. arvensis* et *Ch. coronarium* tandis que le plus élevé a été atteint dans *I. viscosa*. *S. montana* a montré la plus grande teneur en flavonoïdes. Cependant, ces composés n'ont pas été détectés dans l'extrait de *S. arvensis*. L'espèce la plus efficace pour piéger les radicaux libres DPPH était *I. viscosa* tandis que *S. arvensis* était la moins efficace. Fait intéressant, *I. viscosa* a également montré la meilleure capacité de piégeage ABTS tandis que *C. arvensis* était le moins efficace pour piéger ABTS. Pour la capacité de chélation des ions métalliques, *S. arvensis* était la moins efficace tandis que *M. sylvestris*, *M. rugosa* Desr., *R. tingitana* L. et *E. spinosa* était les plus élevées. Une très forte corrélation positive a été trouvée entre les phénols et les flavonoïdes et entre les phénols et la capacité de piégeage du DPPH tandis qu'une corrélation modérée a été observée entre les phénols et la capacité de piégeage ABTS et entre les flavonoïdes et la capacité de piégeage du DPPH. En résumé, quatre espèces (*I. viscosa*, *S. montana*, *R. peregrina* L. et *R. tingitana*) ont été caractérisées par leur richesse en phénols, flavonoïdes et en tant que bons piégeurs de DPPH. De plus, trois autres espèces (*M. sylvestris*, *E. spinosa* et *M. rugosa*) ont été mises en évidence comme de bons agents chélateurs. Ces résultats suggèrent la capacité potentielle de ces extraits d'adventices à être utilisés comme source de composés bioactifs qui pourraient être appliqués dans les industries cosmétique, alimentaire et pharmaceutique.
Mots clés : Valorisation, adventices, vergers d'agrumes, DPPH, ABTS, chélation des métaux, Tlemcen.

▪ **ملخص:** تعتبر الحشائش عادة نباتات غير مرغوب فيها للنظم الزراعية في جميع أنحاء العالم ومع ذلك ، هناك حاجة إلى البحث لاستكشاف نشاط مضادات الأكسدة الخاصة بهم فيما يتعلق بمحتواها في المستقلبات الثانوية النباتية (PSM) لتثمين استخدامها على مستوى المزرعة بدلاً من تدميرها. تم الحصول على الرابط بين المحتوى الكلي من الفينول والفلافونويد والنشاط المضاد للأكسدة (AA) للمستخلص المائي من خمسة عشر نوعًا من الأعشاب [*Aristolochia baetica* L., *Calendula arvensis* L., *Chrysanthemum coronarium* L., *Echium vulgare* L., *Emex spinosa* (L.) Campd., *Fumaria capreolata* L., *Hedera helix* L., *Inula viscosa* L., *Malva sylvestris* L., *Medicago rugosa* Desr., *Oxalis pes-caprae* L., *Reichardia tingitana* L., *Rubia peregrina* L., *Sideritis montana* L. and *Sinapis arvensis* L.] قيد الدراسة في التجربة الحالية. جمعت جميع الحشائش من بستان حمضيات يقع في تلمسان (شمال غرب الجزائر). تم تحديد الفينولات بواسطة كاشف Folin-Ciocalteu والفلافونيدات عن طريق مقايسة قياس الألوان لكلوريد الألومنيوم. تم تقييم الأنشطة المضادة للأكسدة من خلال أنشطة الكسح DPPH (2, 2- diphenyl-1-picrylhydrazyl), ABTS (2, 2'-Azino-bis (3-ethylbenzothiazoline-6-sulphonic acid) ونشاط المعادن المخلّبة. التحليل الاحصائي تم إجراؤه بواسطة تحليل التباين (ANOVA) أحادي الاتجاه مع مراعاة عامل واحد (الأنواع). تم الإعلان عن اختلافات كبيرة بين الأنواع بقيمة p أقل من 0.05 من خلال مقارنة اختبار Tukey. تم إجراء ارتباط Pearson بين PSM و AA. أيضًا تم اجراء تحليل المكون الرئيسي (PCA) ، رسم Boxplot والرسم البياني المكدس. وفقًا للنتائج ، اختلفت الحشائش بشكل كبير (P<0.001) في ثرائها في قدرات الكسح بالفينول والفلافونويد و DPPH و ABTS وقدرة أيونات المعادن المخلبية. تم العثور على أقل محتوى الفينول (P<0.001) في *Ch. coronarium* و *C. arvensis* بينما تم الوصول إلى أعلى نسبة في *I. viscosa*. في مل يتعلق بالفلافونويد ، أظهرت *S. montana* أعلى محتوى ومع ذلك ، لم يتم الكشف عن هذه المركبات في مستخلص *S. arvenis* . أكثر الأنواع فاعلية في إزالة الجذور الحرة (DPPH) كانت I. viscosa بينما S. arvensis كانت الأقل فعالية. ومن المثير للاهتمام ، أظهرت *I. viscosa* أيضًا أفضل قدرة على إزالة الجذور الحرة (ABTS) بينما كانت C. arvensis هي الأقل فاعلية. بالنسبة لقدرة أيونات المعادن المخلبية ، كانت *S. arvensis* هي الأقل فاعلية بينما كانت *M. sylvestris, M. rugosa, R. tingitana, E. spinosa* الأكثر فعالية. تم العثور على علاقة إيجابية قوية للغاية بين الفينولات والفلافونويد وبين قدرة الكسح (DPPH) و الفينولات بينما لوحظ وجود ارتباط متوسط بين الفينولات وقدرة الكسح (ABTS) وبين الفلافونويدات والقدرة على الكسح (DPPH). باختصار ، أربعة أنواع (*I. viscosa, S. montana, R. tingitana, R. peregrina*) تميزت بثرائها في الفينولات والفلافونويدات و بقدرتها على إزالة الجذور الحرة (DPPH) . بالإضافة إلى ذلك ، تم تمييز ثلاثة أنواع أخرى (*M. sylvestris, E. spinosa, M. rugosa*) كعوامل مخلبية جيدة. تشير هذه النتائج إلى القدرة المحتملة لهذه المستخلصات على استخدامها كمصدر للمركبات النشطة بيولوجيًا التي يمكن استخدامها في صناعات مستحضرات التجميل والأغذية والأدوية.
الكلمات المفتاحية: التثمين ،الاعشاب الضارة ، بساتين الحمضيات ، DPPH، ABTS ، تخلب المعادن ، تلمسان.

1. Introduction

Weeds are plants that grow where they are not desirable to thrive and that propagate naturally (without human intervention) in natural or semi-natural habitats (Brunel et *al*., 2005). They are diverse and very abundant in nature and in cultivated fields, belonging to different botanical families and to different genera (Gomaa, 2017, Chafik et *al*., 2013a). Weeds produce alongside other plants a wide range of plant secondary metabolites (PSM), which are recognized to have medicinal properties (Selamoglu et *al*., 2018). Thus, it is recently reaching an interest world-wide for stablishing the link between PSM and antioxidant activities (AA) of plant extracts for valorization of weeds (Ishak et *al*., 2018, Akbar et *al*., 2020, Disciglio et *al*., 2017).

Several studies have been carried out on the chemical composition of plants and their antioxidant activities which are conditioned by different factors as those mentioned below. The species of the same family and even of the same genus do not possess similar behavior in relation to this (Păltinean et *al*., 2017, Maiza-Benabdesselam et *al*., 2007). The plant organs have also a determining effect (Jan et *al*., 2014, Rad et *al*., 2013, Eruygur et *al*., 2012). Moreover, the extraction type affects the amounts of the phenolic compounds extracted and the antioxidant activities (Sriti Eljazia et *al*., 2018, Salim et *al*., 2017, Ćetković et *al*., 2004). In addition, the period and the region of harvest are also determining factors (Chahmi et *al*., 2015 and Conforti et *al*., 2008).

In cultivated fields, the growth of weeds is inevitable by forming a specific flora to each crop. Previous work (first experimental part) conducted by Chemouri et *al*. (2019) examined the effect of mechanical and chemical weeding on the floristic diversity of citrus orchards in Tlemcen (Northwest of Algeria). The floristic inventory was composed of 88 weed species belonging to 71 genera and 30 botanical families. From this study, nineteen[1] weeds were selected for PSM and AA analysis to investigate them. These species have been used from antiquity in the folk medicine and investigated by several scientists for the phytochemical composition of their extracts, their essential oils and hydrosols, and their antioxidant activities. General information about the selected weed species, regarding their folk uses, secondary metabolites and biological activities as reported in the literature were already provided in the Chapter III of the literature review section (page 53).

[1] Initially, nineteen weed species (20% of the total inventoried species) were chosen to be studied in this experimental part, but given the pandemic caused by Covid-19, at the global level, we were unable to obtain the results of nineteen species. For this reason, we only retained fifteen species, for which we possessed homogeneous results that could be statistically processed.

The objective of this study was to evaluate the capacity of the aqueous extracts of those fifteen weeds, widely spread in citrus orchards from Algeria, for scavenging free radicals such as 2, 2- Diphenyl-1-picrylhydrazyl (DPPH) and 2, 2'-Azino-bis (3-ethylbenzothiazoline-6-sulphonic acid (ABTS), as well as for chelating metal ions, which catalyzes free radical reaction (Miguel, 2010), and link these AA with the PSM (focusing on total phenol and flavonoid content).

This work aimed at valorizing fifteen weeds widely spread in citrus orchards of Algeria through their applications as antioxidants in cosmetic, food and pharmaceutical industries, which would increase the farmers' profit along with the citrus fruits and juices. These species would become another source of income for farmers and not a damage that would have to be combated, increasing the costs of citrus production.

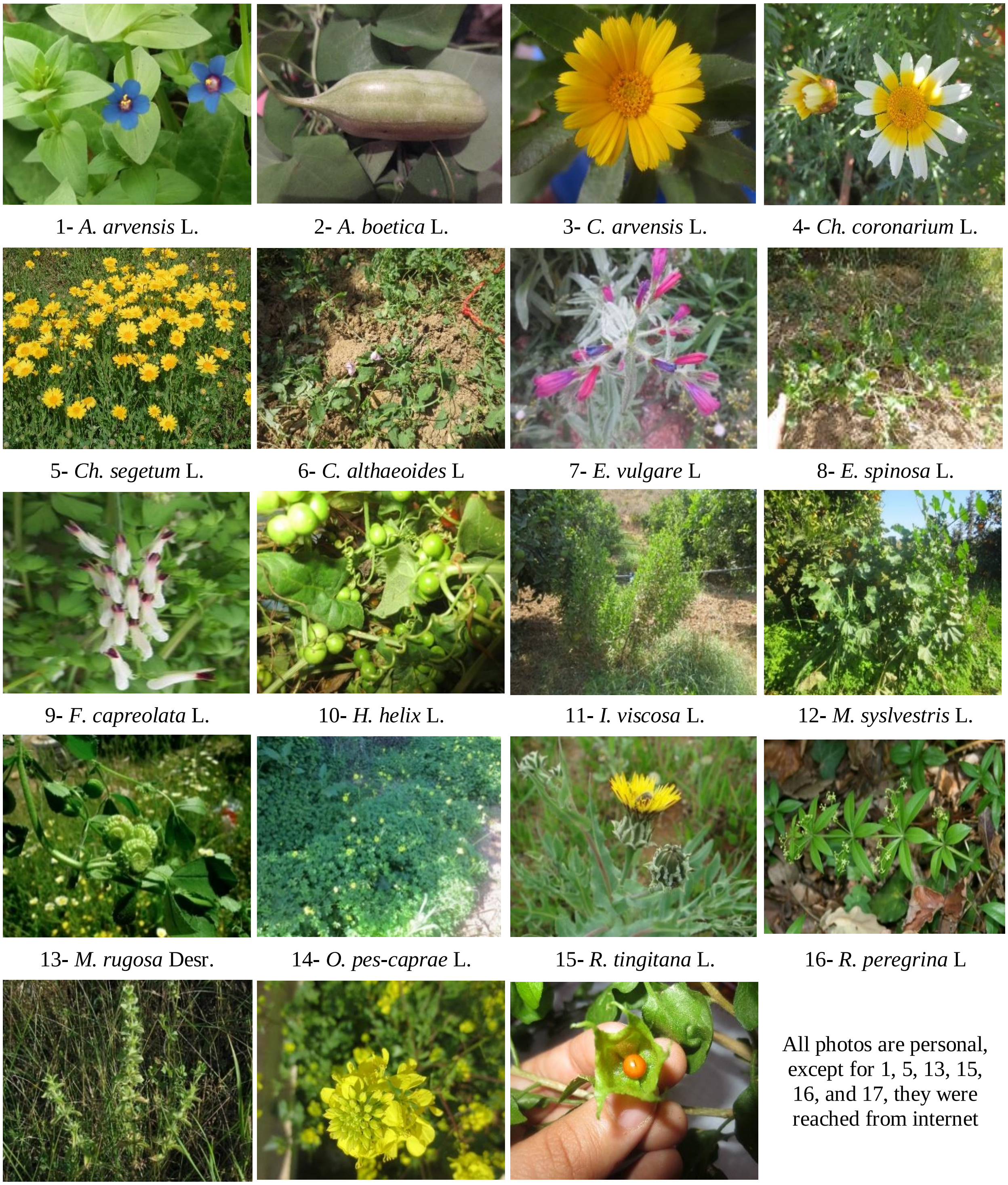
2. Material and Methods

2.1. Plant material

For this study, fifteen weed species (Figure VI.1) were selected from the total of 88 species that compose the flora of a citrus orchards previously described by Chemouri et *al.* (2019). The criterion for choosing those species was to test at random approximately 20% of the species that compose the weed flora (belonging to different botanical families, harvesting them at different growth stages and managing different plant organs) for a general representation of this flora. Plants were harvested, from February to May 2018, in the region of Hennaya (Tlemcen, Algeria), inside a citrus orchard (located at 400 m altitude, 1º22'W longitude, 34º 57'N latitude). The area under study is in a semi-arid climate with temperate winter. The region is characterized by brown soils, with a loamy to sandy-loamy texture, balanced, low in organic matter, and slightly alkaline with a low P_2O_5 reserve. The collected weeds were dried at room temperature in darkness until completely dry. Then, the plant material has been powdered using an electric grinder and stored in the laboratory at ambient temperature for later uses. Table VI.1 provides the description of the fifteen weed species selected for the present study.

2.2. Plant extraction

The aqueous extracts of each weed species were prepared by placing in agitation (130 U/min) 2 g of dried plant material for 12 hours in 20 ml of distilled water. The samples were then centrifuged for 20 min at 3000 rpm; the supernatant was then removed after that and kept at 4ºC until future uses.



1- *A. arvensis* L. 2- *A. boetica* L. 3- *C. arvensis* L. 4- *Ch. coronarium* L.

5- *Ch. segetum* L. 6- *C. althaeoides* L 7- *E. vulgare* L 8- *E. spinosa* L.

9- *F. capreolata* L. 10- *H. helix* L. 11- *I. viscosa* L. 12- *M. syslvestris* L.

13- *M. rugosa* Desr. 14- *O. pes-caprae* L. 15- *R. tingitana* L. 16- *R. peregrina* L

17- *S. montana* L 18- *S. arvensis* L 19- *W. frutescens*

All photos are personal, except for 1, 5, 13, 15, 16, and 17, they were reached from internet

Figure VI.1. Photos of the nineteen[2] weed species selected for the present study

[2] We remember that only fifteen weeds were subjected to the statistical analyzes in this experimental part. However, in this figure we preferred to show the total of the nineteen selected weed species.

Table VI.1. Botanical families, common names, harvest period and organs collected from the fifteen weed species under study.

Weed species	English common name	Botanical family	Harvest period	Organs tested
Aristolochia baetica L.	Andalusian Dutchman's pipe or pipe vine.	Aristolochiaceae	April 2018	Leaves
Calendula arvensis L.	Field marigold.	Asteraceae	February 2018	Leaves and flowers
Chrysanthemum coronarium L.	Garland chrysanthemum, chrysanthemum greens, edible chrysanthemum, crown daisy, chrysanthemum, chop suey green, crown daisy or Japanese-green.	Asteraceae	April 2018	Leaves and flowers
Echium vulgare L.	Viper's bugloss or blueweed.	Boraginaceae	February 2018	Leaves and flowers
Emex spinosa (L.) Campd.	Devil's thorn or lesser jack.	Polygonaceae	April 2018	Leaves
Fumaria capreolata L.	Climbing fumitory, fumitory, rampant fumitory, ramping fumitory, white fumitory, white ramping fumitory, white ramping-fumitory, white- flower fumitory and white-flowered fumitory.	Fumariaceae	April 2018	Leaves and flowers
Hedera helix L.	Common ivy, English ivy, European ivy, or ivy.	Araliaceae	April 2018	Leaves and berries
Inula viscosa L.	False yellowhead, woody fleabane, sticky fleabane, or yellow fleabane.	Asteraceae	April 2018	Leaves
Malva sylvestris L.	Cheeses, high mallow, or tall mallow.	Malvaceae	April 2018	Leaves and flowers
Medicago rugosa Desr.	-	Fabaceae	May 2018	Leaves
Oxalis pes-caprae L.	Bermuda buttercup, African wood-sorrel, Bermuda sorrel, buttercup oxalis, Cape sorrel, English weed, goat's-foot, sour grass, soursob, or soursop.	Oxalidaceae	April 2018	Leaves and flowers
Reichardia tingitana L.	False sow thistle.	Asteraceae	April 2018	Leaves
Rubia peregrina L.	Wild madder.	Rubiaceae	May 2018	Leaves
Sideritis montana L.	Iron wort, mountain tea or shepherd's tea.	Labiateae	April 2018	Leaves and flowers
Sinapis arvensis L.	Charlock mustard, field mustard or wild mustard charlock.	Brassicaceae	May 2018	Seeds

-: not found

2.3. Determination of total phenols

The total phenol content in the aqueous extracts was determined by the Folin-Ciocalteu reagent as described by Singleton and Rossi (1965) where 50 µl of sample at different concentrations and 250 µl of 10% (v/v) Folin-Ciocalteu reagent were added to 200 µl of sodium carbonate (75 mg ml^{-1}). After 1 hour of reaction at room temperature, the absorbance was measured at 765 nm in a Shimadzu 160-UV spectrophotometer.

The gallic acid was used as a standard for the construction of the calibration curve (from 275 to 0.54 µg ml^{-1}, R^2=0.999). The results were expressed as milligram gallic acid equivalent (GAE) per gram of dry weight (DW). All tests were carried out in triplicate and results presented as mean ± standard error of the mean (SEM).

2.4. Determination of total flavonoids

Flavonoids in aqueous extracts were quantified using the aluminum chloride colorimetric assay as mentioned by Isla et *al*. (2011). A volume of 500 µl of $AlCl_3$ (20 mg ml^{-1}) was mixed with 500 µl of sample at different concentrations. The absorbance was measured at 420 nm, after 60 min of incubation at room temperature, in a Shimadzu 160-UV spectrophotometer.

The quercetin was used as a standard (from 50 to 1.56 µg ml^{-1}, R^2=0.996). The results were expressed as milligram quercetin equivalent (QE) per gram of DW. The tests were repeated for three times and results presented as mean ± SEM.

2.5. Determination of antioxidant activities

The antioxidant activity of samples could be evaluated in *vivo* and in *vitro*. In this investigation, we are interested by the *in vitro* evaluation of weed extracts. Due to there is not an approved and standardized method for determining the exact antioxidant capacity of plant material, the performance of more than one test is therefore considered adequate and necessary. As cited in a previous time, there are two main approaches to evaluate the *in vitro* antioxidant activities of samples: the inhibition of lipid oxidation in diverse systems and the capacity to scavenge free radical species (Aazza et *al*., 2011b).

2.5.1. 2, 2- Diphenyl-1-picrylhydrazyl (DPPH) free radical scavenging activity

The ability of the aqueous extracts to scavenging the DPPH radical was determined according to the technique described by Tefiani et *al.* (2016). In brief, 1000 µl of DPPH solution (25 µg ml^{-1}) was added to 25 µl of sample in a dilution series of ten concentrations. After one hour of incubation, in darkness at room temperature, the absorbance was measured at 517 nm in a Jenway 6700 spectrophotometer. Tests were carried out in triplicate and results presented as mean

± SEM. The percentage inhibition was then calculated using the following formula [(Absorbance $_{Control}$−Absorbance $_{Sample}$/Absorbance $_{Control}$) ×100] for each dilution.

The free radical scavenging activity was expressed by the concentration providing 50% inhibition (IC_{50}), which was got by plotting the inhibition percentage against aqueous extract concentrations. The quercetin (from 200 to 0.8 µg ml^{-1}) and 3,5-Di-*tert*-butyl-4-hydroxytoluene (BHT) (from 39 to 5,000 to µg ml^{-1}) were used as positive controls.

2.5.2. 2, 2'-Azino-bis (3-ethylbenzothiazoline-6-sulphonic acid (ABTS) free radical-scavenging activity

The assay of ABTS scavenging activity was determined as reported by Aazza et *al.* (2011a). In brief, 25 µl of each concentrations of the sample were mixed with 1000 µl of ABTS solution and left to react for 6 min. The absorbance was measured at 734 nm in a Jenway 6700 spectrophotometer. The tests were repeated three times and results presented as mean ± SEM.

The quercetin (200 - 0.2 µg ml^{-1}) and the BHT (2.4 - 5,000 µg ml^{-1}) were used as positive controls. The inhibition percentages and the IC_{50} were expressed in the same way as previously described for DPPH assay.

2.5.3. Chelating metal ions

The capacity of chelating ferrous ions by the aqueous extracts of the species studied was evaluated according to the method adopted by Wang et *al.* (2004). Briefly, 25 µl of $FeCl_2$ (0.4 mg ml^{-1}) was added to 25 µl of sample and 925 µl of ethanol (96%). After 30 seconds of reaction with agitation, 25 µl of ferrozine (2.5 mg ml^{-1}) was added; then the mixture was vortexed and incubated for 10 min in darkness. Thereafter, the absorbance was measured at 562 nm in a Jenway 6700 spectrophotometer. Tests were carried out in triplicate and results presented as mean ± SEM.

The 2, 2′, 2″, 2‴-(Ethane-1, 2-diyldinitrilo) tetraacetic acid (EDTA) (from 5000 to 78 µg ml^{-1}) was used as positive control for chelating metal ions. The inhibition percentages and the IC_{50} were expressed as previously mentioned for DPPH assay.

2.6. Statistical analyses

The data were analyzed using one-way ANOVA with weed species as main factor followed by the comparison test of Tukey. Variables (phenols, flavonoids and IC_{50} of DPPH and ABTS scavenging activities and chelating metal ion ability) of all the aqueous extracts obtained from the fifteen weed species studied were compared among different weed extracts using the following model: $Y_{ij}=\mu+\alpha_i+\varepsilon_{ij}$, where µ=average, αi=species (i=15). Statistical analyses were carried out by IBM SPSS Statistics version 26.0.

Significant differences among species were declared at p-value lower than 0.05. The Pearson's correlation was performed between PSM (phenols and flavonoids) and the $1/IC_{50}$ obtained by each antioxidant test (DPPH, ABTS and chelating metal ions) and between the AA performed. The p-values lower than 0.05 were considered significant. To run PCA (Principal Component Analysis), boxplot graphic and stacked chart the PAST statistics, version 4.02 software (Øyvind Hammer, Natural History Museum, University of Oslo) was used (Hammer et *al*., 2001).

3. Results

3.1. Phenol and flavonoid contents

The concentration of phenols and flavonoids is shown in Table VI.2. Significant differences ($p < 0.001$) were observed among weed species regarding those PSM. The lowest phenolic content was found in *Ch. coronarium* L. and *C. arvensis* L., with an average value for both of 12.27±0.095 mg GAE g-1 of DW (dry weight), while the greatest was reached in *I. viscosa* (69.72±0.644 mg GAE g-1of DW) followed by *S. montana* and R. peregrina. Moreover, *I. viscosa* and *S. montana* extracts should be also considered as outliers according to the Figure VI.2, *I. viscosa* and *S. montana* extracts are even outliers (Figure VI.3). In the rest of species, phenols reached an intermediate level among those previously mentioned.

For flavonoids, *S. montana* showed the highest amount (3.88±0.006 mg QE g-1 of DW) while *I. viscosa* and *O. pes-capreae* occupied the second and third position in the ranking (2.79±0.021 and 1.91±0.031 mg QE g-1of DW, respectively). However, this compound has not been detected in *S. arvensis* L. seeds aqueous extract. The other species showed an intermediate level among those previously indicated. No outliers were found for flavonoids.

Table VI.2. Total mean and SEM for phenol and flavonoid contents of the aqueous extracts, belonging to the fifteen weed species under study, in comparison to the literature.

Weed species	**Phenol (mg GAE g^{-1} DW)**		**Flavonoid (mg QEg^{-1} DW)**	
	In the current study	**In the literature‡**	**In the current study**	**In the literature‡**
Aristolochia baetica L.	27.52±0.988^{e}	360±20[6]	0.59±0.015^{d}	35 ± 8[6]
Calendula arvensis L.	12.91±0.166^{a}	24.6±3.5[2] 16.8 ±0.7[1]	0.22±0.006^{bc}	10.2 ± 2.5[2]
Chrysanthemum coronarium L.	11.62±0.023^{a}	27.4±0.5[1]	0.17±0.006^{b}	-
Echium vulgare L.	18.81±0.677^{b}	9.71 ± 0.03[7]	0.62±0.020^{d}	22.06 ± 3.46[22*] (mg g^{-1}) 10.48 ± 0.13[23]
Emex spinosa (L.) Campd.	25.18±0.476^{e}	35.53 ±0.31[8]	0.29±0.020^{c}	10.94 ± 0[8]
Fumaria capreolata L.	25.31±0.751^{e}	18.56[9]	0.90±0.012^{e}	7.73[9]
Hedera helix L.	21.85±0.586^{cd}	39.16 ± 0.48[12] 2.40 ± 0.01[10] 12.67 ± 1.62[11]	1.18±0.031^{f}	2.25 ± 0.05[12] 9.75 ± 0.82[9*] (mg CE g^{-1})
Inula viscosa L.	69.72±0.644^{i}	59.2 ± 0.010[3] 243.70[4]	2.79±0.021^{k}	77.50 ± 6.48[21]
Malva sylvestris L.	30.39±0.588^{f}	15.37 ± 1.13[11] 27.50 ± 1.06[13] 12.839 ±1.61[14]	1.67±0.060^{i}	10.35 ± 0.52[9*] 4.77 ± 0.07[23*] 25.03±2.34[13*] 3.251± 0.55[14*] (mg CE g^{-1})
Medicago rugosa Desr.	19.97±1.140^{bc}	-	0.20±0.068^{b}	-
Oxalis pes-caprae L.	22.48±0.620^{d}	20.58 ± 0.48[17] 8.46±1.20[18] 4.16±0.32[18] 3.32±0.33[18]	1.91±0.031^{j}	25.14±0.78[17*] (mg RE g^{-1})
Reichardia tingitana L.	32.52±0.393^{f}	85.67 ± 1.68[15*] (mg g^{-1} DW)	1.45±0.031^{h}	11.21 ± 1.07[15*] (mg g^{-1} DW)
Rubia peregrina L.	42.90±1.717^{g}	-	1.29±0.012^{g}	-
Sideritis montana L.	56.90±1.111^{h}	97.85±0.98[5] 84.55±1.29[5] 49.05±0.81[5]	3.88±0.006^{l}	3.97[20*] (mg CE g^{-1})
Sinapis arvensis L.	22.45±0.555^{d}	19.72 ± 2.31[19] 9.47±0.41[16] 15.76±0.56[16] 5.04±0.24[16]	0.00±0.000^{a}	1.94 ± 0.72[19*] 2.54±0.16[16*] 1.62±0.11[16*] 3.05±0.03[16*] (mg CE g^{-1})

Note: Values in the current experiment represented the mean of three replications and the SEM. Phenols were expressed as mg GAE $g^{-1}$of DW. Flavonoids were expressed as mg QE g^{-1} of DW. Different letters in the same column (a-l) indicate significant differences among weed species (p<0.001). ‡**References:** [1]Tawaha et *al*. (2007), [2]Akhtar et *al*. (2018), [3]Ozkan et *al*. (2019), [4]Sriti Eljazia et *al*. (2018), [5]Radojević et *al*. (2012), [6]Bourhia et *al*. (2019), [7]Eruygur et *al*. (2012), [8]Makni et *al*. (2018), [9]Pãltinean et *al*. (2017), [10]Deliorman Orhan et *al*. (2012), [11]Piluzza and Bullitta (2011), [12]Salma et *al*. (2018), [13]Ozsoy et *al*. (2017), [14]Mohajer et *al*. (2016), [15]Abd El-Gawad et *al*. (2015), [16]Tabaraki and Ghadiri (2013), [17]Ondua et *al*. (2019),[18]Güçlütürk et *al*. (2012), [19]Başyiğit et *al*. (2020), [20]Balkan et *al*. (2019), [21]Orhan et *al*. (2017), [22]Vukajlović et *al*. (2019) and [23]Conforti et *al*. (2008). [*]Indicate results reported previously in the literature by several authors which were using different units than those used in this experiment without any possibility of conversion for comparison.

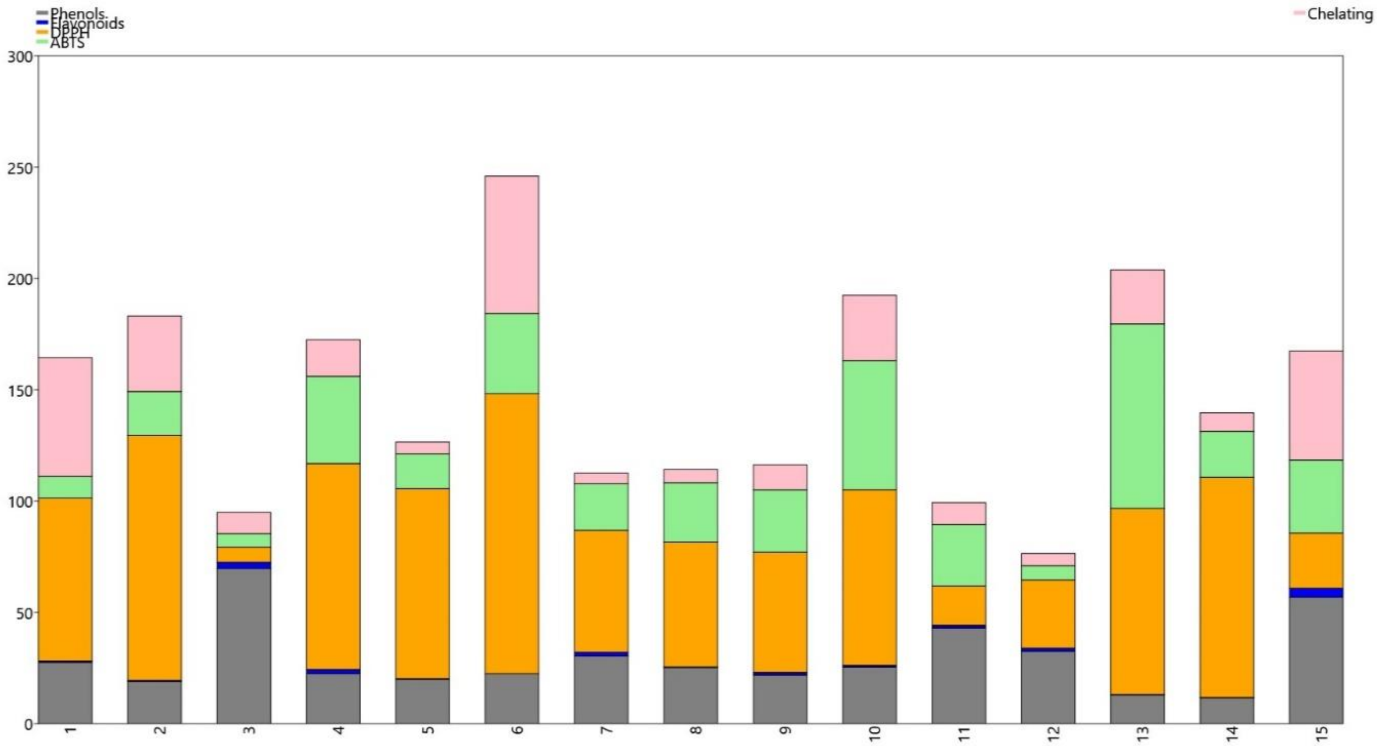


Figure VI.2. Stacked bar chart presenting data of phenol and flavonoid content, scavenging ABTS and DPPH free radicals, and chelating ferrous ion ability by fifteen weed species.

Legend: (1: *A. boetica* L., 2: *E. vulgare* L., 3: *I. viscosa* L., 4: *O. pes-caprae* L., 5: *M. rugosa* Desr., 6: *S. arvensis* L., 7: *M. syslvestris* L., 8: *E. spinosa* (L.) Campd., 9: *H. helix* L., 10: *F. capreolata* L., 11: *R. peregrina* L., 12: *R. tingitana* L., 13: *C. arvensis* L., 14: *Ch. coronarium* L., 15: *S. montana* L.).

3.2. Antioxidant activities

The antioxidant activities of the aqueous extracts from the weeds studied are showed in Table VI.3. A high antioxidant activity is indicated by a low IC_{50} value. The least effective species in DPPH free radical scavenging was *S. arvensis*, (IC_{50}= 125.75±16.513 µg ml^{-1}) while *I. viscosa* (IC_{50}= 6.85±1.329 µg ml^{-1}) was the most effective. The rest of weed species were reaching an intermediate position among those ones. No outliers were found for DPPH scavenging activity. Regarding the ABTS free radical scavenging ability, *C. arvensis* showed the lowest capacity to scavenge ABTS (IC_{50}= 82.93±3.478µg ml $^{-1}$), even being an outlier (Figure VI.3). However, *I. viscosa* presented the highest ability to scavenge ABTS (IC_{50} = 5.97±0.690 µg ml^{-1}). The other weeds reached an intermediate scavenging ABTS ability among the mentioned ones. In terms of chelating metal ion activity, *S. arvensis* presented the lowest ability of metal chelating ions (IC_{50}=61.65±5.123 µg ml^{-1}) while four weed species presented the highest activity (*M. sylvestris*, *M. rugosa*, *R. tingitana* and *E. spinosa,* respectively) with an IC_{50} lower than that obtained from the standard used (EDTA). The rest of weed species showed an intermediate position among those previously mentioned before. No outliers were found for chelating metal ion activity.

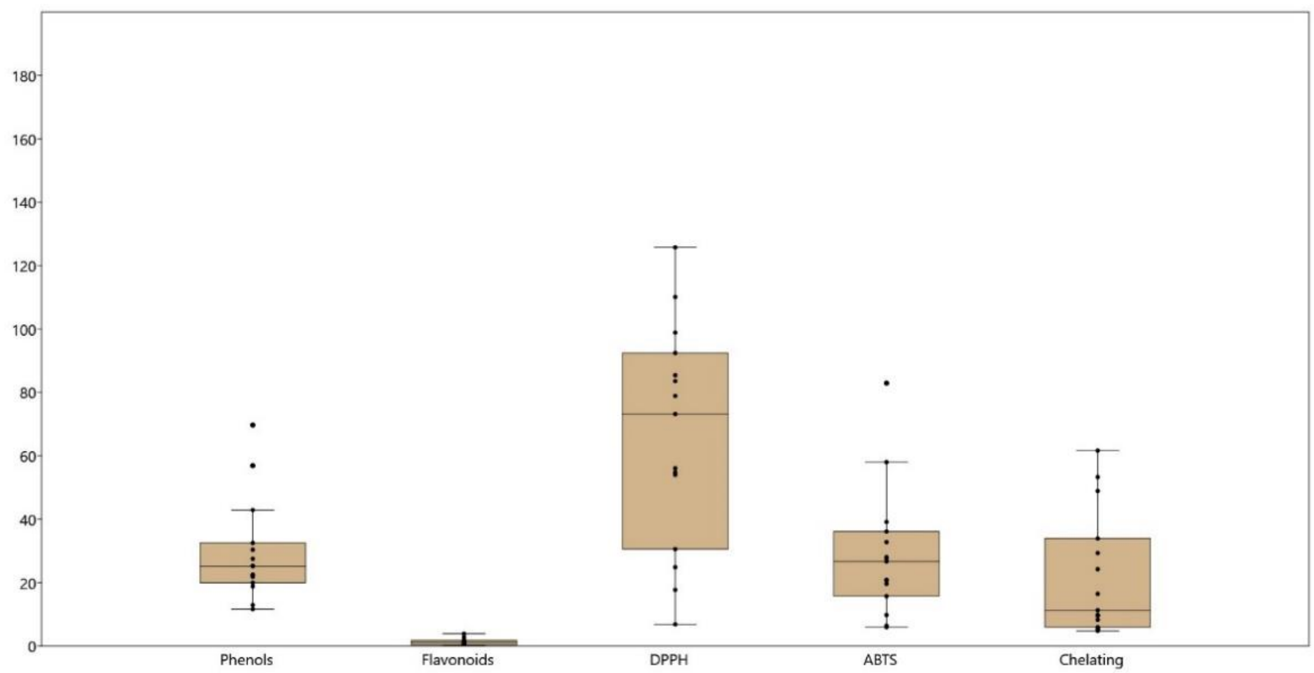


Figure VI.3. Box plot of the phenols, flavonoids, and antioxidant activities of the sample extracts. Horizontal lines within boxes are medians, boxes show inter-quartile ranges and bars show the maximum and minimum values.

Table VI.3. Mean and SEM for DPPH and ABTS scavenging abilities and chelating metal ions of the aqueous extracts, belonging to the fifteen weed species under study, in comparison to the literature.

Weed species / Family	DPPH IC_{50} (µg ml^{-1})		ABTS IC_{50} (µg ml^{-1})		Chelating metal ions IC_{50} (µg ml^{-1})	
	In the current study	**In the literature♣**	**In the current study**	**In the literature**	**In the current study**	**In the literature♣**
Aristolochia baetica L. Aristolochiaceae	73.18±4.986[cde]	150±8[1]	9.78±0.225[b]	-	53.31±1.295[f]	-
Calendula arvensis L. Asteraceae	83.58±1.705[ef]	48.7±1.5%[29*] 33.2±2.12[30*] (mg ml^{-1})	82.93±3.478[i]	42.0±5.4[2*] 48.4 ±1.7[2*] (µmol TE g^{-1} DW)	24.23±1.265[d]	-
Chrysanthemum coronarium L. Asteraceae	98.88±1.692[fg]	64.501±1.229[17]	20.64±0.895[d]	143±5.9[2*] 224±7.1[2*] (µmol TE g^{-1} DW)	8.29±0.085[ab]	-
Echium vulgare L. Boraginaceae	110.15±16.28[gh]	51.38±65.18[6]	19.64±1.560[d]	-	33.93±2.793[e]	2.62 ± 0.03%[31*] At 50 µg ml^{-1}
Emex spinosa (L.) Campd. Polygonaceae	56.09±1.826[cd]	10.89±0.98[15] 10.22 ±0.03[18] 11.13± 0.05[18]	26.69±0.367[e]	9.70 ±0.01[18] 5.79 ±0.54[18]	5.93±0.250[a]	15.99±1.42[15]
Fumaria capreolata L. Papaveraceae	78.90±1.940[def]	68.31±0.35%[19*] At 100 µg ml^{-1} 45,6%[20*] At 50 µg ml^{-1}	58.02±0.880[h]	75.28[33*] (mg TE g^{-1} DW)	29.32±2.027[de]	-
Hedera helix L. Araliaceae	54.00±1.849[c]	107.3[21] 122.47 ± 9.31[22]	28.02±1.59[e]	3.71 ± 0.15[23*] (mmol 100 g^{-1} DW)	11.27±0.400[bc]	51.4[34]
Inula viscosa L. Aiton Asteraceae	6.85±1.329[a]	23.2[7] 30[8] 470±0.03[9]	5.97±0.690[a]	170±0.03[9] 210±0.07[9] 230±0.03[9] 16.75±0.26[13]	9.56±0.382[ab]	18.51 ± 0.74%[16*] 11.35 ± 1.53%[16*] At 3000 µg ml^{-1}

Malva sylvestris L. Malvaceae	54.77±3.124[c]	30%[24*] (0.1 mg dry plant ml^{-1} water) 75±2[25]	20.90±0.519[d]	6.11 ± 0.70[23*] TEAC (mmol/100 g DW) 0.34±0.03[26*] (µmol Trolox g^{-1})	4.76±0.485[a]	176.72[14]
Medicago rugosa Desr. Fabaceae	85.42±0.963[ef]	-	15.71±0.084[c]	-	5.21±0.038[a]	-
Oxalis pes-caprae L. Oxalidaceae	92.45±17.195[efg]	31.39 ± 2.16[27] 17.93[28]	39.15±1.661[g]	24.5 ± 2.91[27]	16.45±0.234[c]	-
Reichardia tingitana L. Asteraceae	30.58±0.497[b]	663.98[35*] (mg ml^{-1})	6.41±0.318[ab]	-	5.46±1.386[a]	-
Rubia peregrina L. Rubiaceae	17.70±0.419[ab]	55.6[10]	27.60±1.056[e]	-	9.79±0.721[ab]	-
Sideritis montana L. Lamiaceae	24.86±1.909[ab]	31.37±2.56[11] 64.55±3.61[11] 527.96 ±4.31[11] 80[12]	32.78±0.201[f]	117.5±2.5[33]	48.90±0.632[f]	-
Sinapis arvensis L. Brassicaceae	125.75±16.513[h]	24.72±0.44[3] 43.78±2.10[3] 21.87±0.69[3] 4.67 ± 0.10[4] 14.79±1.97[5*] (µmol TE g^{-1} DW)	36.13±0.350[fg]	88.67±0.78[3] 397.92 ± 3.20[4*] (µmol TE g^{-1}DW)	61.65±5.123[g]	31.73 ± 1.38[5*] (mg EDTAEs g^{-1})
BHT	38.19±2.203	-	2.50±0.032	-	-	-
Quercetin	1.27±0.060	-	0.10±0.010	-	-	-
EDTA	-	-	-	-	7.98±0.580	-

Note: Values in the current experiment represented the mean of three replications and the SEM. DPPH: 2, 2-diphényl-1-picrylhydrazyle. ABTS: 2, 2'-Azino-bis 3-ethylbenzothiazoline-6-sulphonic acid. IC_{50}= concentration providing 50% of inhibition. BHT and quercetin were used as standards for the ABTS assay. EDTA was used as standard for the chelating metal ions assay. Different letters in the same column (a-h) indicate significant differences among weed species (p<0.001). [‡]**References:** [1]Bourhia et *al*. 2019, [2]Tawaha et *al*. 2007, [3]Tabaraki and Ghadiri (2013), [4]Başyiğit et *al*. (2020), [5]Sarikurkcu et *al*. (2017), [6]Nikolova et *al*. (2011), [7]Ozkan et *al*. (2019), [8]Sriti Eljazi et *al*. (2018), [9]Gökbulut et *al*. (2013, [10]Maxia et *al*. (2012), [11]Radojević et *al*. (2012), [12]Balkan et *al*. (2018), [13]Mahmoudi et *al*. (2016), [14]Mohajer et *al*. (2016), [15]Shahat et *al*. (2015), [16]Orhan et *al*. (2017), [17]Bordoloi et *al*. (2016), [18]Al-Laith et *al*. (2019), [19]Bribi et *al*. (2015), [20]Maiza-Benabdesselam et *al*. (2007), [21]Žugic et al. (2014), [22]Pop et *al*. (2017), [23]Piluzza and Bullitta (2011), [24]Ferreira et *al*. (2006), [25]Tabarki et *al*. (2012), [26]Benso et *al*. (2016), [27]Ondua et *al*. (2019), [28]Gaspar et *al*. (2018), [29]Akhtar et *al*. (2018), [30]Abudunia et *al*. (2016), [31]Eruygur et *al*. (2012), [32]Păltinean et *al*. (2017), [33]Venditti et *al*. (2016), [34]Gülçin et *al*. (2004) and [35]Abd El-Gawad et *al*. (2015). [*]Indicate results reported previously in the literature by several authors which were using different units than those used in this experiment without any possibility of conversion for comparison.

3.3. Pearson's correlations and Principal Component Analysis (PCA)

Pearson's correlations between the PSM (phenols and flavonoids) and the AA (DPPH and ABTS free radicals scavenging activities and chelating metal ion) were established for the aqueous extracts of the fifteen weed species under study.

According to results reported in Table VI.4, it was found a very strong positive correlation between phenols and flavonoids (r= 0.824, p-value<0.01) and between phenols and DPPH scavenging activity (r = 0.852, p-value < 0.01). However, a moderate positive correlation was registered between phenols and ABTS scavenging activity (r = 0.503, p-value<0.01) as well between flavonoids and DPPH scavenging ability (r = 0.565, p-value < 0.01). No correlation (p>0.05) was found for the others.

Table VI.4. Pearson's correlations between PSM (phenols and flavonoids) and AA (DPPH, ABTS and chelating metal ions) given as $1/IC_{50}$.

	Phenols	Flavonoids	DPPH	ABTS	Chelating metal ions
Phenols	1	0.824^{**}	0.852^{**}	0.503^{**}	0.007^{NS}
Flavonoids		1	0.565^{**}	0.258^{NS}	0.064^{NS}
DPPH			1	0.312^{*}	-0.328^{*}
ABTS				1	-0.447^{**}
Chelating metal ions					1

DPPH :2,2-diphényl-1-picrylhydrazyle. ABTS: 2, 2'-Azino-bis 3-ethylbenzothiazoline-6-sulphonic acid. Signification: ** = <0.01, * = <0.05, NS= not significant (>0.05).

The results of the fifteen plants from all tests were used to develop the PCA model (Figure VI.4a). Principal component 1 (PC1) explained up to 67.2% of the total variance and PC2 explained 16.2%. Thus, the two-dimensional graphic presenting the two PCs was able to explain 83.4% of the variance in the data, which is high enough to represent all the variables.

The loading plot of the variables showed that the ABTS scavenging and chelating activities are strongly related and the same can be observed between phenol and flavonoid content and in opposite direction of DPPH assay. This is expected due to the values of AA were given in IC_{50}

values. Nevertheless, the inverse relation was observed between phenols and ABTS. Chelating activity was less evident.

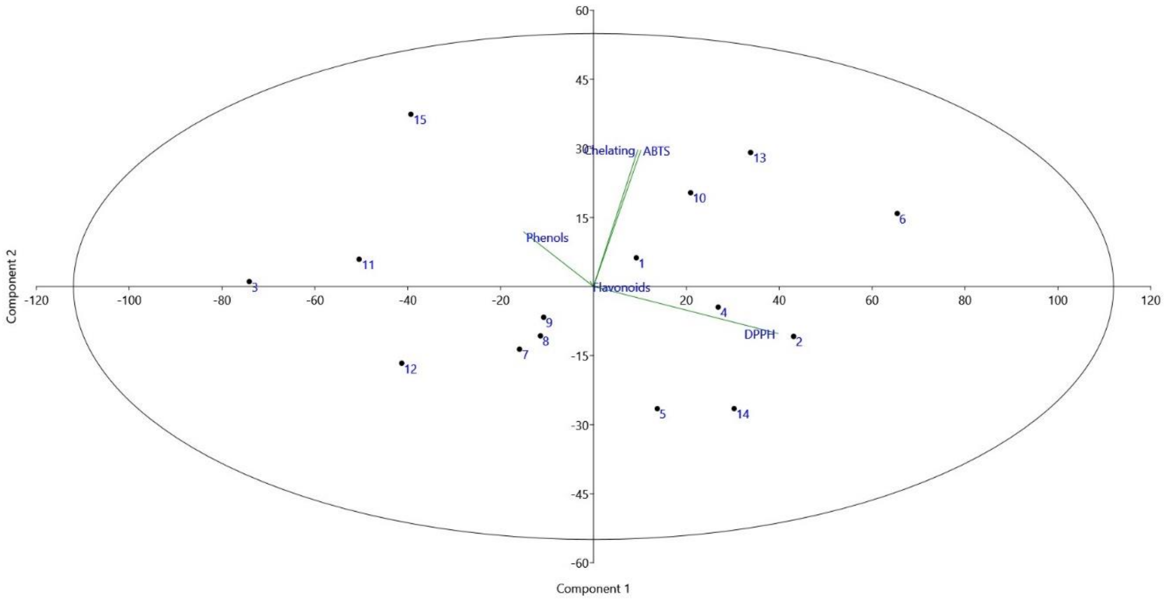
The quantitative values showing these correlations are given in Table VI.4, in which the activities are given as the inverse of IC_{50} values. In fact, *I. viscosa*, *R. peregrina* and *S. montana* with higher amounts of total phenols and lower values of IC_{50} for DPPH assay (better activity) are in opposite position of E. *vulgare*, *O. pes-caprae*, *M. rugosa* and *Ch. coronarium* (Figure VI.4) presenting lower amounts of total phenols and higher IC_{50} values for the DPPH assay (worse activity).

The scatchart (Figure VI.2) makes easier to understand this analysis. The sample extracts of *M. sylvestris*, *E. spinosa*, *H. helix* and *R. tingitana* presented higher chelating ability (lower IC_{50} values) and ABTS scavenging activity than *A. baetica*, *S. arvensis*, *F. capreolata* and *C. arvensis* (Figures VI.2 and VI.4a). Furthermore, the PC1 and PC3 plot explained the 82.1 % of the total variance. In this loading plot, chelating activity and DPPH were in the positive PC1 and PC3, whereas ABTS was in the negative PC3 but positive PC1. Phenols' content was the sole in the negative PC3.

In this loading plot, *I. viscosa*, *R. peregrina* and *S. montana* are grouped as those possessing high amounts of phenols since they are positively correlated with PC1 in contrast to *O. pes-caprae*, *M. rugosa*, *F. capreolata*, *C. arvensis* and *Ch. Coronarium*, which possessed higher IC_{50} values for ABTS assay (they are positively correlated) (Figure VI.4b) and low phenol content (they are negatively correlated).

Figure VI.2 makes easier this observation. *A. baetica*, *E. vulgare* and *S. arvensis* are positively correlated with the IC_{50} values of the ability for scavenging DPPH free radicals and for chelating ferrous ions, therefore, they had low ability for scavenging these free radicals and chelating the metal ion (Figure VI.4b). Those samples were in the opposite direction of *M. sylvestris* L., *E. spinosa* (L.) Campd., *H. helix* L. and *R. peregrina* L. Such means that these plant extracts are better scavengers of DPPH free radicals and chelator of ferrous ions. Figure VI.4 may help this achievement.

a)



b)

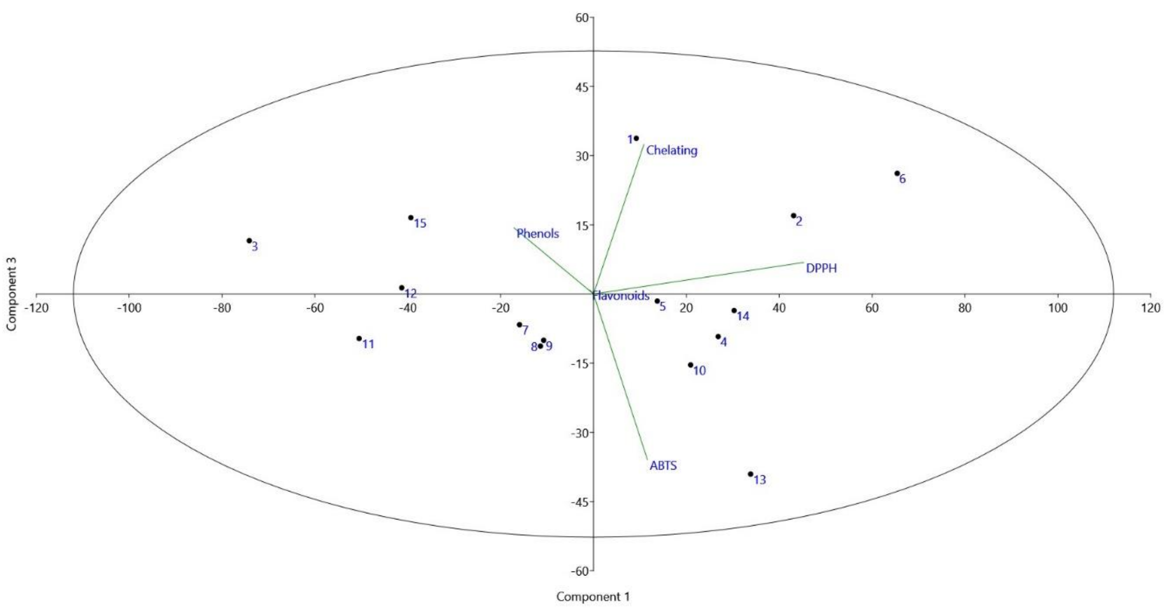


Figure VI.4. (a) Plot of two principal compounds analyses (PCA), using the first and the second component. (b) Plot of two principal compounds analyses (PCA) using the first and the third components.

Legend: (1: *A. boetica* L., 2: *E. vulgare* L., 3: *I. viscosa* L., 4: *O. pes-caprae* L., 5: *M. rugosa* Desr., 6: *S. arvensis* L., 7: *M. syslvestris* L., 8: *E. spinosa* (L.) Campd., 9: *H. helix* L., 10: *F. capreolata* L., 11: *R. peregrina* L., 12: *R. tingitana* L., 13: *C. arvensis* L., 14: *Ch. coronarium* L., 15: *S. montana* L.).

4. Discussion

From a general point of view, the results obtained in the current study for phenols were similar to those reported by the literature while for flavonoids, they were lower than previously mentioned. Regarding extract's AA, the results of the present study showed that some weeds performed higher and others lower abilities of chelation and to scavenge DPPH and ABTS free radicals comparing to the bibliography. Noting that almost for most of the weed species studied (9 over 15) the chelation metal ions ability was reported for the first time in the current investigation. As for the DPPH and ABTS free radicals scavenging capacity, approximately 50% showed higher activity than mentioned in the literature and 50% showed lower activity. These differences could be due to experimental conditions as several factors might be influencing on them (period and region of harvest, organs tested, extraction type and quantification method).

To avoid misinterpretation of results, discussion for PSM and AA will be focused herein on comparison with previous studies that tested similar weed organs and applied a similar type of extraction (aqueous extraction by preference) and used a similar way to express the results.

4.1. Total phenol and flavonoid contents

Regarding phenols, *Ch. coronarium* and *C. arvensis* presented the lowest phenol amounts in the current study. The value obtained for *Ch. coronarium* was lower than that mentioned by Tawaha et *al*. (2007) (27.4±0.5 mg GAE g^{-1} of DW). For *C. arvensis*, it was similar (16.8±0.7 mg GAE g^{-1} of DW) to that reported by Tawaha et *al*. (2007) and lower than that obtained (24.6±3.5 mg GAE g^{-1} of DW) by Akhtar et *al*. (2018). Those differences might be attributed to the fact that in the present study the extraction was done with cold distilled water while in the two other studies it was done with boiling distilled water which may have increased extraction capacity of phenols. Contrarily, the highest amount of phenols was recorded at the current work in the aqueous extract of *I. vicosa*, being this value lightly greater than that cited for this species by Ozkan et *al*. (2019), 59.2 ± 0.010 mg GAE g^{-1} of DW, but very lower than that mentioned by Sriti Eljazi et *al*. (2018), 243.70 mg GAE g^{-1} of DW, supposing those differences among phenols between experiments are due to the location where the studies were conducted, which is in direct relation to the edaphic and climatic conditions of the region and also to the stage of weeds' growth.

The phenol amount of *S. montana* in the aqueous extract of the current study was higher than that of the ethyl acetate extract (49.05±0.81mg GAE g^{-1} of DW) while lower than those of acetone and methanolic extracts (84.55±1.29 and 97.85±0.98 mg GAE g^{-1} of DW, respectively) as mentioned by Radojević et *al*. (2012). It might be due to different extraction capacities by solvents. For *H. helix*, the phenols value obtained was higher than that mentioned by Deliorman Orhan et *al*. (2012) for leaves aqueous extract (2.4±0.01 mg GAE g^{-1} of DW) but lower to that obtained by Salma et *al.* (2018) for leaves aqueous extract (39.16±0.48 mg GAE g^{-1} of DW), supposing that it was due to different growth stage of weeds tested.

For *M. syslvestris* and *O. pes-capreae*, the phenols were higher than that reported by Ozsey et *al*. (2017) and Ondua et *al*. (2019), with reported mean values of 27.50±1.06 and 20.58±0.48 mg GAE g^{-1} of DW, respectively. The phenol amount of *S. arvensis* in the current study was also higher than that previously mentioned by Tabarki and Ghadiri (2013) for the aqueous extracts of leaf, flower, and stem (9.47±0.41, 15.76±0.56, 5.04±0.24 mg GAE g^{-1} of DW, respectively), which might be due to the fact that the weed organs used in the present work (seeds) were highly rich in phenolic compounds, not the same to those tested by these authors.

Considering flavonoids, in the aqueous extract of *S. arvensi*s' seeds under study, these compounds have not been detected, while were quantified at low level in the work realized by Başyiğit et *al.* (2020), with an average value of 1.94 ± 0.72 mg catechin equivalent g^{-1} (CE g^{-1}), being remarkable that in this case the method of flavonoids quantification and the extraction were not the same than that used in the current study. Meanwhile, it is highlighted that *S. montana* showed high flavonoids amount in the current study, which was similar to that obtained by Balkan *et al*. (2019), using a chloroform extract and with the catechin as standard (3.97 mg CE g^{-1}). However, the content of flavonoids obtained for *I. viscosa* in the present study was extremely low comparing to that mentioned by Orhan et *al*. (2017) for leaves after methanolic extraction (77.50 ± 6.48 mg QE g^{-1} extract) due to better extraction capacity of methanol than water.

In addition, the flavonoids amount registered for *O. pes-caprae* was also lower than that obtained by Ondua *et al*. (2019) for water extraction but using rutin as standard (25.14±0.78 mg RE $g^{-1}$of fraction water). Supposing that those differences were due to different way for expressing results. The flavonoids content recorded for *C. arvensis* was lower than that obtained by Akhtar et *al*. (2018) (10.2±2.5 mg QE g^{-1} DW), noting that in the study carried by those authors, boiled water was used for the extraction. Also, for *H. helix*, flavonoids were lower than that obtained by Salma et *al*. (2018) for leaves aqueous extract (2.25±0.05 mg QCE g^{-1} DW). By assuming that could be due to the flavonoid quantification method was different.

4.2. Antioxidant activities

Some *in vitro* AA of the weeds studied were already reported in the literature by others using diverse methodologies. Nevertheless, the current study was made to use a low cost and easy extraction method (aqueous extraction) to apply same methodology for comparison of species. Keeping that in mind, the low IC_{50} value after extraction indicates high antioxidant activity.

In the present study, the least effective's species to scavenge DPPH was *S. arvensis* (125.75±16.513 µg ml^{-1}). In the literature, the DPPH scavenging activity of *S. arvensis* was high, but results were reported using a different way to express them, which were referred to µmol or mg trolox (TE) per gram of extract (Başyiğit et *al*., 2020, Sarikurkcu et *al*., 2017, Tabaraki and Ghadiri, 2013). The DPPH scavenging activity of leaf, flower and stem aqueous extracts have been indicated by the following values (24.72±0.44, 43.78±2.10 and 21.87±0.69 µmol TE g^{-1}, respectively) in the study carried out by Tabaraki and Ghadiri (2013) and with the value of 4.67±0.10 µmol TE g^{-1} as cited by Başyiğit et *al*. (2020) for the seeds extract. Sarikurkcu et *al*. (2017) also mentioned the mean value 14.79±1.97 µmol TE g^{-1} for the ethanolic extract of the aerial parts of *S. arvensis* from Turkey.

Concerning *E. vulgare,* the IC_{50} value found in the current study was two times higher than that signaled by Nikolova et *al*. (2011) for the methanolic extract (IC_{50}=51.38±65.18 µg ml^{-1}). Eruygur et *al*. (2012) reported that the inhibition percentages of the roots ethanolic extract of *E. vulgare* collected from Turkey ranged from 18.85 to 71.2% for the concentrations ranging from 50 to 1000 µg ml^{-1} and for the herbs ethanolic extract the DPPH activity ranged from 15.71 to 43.36% for the concentrations 500 and 1000 µg ml^{-1}, respectively.

The aqueous extract of *I. viscosa* registered the highest DPPH scavenging activity in the present study. The IC_{50} reported herein was three times smaller than that mentioned by Ozkan *et al*. (2019) for a similar extract (IC_{50}=23.2 µg ml^{-1}) and to that reported by Salim et *al*. (2017) for leaves methanolic and ethanolic extracts from Palestina (IC_{50}=18.5 and 20.4 µg ml^{-1}, respectively). It was also four times smaller to that of the leaves aqueous extract (IC_{50}=30±0.08 µg ml^{-1}) of the same species native from City Chaker of region Rawed-Ariana in Tunisia (Sriti Eljazi et *al*., 2018).

Moreover, results from the present study were very lower than the ones of the water extract of *I. viscosa* leaves from Turkey, with the IC_{50} value of 470±30 µg ml^{-1} (Gökbulut et *al*., 2013), and those cited by Chahmi et *al*., (2015) for the mean of the IC_{50} values corresponding to the ethanolic and ethyl acetate extracts of *I. viscosa*, collected from three different regions from

Morocco (IC_{5O}=216 and 923 µg ml^{-1}, respectively). It indicates a very high ability of the present aqueous extract to scavenge the DPPH free radicals, supposing that it is a relevant antioxidant activity inherent to this weed species, independently of the region where the plant grows, with different abiotic and biotic factors that have made all of them that this attribute appears and mark differences with other weed species.

In the next ranking regarding the DPPH scavenging ability, were found *R. peregrina* L. and *S. montana* in the current work. The IC_{50} of *R. peregrina* L. was three times smaller (indicating best scavenging activity) than that mentioned for the ethanolic extract of this species (IC_{5O}=55.6 µg ml^{-1}) as cited by Maxia et *al*. (2012). For *S. montana*, the IC_{5O} obtained in the present study was smaller than those got by Radojević et *al*. (2012), for methanolic, acetone, ethyl acetate and chlorogenic acid extracts (IC_{5O}=31.37±2.56, 64.55±3.61, 527.96±4.31 and 11.65±0.52 µg ml^{-1}, respectively) and also to that reported for the chloroform extract (IC_{5O}=80 µg ml^{-1}) as announced by Balkan et *al*. (2019). By assumption, the previous differences might be due to the dissimilar extraction procedure.

In the present study, *C. arvensis* L. showed a higher ability to scavenge DPPH free radicals than that mentioned by Abudunia et *al*. (2016) (IC_{50}= 33.2±2.12 mg ml^{-1}). At the same time, *R. tingitana*, also recorded a higher activity than that cited by Abd El-Gawed et *al*. (2015) (IC_{50}= 663.98 mg ml^{-1}). However, *O. pes-cacpreae* had low ability to scavenge DPPH in comparison with that cited by Ondua et *al*. (2019) (IC_{50}= 31.39±2.16 µg ml^{-1}). Supposing that all these differences on weeds' DPPH scavenging activities were due to different harvesting periods and locations used at each experiment and different growth stage of the species tested.

Regarding the ABTS scavenging ability, *C. arvensis* registered the lowest capacity to scavenge this free radical in the present study. Revising the literature for this species, lower IC_{50} values for ABTS were found but by expressing the results as µmoles or mg of trolox equivalents per g DW, differently to the present work. Tawaha et *al*. (2007) reported IC_{50} values of 42.0±5.4 and 48.4±1.7 µmol TE g^{-1}DW, respectively, for the methanolic and the aqueous extracts of *C. arvensis* L., respectively. Meanwhile, the aqueous extracts of *I. viscosa* presented the best abilities to scavenge the ABTS in the current study.

Comparing that result to similar studies conducted previously by other scientists, the IC_{50} value of *I. viscosa* found here was extremely lower than the values reported by Gökbulut et *al*. (2013) when flower, leaf and root water extracts were tested (IC_{5O}=170±30, 210±70 and 230±30 µg ml^{-1}, respectively) and two times smaller than that mentioned by Mahmoudi et *al*. (2016) for leaves methanolic extract from North-west of Tunisia (IC_{50}= 16.75±0.26 µg ml^{-1}). Results from the current study are indicating the high ability to scavenge the ABTS free radical for *R. tingitana* and *A. baetica*, however, there is no mention to that in the literature. In the current research, *O. pes-capreae* showed low activity comparing to that obtained by Ondua et *al*. (2019) (IC_{50}= 24.5±2.91 µg ml^{-1}). However, *S. arvensis* recorded higher ability to scavenge ABTS than that cited by Tabarki and Ghadiri (2013) (IC_{50}= 88.67±0.78 µg ml^{-1}). Assuming that all those dissimilarities might be due to differences on harvesting periods and locations used at each experiment and differences also on growth stage of the weed species tested.

As regards to the chelating metal ions, *S. arvensis* recorded the highest IC_{50} value from the fifteen weeds under study, which indicated its weak chelating activity. The chelating activity of this species was also investigated in Turkey by Sarikurkcu et *al*. (2017), however, the results were expressed as mg EDTA equivalents per gram extract getting an average value of 31.73 ± 1.38 mg EDTAE g^{-1} extract for its ethanolic extract.

From the rest of weeds under study, there were four species that recorded the biggest chelating metal ion's ability in the present work. Those weed species were *M. sylvestris*, *M. rugosa*, *R. tingitana* and *E. spinosa*, being the IC_{50} values obtained by them smaller than that of the standard used (EDTA), which had the value of 7.98±0.580 µg ml^{-1}, and that revealed their higher abilities for chelating metal ions. In the literature, Mohajer et *al*. (2016) reported the IC_{50} value of 176.72 µg ml^{-1} for the flowers methanolic extract of *M. sylvestris* collected from Iran, indicating its low chelating capacity comparing to the current result. As for *E. spinosa* L., Shahat et *al*. (2015) attributed an IC_{50} value of 15.99±1.42 µg ml^{-1} for methanolic extract of this same species from Saudi Arabia. No mention is found in the bibliography to chelating activities of *M. rugosa* and *R. tingitana*. With the IC_{50} values close to that of the standard, *Ch. coronarium*, *I. viscosa* and *R. peregrina* were the followings in the ranking (Table VI.3).

For *Ch. coronarium* and *R. peregrina*, there was no information's about their chelating metal ions abilities. However, Orhan et *al*. (2017) tested *I. viscosa* for the chelating activity in flower and leaf methanolic extracts at the concentration of 3,000 µg ml^{-1} and they found different inhibition percentages depending on the plant organ under study, ranging from 18.51±0.74 to 11.35±1.53, respectively. No mention is done in the literature to the rest of weeds for chelating metal ions.

4.3. Pearson's correlations and PCA

In a general context, it was possible to highlight the relationships between PSM (phenol and flavonoid contents) of those weed species and their corresponding AA (DPPH and ABTS scavenging activities) as other scientists previously highlighted in other research studies carried out at field level with plants. Kim et *al*. (2011) reported a good correlation between *Ch. coronarium* L. PSM and their AA and a very good positive correlation between ABTS and DPPH abilities. Moreover, Firuzi et *al*. (2010) signaled for the plants tested in their study, a good correlation between total phenolics content and DPPH activity. As well as Tabaraki and Ghadiri (2013), which mentioned high positive linear correlations between total phenols and DPPH and total phenols and total flavonoids, for aqueous and even methanolic extracts of two species, including among them *S. arvensis* L. In other studies, Žugić et *al*. (2014) cited that the antioxidant activity and the phenolic content increased proportionally for the investigated species, including among them *H. helix* L.

Furthermore, Vukajlović et *al*. (2019) indicated at strong dependence of DPPH test results on phenol content for *Echium* spp. water extracts. By contrast, Bordoloi et *al*. (2016) mentioned a moderate correlation between phenolic content and DPPH free radical scavenging activity for the edible herbs studied, including among them *Ch. coronarium*. Similarly, Akhtar et *al*. (2018) registered a slight correlation coefficient between total phenols and flavonoids content and a weak linear correlation between DPPH capacity and phenols for the 61 medicinal plants studied, including among them *C. arvensis*.

There were also cited a very strong positive correlations between phenols and ABTS/DPPH scavenging abilities and between both scavenging assays (ABTS and DPPH) while moderate correlations between flavonoids and ABTS/DPPH scavenging abilities for twenty-four plant species from the Mediterranean area, including among them *H. helix* and *M. sylvestris* (Piluzza and Bullitta, 2011), However, for some wild edible plants consumed as vegetables in Turkey, including *S. arvensis* and *M. sylvestris*, Sarikurkcu et *al*. (2017) signaled a very strong positive correlation for ABTS *vs.* phenols and for ABTS *vs.* DPPH, a strong correlation for DPPH *vs.* phenols, a moderate correlation for DPPH *vs.* flavonoids and, a weak correlation between ABTS and flavonoids.

Al-Laith et *al*. (2019) obtained a very strong positive correlation for DPPH *vs.* ABTS for three medicinal plants including among them *E. spinosa*, while a negative moderate correlation for DPPH *vs*. phenols. Furthermore, Tawaha et *al*. (2007) declared a strong relationship between phenols and ABTS scavenging ability for aqueous and methanolic extracts of selected Jordanian plant species, including among them *C. arvensis* and *Ch. coronarium*. The relationship between phenols and flavonoids and their antioxidant activities were reported by previous studies but it was not always obvious and depended on the samples.

In the present study, it was possible to determinate a strong positive relationship between phenol and flavonoid contents and between the total phenol contents and the DPPH free radical scavenging ability of the weed species studied. It turned out that the DPPH scavenging ability was also dependent on the total flavonoid contents and that the phenol contents correlated with the ABTS scavenging ability. Moreover, there was a weak relationship between DPPH and ABTS assays. These results agree with those cited in the literature, which demonstrated that the phenols and flavonoids are the compounds mainly responsible for the AA determined in the weed species studied. Furthermore, the weak negative correlation between DPPH and chelating metal ions and a moderate negative correlation between ABTS and chelating metal ions assays were found. Probably, it could be due to the mode of action of the different antioxidant tests and the chemical composition of the weed species investigated.

The results of PCA using the first and the second component permitted to conclude that samples can be grouped by higher amounts of phenols and better activity in what concerns the capacity for scavenging DPPH (*I. viscosa*, *S. montana*, *R. peregrina* and *R. tingitana*) and those ones that not presenting relative high amounts of phenols they may be important for chelating ferrous ions (*M. sylvestris*, *M. rugosa*, *E. spinosa* and *H. helix*) and scavenging ABTS free radicals. On the other hand, the results of PCA using the first and the third component showed that *I. viscosa*, *R, tingitana* and *S. montana* are characterized by relative high amounts of total phenols and good capacity for scavenging the ABTS free radicals, whereas *M. sylvestris*, *E. spinosa*, *H. helix* and *R. peregrina* were good scavengers of DPPH free radicals and chelators of ferrous ions.

At the end, and analyzing the two PCA plots, it was possible to observe that in both cases the feature of the capacity of samples *M. sylvestris*, *E. spinosa* and *H. helix* for chelating metal ions while *R. tingitana* and *I. viscosa*, in some way, for scavenging ABTS free radicals, were maintained. This can be the first step for possible mixtures of these extracts to evaluate the possible synergistic effect among them, acting simultaneously as free radical scavengers and chelating agents to be used as a source of bioactive compounds.

5. Conclusion

In this study, aqueous extract of fifteen weeds growing in a citrus orchard located in the Northwest of Algeria have been tested for PSM (focusing on their total phenol and flavonoid contents) and AA (regarding their DPPH and ABTS scavenging activities and chelating metal ions availability) by studying the effect of species as main factor (considering variability in botanical families examined and divergence in plant organs collected). Among the weeds studied, it is remarkable that four species (*I. viscosa*, *S. montana*, *R. peregrina* and *R. tingitana*) were characterized by their richness in phenols and flavonoids, being also them considered as good DPPH scavengers. In addition, three other species (*M. sylvestris*, *E. spinosa* and *M. rugosa*) were highlighted as good chelating agents.

These results suggest the potential capacity of those weed aqueous extracts might have to be valorized as a source of natural bioactive compounds for its use in food, cosmetic and pharmaceutical industries instead of eliminating them in order to avoid the side effects of synthetic antioxidants. The current research needs to be followed by further studies in which other solvents, differing in their extraction capacity, will be evaluated to determine the best option for extracting different PSM and testing other AA to get appropriate comparison between extracts obtained from diverse weed material. In addition to that, mixing extracts from different weeds might also be a strategic point to be addressed for getting at the same time good scavengers and chelators. Also, other biological activities (such as antidiabetic, anti-inflammatory, and anticancer) could be studied for elucidating the potential value of those weed species as a resource to valorize in the future for the agriculture sector. Furthermore, a study related to the toxicity of the fifteen weed species remains necessary and preliminary before exploiting them.

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Chapter VII

Nutritional value and mineral composition of nineteen weed species growing in citrus orchards for their use as potential source of nutrients in ruminants feeding

A weed is not more than a flower in disguise,
Which is seen through at once, if love give a man eyes.

"A Fable for Critics" (Lowell, 1848)

Experimental Trials

Chapter VII

Nutritional value and mineral composition of nineteen weed species growing in citrus orchards for their use as potential source of nutrients in ruminants feeding

Contents

Nutritional value and mineral composition of nineteen weed species growing in citrus orchards for their use as potential source of nutrients in ruminants feeding

▪ **Abstract:** With the aim of evaluate the nutritive value of eighteen weed species belonging to 15 botanical families and growing in citrus orchards, some major (Ca, Mg, K and P) and trace minerals (Zn, Mn, and Fe) which are important for animal feeding, protein, sugars, ash, organic matter, fats, ADF, NDF and NDF-digestibility (NDFD) were determined. Minerals were analyzed according to the method described by Castro et *al*. (1990). Ash and OM were measured following the method reported by AOAC (1996) while the other determined parameters were obtained by the procedure described in COMMISSION REGULATION (EC) No 152/2009. The data were statistically processed using one-way ANOVA followed by the test of Tukey. Significant differences ($p<0.001$) were observed among weed species regarding all the studied parameters. According to the results of the nutritive value, *S. arvensis* L. contained the higher OM content (94.2±0.07 % DM) while *C. arvensis* L. had the higher ash content (24.2±0.08 % DM). For the CP, *Ch. coronarium* L. and *W. frutescens* Pauquy. were the richest species (26.0±1.84 and 25.7±1.84 % DM, respectively). As for the ADF and NDF, *R. peregrina* L. occupied the first rank for both (31.3±2.26 and 34.6±2.47 % DM, respectively). Regarding sugars, *Ch. segetum* L. possessed the higher amount (6.1±0.42 % DM) whereas *H. helix* L. showed the higher fats amount (11.6±0.85 % DM). Finally, *O. pes-caprae* L. was the species exhibiting the best NDFD (94.2±6.65 % DM). About the mineral composition, *Ch. coronarium* showed the highest K and Ca levels (3.43±0.139 and 2.16±0.078 % DM, respectively) while *E. spinosa* (L.) Campd. presented the higher Mg and Zn concentrations (0.51±0.022 % and 57.97±2.470 mg kg^{-1}, respectively). For Fe, *Ch. segetum* was the richest species (130.93±1.053 mg kg^{-1}). Also, *I. viscosa* L. had the best Mn content (78.54±0.061 mg kg^{-1}) and *H. helix* recorded the higher P level (0.55±0.039 %). Furthermore, the most of species could satisfy the mineral requirements of ruminants as recommended by the NRC. These results suppose the potential use of these species for animal feeding.

Keywords: Valorization, minerals, nutritive value, citrus orchards, weeds.

▪ **Résumé :** Dans le but d'évaluer la valeur nutritive de dix-huit espèces de mauvaises herbes appartenant à 15 familles botaniques et poussant dans les vergers d'agrumes, certains minéraux majeurs (Ca, Mg, K et P) et oligo-éléments (Zn, Mn et Fe) qui sont importants pour l'alimentation animale, protéines, sucres, cendres, matières organiques, graisses, ADF, NDF et NDF-Digestibilité (NDFD) ont été déterminés. Les minéraux ont été analysés selon la méthode décrite par Castro et *al*. (1990). Les cendres et la MO ont été mesurées selon la méthode indiquée par AOAC (1996) tandis que les autres paramètres déterminés ont été obtenus par la procédure décrite dans le RÈGLEMENT (CE) No 152/2009 DE LA COMMISSION. Les données ont été traitées statistiquement à l'aide d'une ANOVA à un facteur suivi du test de Tukey. Des différences significatives ($p <0,001$) ont été observées entre les espèces de mauvaises herbes concernant tous les paramètres étudiés. Selon les résultats de la valeur nutritive, *S. arvensis* L. contenait la teneur en MO la plus élevée (94,2 ± 0,07% MS) tandis que *C. arvensis* L. avait la teneur en cendres la plus élevée (24,2 ± 0,08% MS). Pour le CP, *Ch. coronarium* L. et *W. frutescens* Pauquy. ont été les espèces les plus riches (26,0 ± 1,84 et 25,7 ± 1,84% MS, respectivement). En ce qui concerne l'ADF et le NDF, *R. peregrina* L. occupait le premier rang pour les deux (31,3 ± 2,26 et 34,6 ± 2,47% MS, respectivement). Concernant les sucres, *Ch. segetum* L. possédait la quantité la plus élevée (6,1 ± 0,42% MS) tandis que *H. helix* L. présentait la quantité la plus élevée de matières grasses (11,6 ± 0,85% MS). Enfin, *O. pes-caprae* L. était l'espèce présentant la meilleure NDFD (94,2 ± 6,65% MS). À propos de la composition minérale, *Ch. coronarium* présentait les niveaux les plus élevés de K et de Ca (3,43 ± 0,139 et 2,16 ± 0,078% MS, respectivement) tandis que *E. spinosa* (L.) Campd. a présenté les concentrations plus élevées de Mg et de Zn (0,51 ± 0,022% et 57,97 ± 2,470 mg kg^{-1}, respectivement). Pour Fe, *Ch. segetum* était l'espèce la plus riche (130,93 ± 1,053 mg kg -1). En outre, *I. viscosa* L. avait la meilleure teneur en Mn (78,54 ± 0,061 mg kg -1) et *H. helix* a enregistré le niveau le plus élevé de P (0,55 ± 0,039%). De plus, la plupart des espèces pourraient satisfaire les besoins minéraux des ruminants comme recommandé par le NRC. Ces résultats supposent l'utilisation potentielle de ces espèces pour l'alimentation animale.

Mots clés : Valorisation, minéraux, valeur nutritive, vergers d'agrumes, adventices.

Nutritional value and mineral composition of nineteen weed species growing in citrus orchards for their use as potential source of nutrients in ruminants feeding

▪ **ملخص:** من أجل تقييم القيمة الغذائية لثمانية عشر نوعًا من الحشائش التي تنتمي إلى 15 عائلة نباتية وتنمو في بساتين الحمضيات ،تم تحديد بعض المعادن الرئيسية (K, P, Mg, Ca) والعناصرالنادرة (Mn, Fe, Zn) التي تعتبر مهمة لتغذية الحيوانات والبروتينات والسكريات والرماد والمواد العضوية والدهون و ADF و NDF وقابلية هضم الـ NDF (NDFD). تم تحليل المعادن وفقًا للطريقة التي وصفها Castro et *al*. (1990) و تم قياس الرماد و OM وفقًا للطريقة المشار إليها بواسطة AOAC (1996) بينما قيست المتغيرات الأخرى المحددة من خلال الإجراء الموضح في COMMISSION REGULATION (EC) No 152/2009. تمت معالجة البيانات إحصائيًا باستخدام تحليل التباين (ANOVA) أحادي الاتجاه متبوعًا باختبار Tukey. لوحظت فروق معتبرة (P <0.001) بين أنواع الحشائش لجميع المتغيرات المدروسة. وفقًا لنتائج القيمة الغذائية ، احتوت *S. arvensis* L. على أعلى محتوى من OM (94.2±0.07%) بينما احتوت *C. arvensis* L. على أعلى محتوى رماد (24.2±0.08% DM). بالنسبة لـ CP كانت *Ch. coronarium* L. و *W. frutescens* Pauquy. هي أغنى الأنواع (26.0±1.84, 25.7±1.84% DM) بالنسبة لـ ADF و NDF ، احتلت *R. peregrina* L. المرتبة الأولى لكليهما (31.3±2.26, 34.6±2.47% DM). فيما يتعلق بالسكريات ، كان لدى *Ch. segetum* L. أعلى كمية (6.1±0.42% DM) بينما احتلت *H. helix* L. أعلى كمية من الدهون (11.6±0.85% DM) . أخيرًا ، كانت *O. pes-caprae* L. هو النوع الذي يتمتع بأفضل قابلية هضم لـNDF . فيما يتعلق بالتركيب المعدني ، أظهرت *Ch. coronarium* أعلى مستويات K و Ca (3.34±0.139, 2.16±0.078%DM) بينما أظهرت *E. spinosa* (L.) Campd. أعلى تركيزات Mg و Zn (2.47±) . بالنسبة للحديد ، كانت *Ch. segetum* أغنى الأنواع (130.93±1.053 mg Kg^{-1}) . بالإضافة إلى ذلك ، كان لدى *I. viscosa* L. أفضل محتوى من المنغنيز (78.54±0.061 mg Kg^{-1}) وسجلت *H. helix* أعلى مستوى من الفوسفور (0.55±0.039% DM) . بالإضافة إلى ذلك ، يمكن لمعظم الأنواع تلبية المتطلبات المعدنية للحيوانات المجترة على النحو الموصى به من قبل NRC. تفترض هذه النتائج الاستخدام المحتمل لهذه الأنواع لتغذية الحيوانات.

الكلمات المفتاحية: التثمين ، المعادن ، القيمة الغذائية ، بساتين الحمضيات ، الأعشاب الضارة.

Nutritional value and mineral composition of nineteen weed species growing in citrus orchards for their use as potential source of nutrients in ruminants feeding

1. Introduction

In the world, its counted approximatively 30000 weed species (Duke, 1992), from which 89% of the widely spread species are edible (Rapoport et *al*., 1995). Weeds are generally considered as undesirable and usually destined for elimination. However, they could be extremely useful (Maroyi et *al*., 2013).

Weeds possess high nutritional value and interesting medicinal properties. They are consumed by humans as green vegetables and medicinal herbs and by animals as fodder in many African, Asian, and European countries (Khan et *al*., 2013, Romojaro et *al*., 2013, Cruz-Garcia and Price, 2012). Actually, there is a renewed interest on the use of weeds in animal feeding by the lack of surface lands to cultivate forage species, in addition to the supplement charges occurred.

In the Mediterranean basin, grazing is the major source of alimentation for ruminants since the Mediterranean fields are typically rich in species known as weeds though usually grazed by ruminants (Addis et *al*., 2006). Previous studies showed that weeds presented an interesting nutritional source for livestock and a high concentration of minerals such required for a healthy development of animals (Khan et *al*., 2013, Gutiérrez et *al*., 2008, Bianco and Santamaria, 1998, Marten and Andersen, 1975). In addition to a high forage quality (Kamalak et *al*., 2005).

Previous work (first experimental part) was carried out by Chemouri et *al*. (2019) on the impact of mechanical and chemical weeding on the floristic diversity of citrus orchards in Tlemcen (NW Algeria), in which 88 weed species belonging to 71 genera and 30 botanical families were listed. From this study, nineteen[1] weeds were selected to be studied for their mineral composition and nutritive value. Some of these species have been investigated by certain scientist, the previous studies reported in the literature are briefly shown in Table VII.1 while more details on the investigated weed species were presented in the Chapter IV (from 76 to 80p). Nothing that the mineral composition and the nutritive value of some species have been reported for the first time in the current study.

The objectives of this study were to evaluate the nutritive value and the mineral composition of eighteen[1] weed species (which represent approximatively 20% of the total species) widely spread in citrus orchards of Algeria. This investigation aimed at valorizing eighteen weeds

[1] Initially, nineteen weed species (20% of the total species) were chosen to be studied in this experimental part, but given the pandemic caused by Covid-19, at the global level, we were unable to obtain the results of the nineteen species. For this reason, we only retained eighteen species, for which we possessed homogeneous results that could be statistically processed.

that compose the citrus flora of Algeria through their applications as source of nutrients for animal feeding, which would solve the problem of fodder in animal-plant production.

2. Materials and methods

2.1. Plant material

Eighteen weed species (Table VII.1) were collected inside a citrus orchard (400 m altitude, 1º22'W longitude, 34º 57'N latitude) from the region of Hennaya (Tlemcen, Algeria), from February to June 2018. The eighteen species were selected (at random and represent the 20% of the total flora) from the inventory of the weed flora of a citrus orchard previously established by Chemouri et *al.* (2019). The plant powder (Figure VII.1) was obtained using an electric grinder and then stored at the laboratory (ambient temperature) for future uses. Table VII.1 provides the scientific nomenclature, botanical families, mineral composition, and the nutritive value of these species as reported in the literature.

Figure VII.1. Plant material (powder) (Chemouri, 2019).

2.2. Nutritive value analysis

The dry matter (DM) was determined after reaching constant weight at 105ºC in laboratorial oven (Figure VII.2) as described by COMMISSION REGULATION (EC) No 152/2009. Organic matter (OM) and ash contents were obtained after calcination at 600ºC for 2 hours (Figure VII.3), according to the method 942.05 (AOAC, 1996).

Figure VII.2. The determination of DM (Chemouri, 2019).

Table VII.1. Botanical families, harvest date and the parts used, mineral and nutritive value of the eighteen[2] studied weed species.

Weed species nomenclature and botanical families	Parts used	Harvest date	Mineral composition	Nutritive value	References
Anagallis arvensis L. Anacardiaceae	Leaves and flowers	June 2018	-	-	-
Aristolochia baetica L. Aristolochiaceae	Leaves	April 2018	-	-	-
Calendula arvensis L. Asteraceae	Leaves and flowers	February 2018	-	-	-
Chrysanthemum coronarium L. Asteraceae	Leaves and flowers	April 2018	Fe, Zn, Ca, Mg, Na and P	DM, OM, CP, ADF, NDF, acid detergent lignin and digestibility	Cabiddu et *al*. (2006), Valente et *al*. (2003) and Sulas et *al*. (1999).
Chrysanthemum segetum L. Asteraceae	Leaves and flowers	June 2018	-	-	-
Convolvulus althaeoides L. Convolvulaceae	Leaves and flowers	June 2018	Fe, Cu, Mn, Zn, Ca, Mg, Na, P, K, S, Al, B, Cr, Fe, Mo, N and Pb.	Ash, OM, and total sugars.	Baillie et *al*. (2018) and Aly Ahmed et *al*. (2014).
Echium vulgare L. Boraginaceae	Leaves and flowers	February 2018	-	Lipid, phytosterols.	Nogala-Kalucka et *al*. (2010)
Emex spinosa (L.) Campd. Polygonaceae	Leaves	April 2018	-	Ash, lignin, sugars, and fatty acids.	Makni et *al*. (2019) and Shaltout et *al*. (2008).
Fumaria capreolata L. Fumariaceae	Leaves and flowers	April 2018	Fe, Cu, Mn, Zn, Ca, Mg, Na, P, K, S, Al, B, Cr, Fe, Mo, N and Pb.		Baillie et *al*. (2018).
Hedera helix L. Araliaceae	Leaves and berries	April 2018	-	Reducing sugars	Uddin et *al*. (2011)
Inula viscosa L. Asteraceae	Leaves	April 2018	-	Lipids and fatty acids.	Salim et *al*., (2017) and Mahmoudi et *al*., (2015).

[2] We remember that only eighteen weeds were subjected to the statistical analyzes in this experimental part. However, in this table we preferred to show the total of the nineteen selected weed species.

Malva sylvestris L. Malvaceae	Leaves and flowers	April 2018	Fe, Cu, Mn, Zn, Ca, Mg, Na, P, K, S, Cr, Cd, Co and Pb.	DM, ash, azote (N), CP, reducing sugars, crude fibre and Fats.	Tunçtürk et *al*. (2018), Romojaro et *al*. (2013) Civelek and Balkaya (2013) and Barros et *al*. (2010).
Medicago rugosa Desr. Fabaceae	Leaves	May 2018	S, Ca and P.	Ash, ether extract, crude fiber, crude protein, CP, ADF, NDF.	Porqueddu, (2001), Dougall and Bogdan, 1966
Oxalis pes-caprae L. Oxalidaceae	Leaves and flowers	April 2018	Fe, Cu, Mn, Zn, Ca, Mg, Na, P, K, S, Al, B, Cr, Fe, Mo, N and Pb.	Total ash, acid insoluble ash, water soluble ash.	Baillie et *al*. (2018), Edrah et *al*. (2018) and Romojaro et *al*. (2013).
Reichardia tingitana L. Asteraceae	Leaves	April 2018	Ca, K, Fe and Na.	Ash, CF	Metwally Gamal (2017). Sayed, 1993
Rubia peregrina L. Rubiaceae	Leaves	May 2018	-	-	-
Sideritis montana L. Labiateae	Leaves and flowers	April 2018	-	Fatty acids and vitamins.	Emre et *al*. (2011).
Sinapis arvensis L. Brassicaceae	Seeds	May 2018	Fe, Cu, Mn, Zn, Ca, Mg, Na, K and P.	Ash, CP, ADF, NDF, OMD, moisture, lipids, vitamins, and carbohydrates.	Başyiğit et *al*. (2020), Lenzi et *al*. (2019) and De Oliveira et *al*. (2019) Kamalak et *al*. (2005).
Withania frutescens Pauquy. Solonaceae	Leaves	March 2018	-	-	-

-: Not found.

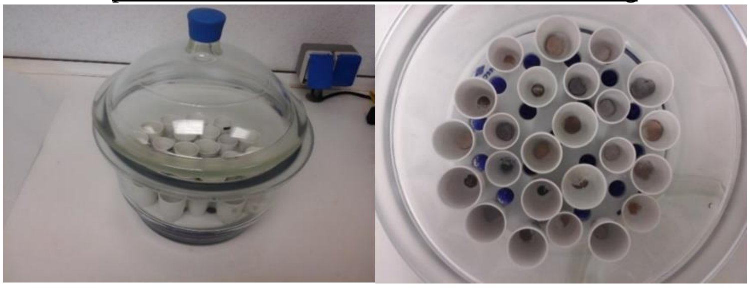

Figure VII.3. The determination of OM (Chemouri, 2019)

Crude proteins (CP) were determined using the Kjeldahl procedure reported by the REGULATION COMMISSION (EC) No 152/2009. The CP were calculated by the following formula (Pirie 1955 and Oelberg 1956): CP (% in DM) = Total nitrogen (% in DM) × 6.25.

Acid Detergent Fiber (ADF) and Neutral Detergent Fiber (NDF) were quantified by the procedure described by the REGULATION COMMISSION (EC) No 152/2009. The ADF were determined after extraction with a solution of ethylene diamine tetracetic acid and the NDF after extraction with a detergent solution. The NDF has been used to estimate the digestibility (NDFD) of the weed species using the method described by Goering and Van Soest (1970). The total sugars were determined according to the Luff-Schorrl method described by the REGULATION COMMISSION (EC) No 152/2009. Fats were quantified after extraction with petroleum ether according to the procedure reported by the REGULATION COMMISSION (EC) No 152/2009. The results were expressed in percentage of dry weight (% DW).

2.3. Mineral contents analysis

Concentrations of phosphorus (P), calcium (Ca), magnesium (Mg), potassium (K), iron (Fe), manganese (Mn), and zinc (Zn) were determined after extraction with a 96% sulfuric acid solution and hydrogen peroxide (Figure VII.4), according to the method described by Castro et *al.* (1990). The minerals were measured spectrophotometrically at 660 nm. The phosphorus content was measured using the JENUAY 6300 spectrophotometer (Figure VII.5), while the rest of the minerals were determined with an ICP-OES team (Perkin Elmer, model Optima 4300 DV). For major minerals (P, K, Ca, and Mg), the results were expressed in percentage of DM (% DM), whereas for trace minerals (Fe, Zn, and Mn), they were expressed in milligrams per kilogram (mg kg^{-1}).

Figure VII.4. Extraction of minerals with Sulfuric acid (Chemouri, 2019).

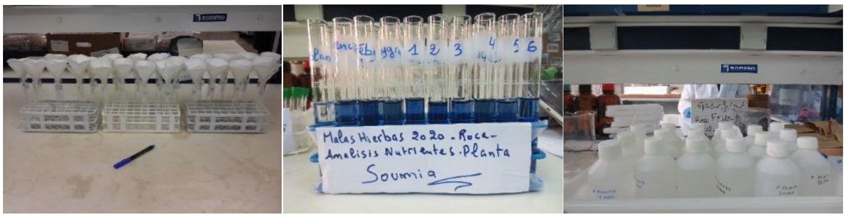

Figure VII.5. Determination of phosphorus concentration (Chemouri, 2019).

2.4. Statistical analysis

The data were analyzed using one-way ANOVA followed by the test of Tukey. Variables: nutritive value (DM, OM, ash, CP, fats, total sugars, ADF, NDF and NDFD) and minerals (Ca, P, Mg, K, Zn, Fe and Mn) of the eighteen weed species studied were compared among the different weeds using the following model: $Y_{ij}=\mu+\alpha i+\varepsilon_{ij}$, where μ=average, αi=species (i=18). Statistical analyses were carried out by IBM SPSS Statistics version 26.0. The p-values lower than 0.05 were considered significant.

3. Results

3.1. Nutritive value

The DM, OM, ash, CP, ADF, NDF, fats, total sugars content and NDFD are shown in Table VII.2. The nutritive value of the weeds investigated herein differed significantly ($p<0.001$) among species.

Regarding the OM, *C. arvensis* L. showed the lowest amount (75.8±0.08 % DM), followed by *E. vulgare* L. (78.1±0.36 % DM). In contrast, *S. arvensis* L. registered the higher amount (94.2±0.07 % DM), succeeded by *C. althaeoides* L. (91.7±0.28 % DM). Therefore, *S. arvensis* has shown the lowest ash amount (5.8±0.07 % DM), followed by *C. althaeoides* (8.3±0.28 % DM), while *C. arvensis* recorded the higher amount (24.2±0.08 % DM), succeeded by *E. vulgare* (21.9±0.36 % DM).

With regard to the crude proteins *R. peregrina* L. registered the lowest amount (9.4±0.71 % DM), followed by *A. arvensis* L., *O. pes-capreae* L. and *I. viscosa* L. (11.7±0.85, 12.0±0.85, 12.3±0.85 % DM, respectively), whereas *Ch. coronarium* L. and *W. frutescens* Pauquy. presented the highest amounts (26.0±1.84 and 25.7±1.84 % DM).

For ADF, *A. arvensis* showed the lowest content (12.8±0.92 % DM) and *W. frutescesns* and *Ch. coronarium* were the following in the rank (14.0±0.99 and 14.1±0.99 % DM, respectively). While *R. peregrina* (31.3±2.26 % DM) marked the highest amount, followed by *H. helix* L. (26.2±1.84 % DM).

As for NDF, *C. arvensis* and *Ch. coronarium* registered the lowest amount (16.8±1.13 and 17.5±1.20 % DM, respectively), succeeded by *A. arvensis* (18.0±1.27 % DM). Whereas *R. peregrina* revealed the higher amount (34.6±2.47 % DM), and in the next ranking, *M. rugosa* Desr. And *H. helix* were mentioned (31.1±2.26 and 30.6±2.12 % DM, respectively).

Regarding total sugars, *Ch. coronarium* registered the less value (0.6±0.00 % DM), *W. frutescens*, *C. arvensis* L., *E. vulgare* and *E. spinosa* (0.8±0.00, 0.9±0.00, 0.9±0.00, 1.1±0.14 % DM, respectively) were in the next rank. While *A. arvensis* was the rich one (14.4±0.99 % DM), followed by *Ch. segetum* L. (9.9±0.71 % DM).

For the total fats, five species (*E. spinosa*, *E. vulgare*, *R. peregrina*, *C. althaoides* and *C. arvensis*) revealed the less amounts (1.4±0.14, 1.5±0.14, 1.6±0.14 and 1.7±0.14, 1.8±0.14 % DM, respectively), while *H. helix* had the higher amount (11.6±0.85), followed by *F. capreolata* L. (5.6±0.42 % DM).

Concerning the NDFD, *H. helix* was the less digestive (31.6±2.26 % DM), followed by *S. arvensis* (35.1±2.47 % DM), whereas *O. pes-capreae* was the most digestive (94.2±6.65 % DM), succeeded by *C. arvensis* (86.4±6.08 % DM) and *W. frutescens* (82.9±5.80 % DM).

Table VII.2. Total mean and standard error of the mean for nutritive value of the eighteen studied weeds.

	Nutritive value (% DM)							
Weed species	**OM**	**Ash**	**Crude protein**	**ADF**	**NDF**	**Total sugars**	**Fats**	**NDF Digestibility**
Anagallis arvensis L.	91.2±0.05^{gh}	8.8±0.05^{bc}	11.7±0.85^{ab}	12.8±0.92^{a}	18.0±1.27^{ab}	14.4±0.99^{i}	1.9±0.14^{ab}	56.2±3.96^{cd}
Aristolochia baetica L.	87.5±0.21^{f}	12.5±0.21^{d}	14.1±0.99^{abc}	19.9±1.41^{cdefg}	26.7±1.91^{cde}	7.5±0.57^{g}	2.9±0.14^{bc}	70.8±4.95^{defg}
Calendula arvensis L.	75.8±0.08^{a}	24.2±0.08^{i}	22.7±1.56^{fgh}	14.2±0.99^{abc}	16.8±1.13^{a}	0.9±0.00^{ab}	1.8±0.14^{a}	86.4±6.08^{gh}
Chrysanthemum coronarium L.	81.2±0.37^{c}	18.8±0.37^{g}	26.0±1.84^{h}	14.1±0.99^{ab}	17.5±1.20^{a}	0.6±0.00^{a}	2.4±0.14^{abc}	77.6±5.52^{efgh}
Chrysanthemum segetum L.	88.1±0.06^{f}	11.9±0.06^{d}	14.2±0.99^{abc}	17.7±1.20^{abcde}	20.2±1.41^{abc}	9.9±0.71^{h}	3.2±0.28^{cd}	60.8±4.24^{cde}
Convolvulus althaeoides L.	91.7±0.28^{h}	8.3±0.28^{b}	15.9±1.13^{bcde}	17.3±1.20^{abcde}	24.5±1.77^{bcde}	7.1±0.49^{fg}	1.7±0.14^{a}	59.9±4.24^{cde}
Echium vulgare L.	78.1±0.36^{b}	21.9±0.36^{h}	14.5±0.99^{abcd}	20.6±1.48^{defgh}	24.4±1.70^{bcde}	0.9±0.00^{ab}	1.5±0.14^{a}	51.1±3.68^{be}
Emex spinosa (L.) Campd.	85.8±0.18^{e}	14.2±0.18^{e}	19.7±1.41^{defg}	19.2±1.34^{bcdef}	21.3±1.56^{abc}	1.1±0.14^{ab}	1.4±0.14^{a}	66.5±4.67^{cdef}
Fumaria capreolata L.	83.4±0.04^{d}	16.6±0.04^{f}	22.0±1.56^{fgh}	19.7±1.41^{bcdef}	22.2±1.63^{abcd}	2.3±0.14^{bc}	5.6±0.42^{e}	69.5±4.94^{cdefg}
Hedera helix L.	90.9±0.10^{g}	9.1±0.10^{c}	20.1±1.41^{efg}	26.2±1.84^{hi}	30.6±2.12^{ef}	1.5±0.14^{abc}	11.6±0.85^{f}	31.6±2.26^{a}
Inula viscosa L.	84.0±0.26^{d}	16.0±0.26^{f}	12.3±0.85^{ab}	18.4±1.34^{abcde}	21.2±1.48^{abc}	2.3±0.14^{bc}	4.0±0.28^{d}	56.6±3.96^{cd}
Malva sylvestris L.	85.2±0.12^{e}	14.8±0.12^{e}	24.1±1.70^{gh}	16.6±1.13^{abcd}	20.3±1.41^{abc}	3.1±0.28^{cd}	2.1±0.14^{ab}	62.3±4.38^{cde}
Medicago rugosa Desr.	91.0±0.02^{gh}	9.0±0.02^{bc}	22.1±1.56^{fgh}	24.2±1.70^{fgh}	31.1±2.26^{ef}	4.8±0.28^{e}	1.5±0.14^{a}	58.7±4.10^{cd}
Oxalis pes-caprae L.	85.7±0.10^{e}	14.3±0.10^{e}	12.0±0.85^{ab}	25.6±1.84^{ghi}	29.0±2.05^{def}	5.7±0.42^{ef}	2.2±0.14^{abc}	94.2±6.65^{h}
Reichardia tingitana L.	-	-	-	-	-	-	-	-
Rubia peregrina L.	84.0±0.06^{d}	16.0±0.06^{f}	9.4±0.71^{a}	31.3±2.26^{i}	34.6±2.47^{f}	4.6±0.28^{de}	1.6±0.14^{a}	64.4±4.53^{cdef}
Sideritis montana L.	88.2±0.19^{f}	11.8±0.19^{d}	18.3±1.27^{cdef}	16.3±1.13^{abcd}	20.9±1.41^{abc}	6.2±0.42^{efg}	1.9±0.14^{ab}	73.5±5.23^{defg}
Sinapis arvensis L.	94.2±0.07^{i}	5.8±0.07^{a}	25.1±1.83^{gh}	22.9±1.63^{efgh}	25.6±1.84^{cde}	6.1±0.42^{e}	2.1±0.14^{ab}	35.1±2.47^{ab}
Withania frutescens Pauquy.	83.7±0.14^{d}	16.3±0.15^{f}	25.7±1.84^{h}	14.0±0.99^{ab}	18.4±1.27^{b}	0.8±0.00^{ab}	2.4±0.14^{abc}	82.9±5.80^{fgh}

-: Not determined. All results were expressed as % DM. Values represented the mean of three replications and the standard error of the mean. Different letters in the same column indicate significant differences between species ($p<0.001$).

3.2. Mineral contents

The mineral concentrations (major and trace minerals) were presented in Table VII. 3. Significant differences ($p<0.001$) were registered between the weed species. Regarding the P content, *A. baetica* L. was the less rich (0.18±0.013% DM), followed by *A. arvensis* (0.24±0.017 % DM), while *H. helix* was the rich one (0.24±0.017 % DM), succeeded by *S. arvensis* (0.53±0.037 % DM).

Weeds varied greatly in their K content with levels ranging from 3.43±0.139, 3.21±0.134 and 2.24±0.080 %, respectively for *Ch. coronarium*, *C. arvensis* and *O. pes-caprae,* respectively to 0.39±0.035 and 0.51±0.008 % for *S. arvensis* and *I. viscosa*.

About Ca amounts, the richest species was found to be *Ch. coronarium* (2.16±0.078 %). In the next ranking *C. arvensis* and *E. vulgare* were found (1.79±0.080 and 1.69±0.025 % respectively). In contrast, *S. arvensis* was the less rich (0.44±0.021 %). Followed by *C. althaeoides, O. pes-caprae, M. rugosa* and *H. helix (*0.69±0.007, 0.73±0.025, 0.77±0.018 and 0.79±0.018 %, respectively).

Among all the weed species, the concentration of Mg was found ranging between 0.51±0.022 and 0.42±0.001 % for *E. spinosa* and *C. arvensis* and between 0.20±0.001 and 0.15±0.002 % for *E. vulgare* and *A. arvensis*.

The high level of Fe was noted in *Ch. segetum* (130.93±1.053 mg kg^{-1}), followed by *Ch. coronarium* (99.71±0.913 mg kg^{-1}) and after *I. viscosa* (92.04±0.715 mg kg^{-1}). In contrast, *S. arvensis* and *H. helix* presented the lowest Fe levels (24.11±0.984 and 25.91±0.222 %, respectively).

In relation to zinc, *E. spinosa* recorded the higher level (57.97±2.470 mg kg^{-1}) followed by *C. arvensis* (49.77±0.435 mg kg^{-1}). Whereas this trace mineral was not detected in *O. pes-caprae* (0.00±0.000 mg kg^{-1}).

Additionally, *I. viscosa* had the highest Mn concentration (78.54±0.061 mg kg^{-1}), followed by *Ch. coronarium* (41.54±0.157 mg kg^{-1}). While *C. althaeoides* had the lowest concentration (16.84±0.197 mg kg^{-1}) succeeded by *E. vulgare* (18.59±0.126 mg kg^{-1}).

Table VII.3. Total mean and standard error of the mean for mineral composition (major and trace minerals) of the eighteen studied weeds.

Weed species	Trace minerals (mg kg $^{-1}$ DM)			Major minerals (% DM)			
	Fe	Zn	Mn	K	Ca	Mg	P
Anagallis arvensis L.	64.22±0.418^{g}	32.63±1.701^{f}	29.86±0.301^{i}	1.14±0.021^{b}	0.98±0.030^{cd}	0.20±0.001^{b}	0.24±0.017^{ab}
Aristolochia baetica L.	43.75±0.365^{d}	12.61±0.392^{d}	27.18±0.212^{g}	1.72±0.66^{def}	1.02±0.042^{cde}	0.29±0.001^{gh}	0.18±0.013^{a}
Calendula arvensis L.	39.49±0.288^{c}	49.77±0.435^{i}	30.55±0.269^{j}	3.21±0.134^{j}	1.79±0.080^{i}	0.42±0.001^{l}	0.41±0.029^{fg}
Chrysanthemum coronarium L.	99.71±0.913^{j}	35.21±1.097^{f}	41.54±0.157^{m}	3.43±0.139^{k}	2.16±0.078^{j}	0.38±0.001^{k}	0.43±0.030^{fgh}
Chrysanthemum segetum L.	130.93±1.053^{k}	9.08±0.518^{bc}	39.35±0.099^{l}	1.72±0.073^{def}	1.20±0.074^{fg}	0.28±0.002^{fg}	0.24±0.018^{abc}
Convolvulus althaeoides L.	52.16±1.080^{e}	6.28±0.540^{b}	16.84±0.197^{a}	1.09±0.012^{b}	0.69±0.007^{b}	0.22±0.002^{c}	0.37±0.027^{defg}
Echium vulgare L.	45.54±0.179^{d}	7.44±0.815^{b}	18.59±0.126^{b}	2.01±0.019^{gh}	1.69±0.025^{i}	0.15±0.002^{a}	0.39±0.028^{efg}
Emex spinosa (L.) Campd.	58.79±0.915^{f}	57.97±2.470^{j}	25.69±0.286^{f}	2.04±0.003^{h}	1.21±0.048^{g}	0.51±0.022^{m}	0.29±0.021^{bcde}
Fumaria capreolata L.	35.49±0.564^{b}	45.00±0.927^{h}	25.96±0.192^{f}	1.84±0.001^{efg}	1.13±0.017^{efg}	0.32±0.001^{i}	0.44±0.032^{ghi}
Hedera helix L.	25.91±0.222^{a}	28.12±0.156$_{e}$	23.81±0.163^{e}	1.61±0.029^{d}	0.79±0.018^{b}	0.26±0.001^{de}	0.55±0.039^{i}
Inula viscosa L.	92.04±0.715^{i}	46.42±0.420$_{h}$	78.54±0.061^{n}	0.51±0.008^{a}	1.08±0.008^{def}	0.30±0.000^{hi}	0.33±0.023^{bcdef}
Malva sylvestris L.	44.92±0.108^{d}	39.09±0.724$_{g}$	27.39±0.256^{g}	1.67±0.016^{de}	1.39±0.012^{h}	0.30±0.003^{h}	0.45±0.032^{ghi}
Medicago rugosa Desr.	34.58±0.093^{b}	10.34±0.387$_{cd}$	21.63±0.184^{c}	1.03±0.030^{b}	0.77±0.018^{b}	0.25±0.004^{d}	0.36±0.025^{defg}
Oxalis pes-caprae L.	44.34±0.452^{d}	0.00±0.000$_{a}$	28.97±0.356^{h}	2.24±0.080^{i}	0.73±0.025^{b}	0.27±0.001^{ef}	0.28±0.021^{abcd}
Reichardia tingitana L.	-	-	-	-	-	-	-
Rubia peregrina L.	70.73±0.411^{h}	11.42±0.530$_{cd}$	21.56±0.093^{c}	1.40±0.030^{c}	1.04±0.012^{cde}	0.38±0.001^{k}	0.36±0.025^{defg}
Sideritis montana L.	57.98±0.621^{f}	40.10±0.703$_{g}$	31.71±0.347^{k}	1.88±0.056^{fgh}	0.95±0.037^{c}	0.30±0.000^{h}	0.35±0.025^{cdefg}
Sinapis arvensis L.	24.11±0.984^{a}	33.22±0.059$_{f}$	23.52±0.122^{e}	0.39±0.035^{a}	0.44±0.021^{a}	0.27±0.005^{def}	0.53±0.037^{hi}
Withania frutescens Pauquy.	58.57±0.347^{f}	26.32±1.046$_{e}$	22.46±0.261^{d}	1.81±0.040^{ef}	0.99±0.027^{cd}	0.35±0.001^{j}	0.43±0.030^{fgh}

-: Not determined. Values represented the mean of three replications and the standard error of the mean. Different letters in the same column indicate significant differences between species ($p<0.001$).

4. Discussion

In a general view, it should be noted that revising the literature for the nutritive value and the minerals content of the eighteen weed species studied, we have not found previous studies in the bibliography for most of the eighteen species. Thus, conduct us to discuss our results only with the references available.

4.1. Nutritive value

Overall, the results obtained for all the measured parameters revealed that the weed species studied correspond to the recommendations of the National Research Council (NRC) for ruminants' requirements.

For the OM, *C. arvensis* recorded the lowest amount, followed by *E. vulgare*. In contrast, *S. arvensis* registered the higher amount, succeeded by *C. althaeoides*. Regarding *C. arvensis*, *E. vulgare* and *S. arvensis*, there was no references to compare. For *C. althaeoides*, the OM amount obtained was higher than that obtained for flower, leaf, and stem (84.78±0.29, 87.5±0.27 and 82.7±0.37 % DM, respectively), as cited by Aly Ahmed et *al*. (2014), this difference might be due to the harvest region. From the rest of species *Ch. coronarium* had a close OM amount to that (92.02±0.1 % DM) reported by Sulas et *al.* (1999). Also, for *O. pes-caprae*, our results agree with those (84.5 %) reported by Edrah et *al*. (2018).

Therefore, *S. arvensis* has shown the lowest ash amount, wish was similar to that reported by Kamalak et *al*. (2005) at late maturity (5.6±0.122 % DM) but higher to those reported for wild mustard raw and cooked (1.40±0.25 and 2.00±0.35 g 100 g^{-1}, respectively) (De Oliveira, 2019), supposing that it was due to the dissimilar parts used noting that seeds were used in our study. It was followed by *C. althaeoides*, the value obtained was smaller to the results cited by Aly Ahmed et *al*. (2014), for leaf, flower, and stem (12.46±0.21, 15.2±0.29 and 17.2±0.37 % DM, respectively). By assumption, this difference could be due to the harvest region which is in direct relation with the mineral composition of plants. While *C. arvensis* recorded the higher amount, succeeded by *E. vulgare*. For both species we have not found references revising the literature. For the rest of species, the ash amount obtained for *E. spinosa* was lower to that (33.7%) cited by Shaltout et *al*. (2008). As for *M. sylvestris*, the value obtained was slightly higher comparing to that (13.2±1.4 %) reported by Tunçtürk et *al*. (2008) and also to those mentioned by Barros et *al*. (2010) for leaves and flowers (13.53±0.11 and 10.54±0.30 %, respectively). *M. rugosa* had an ash amount slightly lower comparing with the value (13.19 %) provided by (Dougall and Bogdan, 1966). As for *O. pes-caparae*, our results were similar to those obtained by Edrah et *al*. (2018) (15.5 %). The previous differences might be due to the edaphic characteristics which varied mainly.

Regarding the CP composition, *R. peregrina*, registered the lowest amount, followed by *A. arvensis*, *O. pes-capreae* and *I. viscosa*. For all of them we have not found results in the literature to compare. Whereas *Ch. coronarium* and *W. frutescens* presented the highest amount. In the present study, the CP value obtained for *Ch. coronarium* were higher than that (12.53 % DM) obtained by Cabiddu et *al*. (2006). Also, it was higher than those mentioned by Valente et *al*. (2003) for early and late flower (58 and 87 g kg^{-1} DM, respectively). These dissimilarities could be due to the abiotic conditions. For *W. frutescens*, there was no previous results in the literature. Regarding the rest of species, the CP content obtained for *M. sylvestris* was higher in comparison to the amount (8.57±0.26) registered by Tunçtürk et *al*. (2018). For *M. rugosa* the CP content obtained was similar to that (21.53 %) reported by Dougall and Bogdan (1966). As for *S. arvensis*, the CP content was higher to that (7.7±0.101%) mentioned by Kamalak et *al*. (2005) at late maturity. Assuming that these variations might be affected to the plant material.

For ADF, A. *arvensis* showed the lowest content, *W. frutescesns* and *Ch. coronarium* were the following in the rank. For *Ch. coronarium*, the ADF value was slightly lower than that (19.12 % DM) cited by Cabiddu et *al*. (2006). As for *W. frutescesns*, we have not found results in the literature. In contrast, *R. peregrina* marked the highest amount, followed by *H. helix* for both species, there was no references in the bibliography. Concerning the other species, the ADF amount registered for *S. arvensis* was lower to that (65.8±0.372%) mentioned by Kamalak et *al*. (2005) at late maturity which might be due to the stage of maturity of the species. For *M. rugosa*, the value obtained was slightly higher than that (208 g kg^{-1}) previously cited by Porqueddu (2001) for the same species (cultivar Paraponto).

As for the NDF amounts, *C. arvensis* and *Ch. coronarium* registered the lowest amount, succeeded by *A. arvensis*. For *C. arvensis* and *A. arvensis*, there was no references in the literature. While for *Ch. coronarium*, the NDF amount was lower than those cited by Cabiddu et *al*. (2006) (29.56 % DM) and Sulas et *al*. (1999) (22.9±2.1 % DM) that could be due to the abiotic parameters thus representing a determined factors. Whereas *R. peregrina* revealed the higher amount, and in the next ranking, *M. rugosa* and *H. helix* were mentioned. For these species we have not found previous references in the literature. From the rest of species, *S. arvensis* presented an NDF amount lower than that (74.1±0.350%) cited by Kamalak et *al*. (2005) at late maturity which could be due to the stages of harvest. *M. rugosa* had an NDF content similar to that (302 g kg $^{-1}$) previously published by Porqueddu (2001) for the same species (cultivar Paraponto).

Considering total sugars, *Ch. coronarium* registered the less value, *W. frutescens*, *C. arvensis*, *E. vulgare* and *E. spinosa* were in the next rank. While *A. arvensis* was the rich one, followed by *Ch. segetum*. For all these species, there was no references in the literature. As regards to the other species, *C. althaeoides* recorded a lower total sugars value comparing to those obtained by Aly Ahmed et *al*. (2014) for flower, leaf, and stem (24.6±0.18, 27.2±0.22 and 19.3±0.24%, respectively). The sugars amount obtained for *S. arvensis* was higher to those cited by De Oliveira et *al*. (2019) for this species raw and cooked (4.00±0.74, 3.20±1.31 g. 100 g^{-1}, respectively), where these differences could be due to the harvest regions.

For fats, five species (*E. spinosa*, *E. vulgare* L., *R. peregrina*, *C. althaoides* and *C. arvensis*) revealed the less amounts, while *H. helix* presented the higher amount, followed by *F. capreolata*. For all these species, there was no references in the literature. For the rest of species, the fats amount recorded for *M. sylvestris* was similar to that obtained for leaves and flowers (2.76±0.40 and 2.84±0.37 %, respectively), as reported by Barros et *al*. (2010). For *S. arvensis* the value obtained was also similar to the lipids content (1.90±0.68 g 100 g^{-1}) for this species raw as mentioned by De Oleveira et *al*. (2019). As for *E. vulgare* the fats amount was very lower comparing to the total lipids content (35.58±0.09%) obtained for seeds in a previous study conducted by Nogala-Kalucka (2010). Additionally, *I. viscosa* presented a fats content lower to the lipids content (14.38 %) obtained for leaves in the study conducted by Salim et *al*. (2017). Also *M. rugosa* recorded a lower fats amount comparing with the ether extract concentration (3.07%) reported by Dougall and Bogdan (1966).

As regards to the NDFD, *H. helix* was the less digestive, followed by *S. arvensis*, while *O. pes-capreae* was the most digestive species, succeeded by *C. arvensis* and *W. frutescens*. From the rest of species, the NDFD obtained by *Ch. coronarium* was lower to the *in vitro* DM digestibility (85.7±0.2%) mentioned by Sulas et *al*. (1999). Also, the NDFD was lower to the organic matter digestibility reported for *S. arvensis* at early flowering, mid-flowering, and late maturity (72.4, 69.9 and 60.0 %, respectively) in the study carried out by Kamalak et *al*. (2005).

4.2. Mineral contents

For the mineral composition, we will discuss our results with similar works already mentioned in the literature on one side and on the other side by referring to the recommendations of NRC for mineral requirements for ruminants. In general, the most weeds studied can satisfy the mineral requirements of ruminants.

4.2.1. Major minerals

Regarding the phosphorus, *A. baetica* was the less rich, followed by *A. arvensis*, while *H. helix* was the rich one, succeeded by *S. arvensis*. There was no references for these species, except for *S. arvensis* where the value obtained was smaller than those registered by Lenzi et *al*. (2019) (54.08 ±11.56 mg 100 g^{-1} FW) and De Oliveira et *al.* (2019) (451.0±43.0 mg 100g^{-1}). Concerning the rest of the species, *F. capreolata*, showed a higher phosphorus amount than that (0.25% DM, 2.5 g kg^{-1}DM) mentioned by Baillie et *al.* (2018), that may be due to the environmental factors. As for *M. sylvestris*, the P amount was also higher comparing to the values (0.25±0.025%, 2.50±0.25 g kg^{-1}) reported by Tunçtürk et *al*. (2018) nothing that the P concentration is conditioned by the species and the stage of maturity. While for *O. pes-caprae*, the P amount obtained was similar to that (0.29% DM, 2.9 g kg^{-1}DM) cited by Baillie et *al*. (2018).

Regarding the potassium, *Ch. coronarium* was the richest species, followed by *C. arvensis* and then *O. pes-caprae*. In the literature there was no data for *C. arvensis*. For *Ch. coronarium* the value obtained herein was higher than that (1.80±0.09) reported by Akrout et *al*. (2010), but lower to that (620 mg 100 g^{-1}) published by Wills et *al*. (1984). As for *O. pes-caprae,* the concentration obtained was similar to that (24.4 g kg^{-1}) reported by Baillie et *al.* (2018). While *S. arvensis* and *I. viscosa* were the less rich. The amount of K in *I. viscosa* was not found in previous studies. For *S. arvensis* the value obtained was very smaller comparing to those reported by De Oliveira et *al*. (2019) for the same species raw and cooked (3413.0±585.5 and 2519.0±278.7 mg 100 g^{-1}, respectively). It was also lower to that (435.65 ± 11.47 mg 100 g-1) mentioned by Paula Filho et *al*. (2018) and to that (5.54±0.84 mg g^{-1}) cited by Başyiğit et *al*. (2020) for wild mustard seeds.

From the rest of species, *M. syslvestris* presented a K value slightly higher to that (1.49 g 100 g^{-1}) reported by Civelek and Balkaya (2013) and slightly lower to that (21.17±1.14 g kg^{-1}) obtained by Tunçtürk et *al*. (2008). But very lower comparing with those (4067±120 and 2033±33 mg 100 g^{-1}, respectively) reported for leaves and immature fruits in the study realized by Romojaro et *al*. (2013).

Concerning the calcium content, the richest species was found to be *Ch. coronarium*. The value obtained was higher to that (1.65±0.11g 100 g^{-1}) reported by Akrout et *al*. (2010). In the next ranking, *C. arvensis* and *E. vulgare* were found. There was no previous data reported in the literature for these species. In contrast, *S. arvensis* was the less rich, where the concentration obtained was lower in comparison to those cited by Lenzi et *al*. (2019) (54.08 ±11.56 mg 100 g^{-1} FW), and De Oliveira et *al*. (2019) (451.0±43.0 mg 100g^{-1}). *C. althaeoides,* *O. pes-caprae, M. rugosa* and *H. helix* were the following in the ranks. For *C. althaeoides* and *H. helix,* there was no reported data. As for *O. pes-caprae*, the Ca amount recorded was lower than that (10.9 g kg^{-1} DM) cited by Baillie et *al*. (2018). For *M. rugosa*, the value obtained was also smaller comparing to that (1.06 % DM) mentioned by Dougall and Bogdan (1966). For the rest of species, *F. capreolata* registered a lower value comparing to that (21.4 g kg^{-1} DM) obtained by Baillie et *al*. (2018). As for *M. syslvestris* the concentration of Ca measured in the current study was similar to that (1.54 g 100 g $^{-1}$) obtained in the study conducted by Civelek and Balkaya (2013) but lower to that (46.87±1.16 g kg^{-1}) published by Tunçtürk et *al*. (2008).

The concentration of Mg varied from 0.51±0.022 % and 0.42±0.001 % for *E. spinosa* and *C. arvensis* to 0.20±0.001 % and 0.15±0.002 % for *E. vulgare* and *A. arvensis*. There were no previous published results for all these species. Among the rest of species, *F. capreolata* registered a high Mg content in comparison with the results reported by Baillie et *al*. (2018) (2.2 g kg^{-1} DM). As for *M. sylvestris*, our results were close to those reported by Civelek and Balkaya (2013) (0.21 g 100 g $^{-1}$), but lower to that cited by Tunçtürk et *al*. (2008) (10.61±1.08 g kg $^{-1}$). For *O. pes-caprae*, the Mg amount obtained herein was also smaller to that previously mentioned by Baillie et *al*. (2018) (4.1 g kg^{-1} DM). In addition, *S. arvensis* presented a magnesium concentration lower than those cited by Lenzi et *al*. (2019) (75.99 ±13.21 mg 100 g^{-1} FW) and De Oliveira et *al*. (2019) (267.2±8.6 mg 100 g^{-1}), and also lower to the value reported for seeds (1.01 ± 0.10 mg g^{-1}) by Başyiğit et *al*. (2020).

4.2.2. Trace minerals

Among the studied weed species, *Ch. segetum* showed the high level of Fe, followed by *Ch. coronarium* and then *I. viscosa* L. For *I. viscosa*, we have not found information in the literature. As for *Ch. coronarium*, the value obtained herein was clearly higher than that (8.104± 0.857 µg g^{-1} DW) cited by Bordoloi et *al*. (2016). In contrast, *S. arvensis* and *H. helix* had the lowest Fe concentrations. For *S. arvensis*, the amount obtained was higher than that (5.03 ±4.31 mg 100 g^{-1} FW) found by Lenzi et *al*. (2019) and to that (62.2±22.2 mg 100g^{-1}) cited by De Oliveira et *al*. (2019).

For the rest of species, our findings for iron were inferior comparing with previously published results. The value obtained for *F. capreolata* was smaller than that (264 mg kg $^{-1}$ DM) mentioned by Baillie et *al*. (2018). Also, the Fe content registered for *M. sulvestris* was lower comparing to those reported by Tunçtürk et *al*. (2008) (366.26±1.12 mg kg $^{-1}$) and Civelek and Balkaya (2013) (613.24 mg kg $^{-1}$). Moreover, *O. pes-caprae* recorded a low amount in comparison to that (270 mg kg $^{-1}$ DM) previously cited by Baillie et *al*. (2018). For

E. vulgare the value obtained herein was also lower to those reported by Bošković et *al*. (2018) for petroleum ether and acetone extracts (62.35 and 89.00 µg g^{-1}, respectively), but higher to those mentioned for ethanol, ethyl acetate and chloroform extracts (26.12, 21.34 and 42.34 µg g^{-1}, respectively).

I. viscosa had the highest Mn concentration, followed by *Ch. coronarium*. While *C. althaeoides* had the lowest concentration succeeded by *E. vulgare*. For all these species there was no previous published data regarding the Mn concentration.

Regarding the rest of species, our results for Mn were lower with respect to previously published data. The value recorded for *F. capreolata* was lower than that (399 mg kg^{-1} DM) obtained by Baillie et *al*. (2018). The Mn value registered for *M. sylvestris* was also smaller in comparison to those cited by Tunçtürk et *al*. (2008) (79.54±4.77 mg kg^{-1}) and Civelek and Balkaya (2013) (37.84 mg kg^{-1}).

Furthermore, the Mn concentration in *O. pes-caprae* was lesser to the amount (229 mg kg^{-1} DM) reported by Baillie et *al*. (2018). Also, for *S. arvensis,* the Mn concentration obtained was smaller in comparison to those found by Lenzi et *al*. (2019) (3.48 ±1.61 mg 100 g^{-1} FW) and De Oliveira et *al*. (2019) (4.8±1.6 mg 100g^{-1}). Also, for *E. vulgare* the Mn amount obtained was lower to those reported by Bošković et *al*. (2018) for ethanol, petroleum ether and acetone extracts (49.76, 56.21 and 82.00 µg g^{-1}, respectively) but higher to those mentioned for ethyl acetate and chloroform extracts (15.32 and 9.88 µg g^{-1}, respectively).

E. spinosa recorded the higher level of Zn followed by *C. arvensis.* The Zn content of both species have not been reported in the literature. Whereas in the present study this trace mineral has not been detected in *O. pes-caprae*. However, the concentration of Zn in *O. pes-caprae* was reported in previous results published by Tunçtürk et *al*. (2008) (3.67±0.49 mg 100 g^{-1} DW) and Baillie et *al*. (2018) (46 mg kg^{-1} DM). For the other species, *F. capreolata* had a slightly lower concentration in comparison with that (59 mg kg^{-1} DM) cited by Baillie et *al*. (2018). As for *M. syslvestris* the Zn amount obtained in the present investigation was close to that (45.86±6.05 mg kg^{-1}) mentioned by Tunçtürk et *al*. (2008) and higher to that (26.38 mg kg^{-1}) measured by Civelek and Balkaya (2013). For *S. arvensis,* the zinc concentration obtained was lower comparing to those cited by Lenzi et *al*. (2019) (0.46 ±0.09 mg 100 g^{-1} FW) and De Oliveira et *al*. (2019) (4.4±0.8 mg 100g^{-1}).

For *E. vulgare,* the Zn content obtained in the present study was lower to those mentioned by Bošković et *al*. (2018) for ethanol, ethyl acetate, chloroform, petroleum ether and acetone extracts (17.69, 32.17, 28.47, 28.01 and 10.39 µg g^{-1}, respectively).

4.2.3. Ruminants' requirements for minerals and Maximum Tolerable Levels (MTL)

In this step, the NRC recommendations for mineral requirements will be taken into consideration as well as the Maximum Tolerable Levels (MTL). Table VII.4 gives mineral requirements and MTL for each mineral for ruminants (beef cattle, dairy cattle, sheep, and goats) as reported by the NRC.

Table VII.4. Mineral requirements in total diet for ruminants and Maximum Tolerable Level (MTL) for ruminants.

a. Mineral requirements in total diet for ruminants

Class of animal	**Major minerals (%)**				**Trace minerals (mg kg $^{-1}$)**		
	Ca	Mg	K	P	Zn	Mn	Fe
Beef cattle	0.60-0.80	0.10-0.20	0.60-0.70	0.6-0.70	30	20-40	50
Dairy cattle	0.60-1.00	0.07-0.10	0.65-1.12	0.40-0.76	40	40	50-100
Sheep	0.20-0.82	0.12-0.18	0.50-0.80	0.16-0.38	20-33	20-40	30-50
Goats	0.03-0.80	0.18-0.40	0.80-1.50	0.25-0.40	40-500	40-1000	35-95

b. Maximum Tolerable Level (MTL) of minerals for ruminants

Class of animal	**Major minerals (%)**				**Trace minerals (mg kg $^{-1}$)**		
	Ca	Mg	K	P	Zn	Mn	Fe
Beef cattle	2	0.40	3	1	500	1000	1000
Dairy cattle	2	0.50	3	1	500	1000	1000
Sheep	1.50	0.60	2	0.60	750	1000	500
Goats	1.50	0.60	2	0.60	-	2000	500

-: not found. All data reported in the table were collected from the National Research Council (NRC, 2001, 2000, 1985, 1981).

According to the results obtained in the current study for major minerals, the concentration of calcium ranged from 0.44 to 2.16%, therefore, all weed species had Ca concentrations that can meet the needs of ruminants (Table VII.4a). In contrast, all species presented Ca contents inferior to the MTL for sheep and goats, as for beef and dairy cattle, one species (*Ch. coronarium)* had a value (2.16%) slightly higher than the limit mentioned for both (2%) (Table VII.4b).

The Mg contents were from 0.15 to 0.51%, thus, all species had Mg values that correspond to the levels required for all ruminants (Table VII.4a). As for the MTL, all weeds had Mg levels lower than that tolerable except for *C. arvensis* (0.42%) and *E. spinosa* (0.51%) which presented Mg concentrations slightly higher than those acceptable for beef and dairy cattle (0.40 and 0.50 %, respectively) (Table VII.4b).

Potassium varied from 0.39 to 3.43 %, all the weeds can satisfy the K requirements of ruminants (Table VII.4a) except *S. arvensis,* which presented a low K content (0.39 %), nothing that in the present investigation we have used the seeds of this species and not the whole plant. Apart from *C. arvensis* (3.21%) and *Ch. coronarium* (3.43%), that presented values slightly higher than the MTL of beef and dairy cattle (3% for both), all the weed species were under the tolerable limits. (Table VII.4b),

For phosphorus, the amounts obtained were between 0.18 and 0.55%, the totality of weeds meet the requirements of ruminants (Table VII.4a). All the weeds presented a secured P levels, inferior to the MTL for all animals cited previously (Table VII.4b).

For trace minerals, the Fe concentrations ranged between 24.11 and 130.93 mg kg $^{-1}$. All weeds can satisfy the needs of ruminants (Table VII.4a), except for *M. rugosa*, *S. arvensis* and *H. helix* which had Fe values (34.58, 24.11 and 25.91 mg kg^{-1}, respectively) lower than the minimum requirements of goats. All the studied weeds registered Fe levels inferior to the MTL for all the cited animals (Table VII.4b).

Regarding the manganese, results varied from 16.84 to 78.54 %. With the exception of *C. althaeoides* and *E. vulgare,* which had Mn levels lower to the minimum requirements of beef cattle and sheep, all the weed species meet the needs of ruminants (Table 4a). In addition, all the investigated weeds recorded Mn levels inferior to the MTL for all the cited animals (Table 4b).

As for zinc, the amounts obtained were from 00.00 to 57.97 mg kg $^{-1}$. Eight species (*C. althaeoides* and *E. vulgare*, *M. rugosa*, *R. peregrina*, *A. baetica*, *Ch. segetum*, *H. helix* and *W. frutescens*) presented low concentrations that cannot satisfy the requirements of the cited animals (Table 4a). As for *O. pes-caprae*, zinc have not been detected. While for the rest of species, they can meet the needs of the mentioned animals. The totality of weeds had Mn levels inferior to the MTL for all the cited animals (Table 4b).

5. Conclusion

In the current study, eighteen weed species growing in citrus orchards were valorised from the forage quality viewpoint. The results distinguished differences among the studied weeds according to their minerals and nutritive value.

Among all the species, *S. arvensis* and *C. althaeoides* showed the best OM content while *C. arvensis* and *E. vulgare* had the highest ash amount. *Ch. coronarium* and *W. frutescens* presented a good CP content. *R. peregrina* and *H. helix* possessed the highest ADF and NDF contents also *M. rugosa* contained a good NDF level. *A. arvensis* and *Ch. coronarium* contained the best total sugars level, whereas *H. helix* and *F. capreolata* had the highest fats content. Finally, *O. pes-caprae*, *C. arvensis* and *W. frutescens* exhibited the best NDF-digestibility.

For the mineral composition, *Ch. coronarium* had the highest K and Ca levels while *E. spinosa* presented the higher Mg and Zn concentrations. *Ch. segetum* was the richest species regarding iron and *I. viscosa* was the richest one concerning Mn whereas *H. helix* recorded the higher P level. These results suggest the potential use of these weeds for ruminants feeding in order to reduce fodder costs for breeders.

Generally, weeds are neglected and underutilized whereas they possess an economic value, and they could play a vital role in animal-vegetal association. The results obtained revealed their important nutritive value and their rich mineral composition, which suppose the possible use of weeds to feed animals and ruminants more specifically, instead of destroying them. Further studies need to be realized, in order to evaluate the toxicity of these species for a secure use.

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General Conclusion

This thesis was focused on three main parts, the first concerned with the evaluation of the impact of mechanical and chemical weeding on the floristic diversity of citrus orchards, the second part was interested in the evaluation of the antioxidant activities (DPPH, ABTS and chelating power) and the phenols and flavonoids content of nineteen weed species (representing 20 % of the total weed flora for a general representation of the citrus associated flora, which was composed by 88 weeds) and the third part treated the evaluation of the nutritive value and the mineral composition of the same nineteen weeds. The second and third section are part of a context of valorization of weeds to be used for beneficial uses instead of eliminating them.

The first part was based on 168 floristic surveys carried out during 2016-2017, before and after each weeding (mechanical and chemical) in comparison to a control without weeding. A total of 88 species, belonging to 71 genera and 30 botanical families were identified. Poaceae, Asteraceae, Apiaceae and Fabaceae were the dominant families. These four botanical families alone represented 55% of the total flora. Whereas, dicotyledonous, therophytes and Mediterranean elements were the most represented.

The results showed that the method of weeding, influenced the floristic composition of the weed flora of the citrus orchard studied. This affected mainly the weed flora's composition and the frequency of species composing it. Over-all, four species occupied the first places; they were the most common perennial weeds present in Algeria: *Oxalis pes-caprae* L., *Convolvulus arvensis* L., *Cynodon dactylon* (L.), and *Cyperus rotundus* L. In agronomy, the notion of biological type is closely related to the cultural context, thus representing an important point to consider. Superficial and irregular weeding favors perennial weeds. The therophytes are adapted to productive and disturbed habitats because they are the only ones capable of growing between two weeding operations. Therefore, farming techniques affect the composition, evolution, and dynamics of the weed flora.

The methods practiced controlling weeds (mainly mechanical and chemical control) are not the only factors that affect the floristic composition and the dynamic of the weed flora of citrus orchards. It is also subjected to other agricultural practices. Thus, this part of study needs to be followed by other studies to illuminate this point. It is recommended to continue the current study for at least three years to see if there is reproducibility, expand the study area and follow it with an inquiry to evaluate the situation of citrus growing in Algeria on all plans, and more specifically on the use of chemical inputs. Additionally, the exploitation of the allelopathic effect possessed by many plants may be a very promising alternative method of weed control.

The second part was founded on the evaluation of the total phenol and flavonoid contents of the aqueous extracts of fifteen weed species growing in citrus orchards on the Northwest of Algeria (considering two main factors, variability in botanical families and divergence in organ tested between them) and their corresponding antioxidant activities (DPPH and ABTS scavenging activities and chelating metal ions capacity).

Among the weeds studied, it was remarkable that four species (*Inula viscosa* L., *Sideritis montana* L., *Rubia peregrina* L. and *Reichardia tingitana* L.) were characterized by their richness in phenols and flavonoids, being also them considered as good DPPH scavengers. In addition, three other species (*Malva sylvestris* L., *Emex spinosa* (L.) and *Medicago rugosa* Desr.) were highlighted as good chelating agents. The results obtained suggest the potential capacity of those weeds which might be valorized as a source of natural secondary metabolites and natural antioxidants instead of eliminating them. They could be used in food, cosmetic and pharmaceutical industries in order to avoid the side effects of synthetic antioxidants.

This research part needs to be followed by further studies in which other solvents, differing in their extraction capacity, will be evaluated to determine the best option for extracting different secondary metabolites and testing other antioxidant activities in order to get appropriate comparison between extracts obtained from diverse weed material. In addition to that, mixing extracts from different weeds, might also be a strategic point to be addressed for getting at the same time good scavengers and chelators. Also, other biological activities (such as antidiabetic, anti-inflammatory, and anticancer) could be studied for elucidating the potential value of those weed species as a resource to valorize in the future for the agriculture sector. Furthermore, a study related to the toxicity of the fifteen weed species remains necessary and preliminary before exploiting them.

The third part allowed us to determine the mineral composition (major and trace minerals) and the nutritive value (OM, ash, CP, sugars, ADF, NDF, fats and NDFD) of eighteen weed species, growing in citrus orchards on the Northwest of Algeria (considering two main factors, variability in botanical families and divergence in organ tested between them).

Among the eighteen weed species studied, *Sinapis arvensis* and *Convolvulus althaeoides* presented the best OM content while *Calendula arvensis* and *Echium vulgare* possessed the highest ash amount. *Chrysanthemum coronarium* and *Withania frutescens* had a good CP content. *R. peregrina* and *Hedera helix* showed the highest ADF and NDF contents, additionally *Medicago rugosa* contained a good NDF level. *Anagallis arvensis* and *Ch. coronarium* presented the best total sugars levels, while *H. helix* and *Fumaria capreolata* had the highest fats contents. Finally, *Oxalis pes-caprae, C. arvensis* and *W. frutescens* exhibited the best NDF-digestibility.

About the mineral composition, *Ch. coronarium* showed the highest K and Ca levels while *E. spinosa* presented the higher Mg and Zn concentrations. For Fe, *Chrysanthemum segetum* was the richest species. Also, *I. viscosa* had the best Mn content and *H. helix* recorded the higher P level. Moreover, the most of the investigated weed species could satisfy the mineral requirements of ruminants (dairy cattle, beef cattle, sheep, and goats) as recommended by the NRC, being also below maximum tolerable levels for feeding those animals. These results suppose the potential use of these species for animal feeding in order to reduce fodder costs for breeders.

This part of investigation could be pursued by additional studies in which, other more precise parameters to evaluate the nutritional value of a forage can be taken into consideration, such as energy, the different sugars, and fatty acids composition, also a comparison between the nutritional value of fresh and preserved weeds can be considered. As cited previously, the study of the toxicity of the fifteen weed species stills essential and primordial before any uses of them for these purposes.

List of references

Aazza, S., Lyoussi, B., Miguel, M.G. (2011a). Antioxidant activity of some Moroccan hydrosols. *J. Med. Plants Res.*, 5, 6688-6696. https://doi.org/10.5897/JMPR11.1176

Aazza, S., Lyoussi, B., Miguel, M.G. (2011b). Antioxidant and antiacetylcholinesterase activities of some commercial essential oils and their major compounds. *Molecules*, 16, 7672-7690. https://doi.org/10.3390/molecules16097672

Abbas, J.A., Al-Saleh, F.A. (2002). Medicinal Plants of Bahrain. Univ. of Bahrain (in Arabic).

Abd El-Ghani, M., Soliman, A., Hamdy, R., Bennoba, E. (2013). Weed flora in the reclaimed lands along the northern sector of the Nile Valley in Egypt. *Turkish Journal of Botany*, 37(3), 464-488. https://doi.org/10.3906/bot-1205-11

Abdel Aziz Mohamed, M. (2012). Weed Survey on Wheat in the Northern State, Sudan. U. of K. *J. Agric. Sci.*, 20(2), 26-43.

Abdel Aziz Mohamed, M., Babiker Mohamed Mahgoub, B.M., Eltom Elamin, S. (2015). Chemical weed control in garlic (*Allium sativum* L.) in Dongola Locality, Northern State, Sudan. SUST. *Journal of Agricultural and Veterinary Sciences* (SJAVS), 16(2), 109-118.

Abdelkrim, H. (1995). Contribution à la connaissance des groupements de mauvaises herbes des cultures du secteur algérois : Approche syntaxonomique et phénologique. Thes. Doc. en Sc., Univ. Paris-Sud, Centre d'Orsay, 151pp.

Abdelkrim, H. (2004). Contribution à l'étude écologique et phytosociologique des adventices des cultures et des jachères dans le secteur phytogéographique de l'Algérois (Algérie). *Phytocoenologia*, 34(2), 287-326. https://doi.org/10.1127/0340-269X/2004/0034-0287

Abdelmageed, M., Ali, M., Hamadnalla, H. M. Y. (2020). Eco-taxonomic Study of the Weeds of Um Algura Area- Western Gezira State, Sudan. *Research and Reviews: Journal of Ecology and Environmental Sciences*, 8(2), 11-16.

Abudunia, A.M., Marmouzi, I., Faouzi, M.E.A., Ramli, Y., Taoufik, J., El Madani, N., Essassi, E.M., Salama, A., Khedid, K., Ansar, M., Ibrahimi, A. (2016). Anticandidal, antibacterial, cytotoxic and antioxidant activities of Calendula arvensis flowers. *Journal de Mycologie Medicale*, 655, 1-8. http://dx.doi.org/10.1016/j.mycmed.2016.11.002

Achenbach, H., Fischer, A. (1997). 6-O-beta-D-glucoside of aristolochic acid IIIa and other components from the roots of Aristolochia baetica**,** *Planta Med.*, 63 (6), 579. http://doi.org/10.1055/s-2006-957777

Addis, M., Pinna, G., Molle, G., Fiori, M., Spada, S., Decandia, M., Scintu, M. F., Piredda, G., Pirisi, A. (2006). The inclusion of a daisy plant (*Chrysanthemum coronarium*) in dairy sheep diet: 2. Effect on the volatile fraction of milk and cheese. *Livestock Science*, 101(1-3), 68-80. https://doi.org/10.1016/j.livprodsci.2005.09.009

Agelet, A., Valles, J. (2001). *Ethnopharmacology*, 77, 57.

Ahmed, E., Arshad, M., Khan, M. Z., Amjad, M. S., Sadaf, H. M., Riaz, I. (2017). Secondary metabolites and their multidimensional prospective in plant life Secondary metabolites and their multidimensional prospective in plant life. *Journal of Pharmacognosy and phytochemistry*, 6(2), 205-214.

Aikawa, J.K. (1981). Magnesium: its biologic significance. CRC Press, Boca Raton, Florida.

Aimé, S. (1991). Etude écologique de la transition entre les bioclimats subhumides, semi-aride et aride dans l'étage thermo-méditerranéen du Tell oranais (Algérie nord-occidentale). Thes. Doc., Univ. Aix-Marseille III.

Akbar, M., Sherazi, I. N., Iqbal, M. S., Khalil, T., Waqas, H. M. (2020). Antibacterial and antioxidant activities of slender amaranth weed. *Planta Daninha*, 38, 1-8. https://doi.org/10.1590/s0100-83582020380100006

Akgunlu, S.B., Sekeroglu, N., Koca-Caliskan, U., Ozkutlu, F., Ozcelik, B., Muhittin Kulak, M., Gezici, S. (2016). Research on selected wild edible vegetables: Mineral content and antimicrobial potentials. *Annals of Phytomedicine*. 5(2), 50-57.

Akhtar, N., Ihsan-ul-Haq, Mirza, B. (2018). Phytochemical analysis and comprehensive evaluation of antimicrobial and antioxidant properties of 61 medicinal plant species. *Arabian Journal of Chemistry*, 11(8), 1223-1235. https://doi.org/10.1016/j.arabjc.2015.01.013

Akrout, A., El Jani, H., Zammouri, T., Mighri, H., Neffati, M. (2010). Phytochemical screening and mineral contents of annual plants growing wild in the southern of Tunisia. *Journal of Phytology*, 2(1), 34-40.

Alarcón de la Lastra, C., López, A., Motilva, V. (1993). Gastroprotection and prostaglandin E2 generation in rats by flavonoids of *Dittrichia viscosa*. *Planta Medica.*, 59(6), 497-501. https://doi.org/10.1055/s-2006-959747

Albrecht, H. (2003). Suitability of arable weeds as indicator organisms to evaluate species conservation effects of management in agricultural ecosystems. *Agriculture, Ecosystems and Environment*, 98, 201-211. https://doi.org/10.1016/S0167-8809(03)00081-1

Al-Dissi, M.N., Salhab, A.S., Al-Hajj, H.A. (2001). Effect of *Inula viscosa* leaf extracts on abortion and implantation in rats. *Journal of Ethnopharmacology*, 77(1), 117-121. https://doi.org/10.1016/s0378-8741(01)00261-6

Alfy, T. El, Tantawy, M. E. El, Motaal, A. A., El, F., Gamal, Z., Culture, T. (2015). Pharmacological, biological study and GC / MS analysis of the essential oil of the aerial parts and the alcohol soluble fraction of the n. Hexane extract of the flowers of *Reichardia tingitana* L. *Canadian Journal of Pure and Applied Sciences*, 9(1), 3167-3175.

Alhasi, S. M., El-Barasi, Y. M., Rahil, R. O., Saaed, M. W. B. (2017). Weed Flora of the Barley (*Hordeum vulgare* L.) and Wheat (*Triticum aestivum* L.) fields in Al-Marj Plain Libya. *Continuous Research Online Library*, 1(1), 1-9. https://doi.org/10.28915/control.0005.1

Alhasi, S.M., El-Barasi, Y.M. (2017). The Weed Flora of some Crop Fields in Al- Oylia region, Al-Marj, Libya, Univ. of Benghazi, Faculty of Education Al Marj. *Global Libyan Journal*, 23, 1-11.

Ali-Shtayeh, M.S., Jamous, R.M., Abu Zaitoun, S.Y., Qasem, I.B. (2014). *In-vitro* screening of acetylcholinesterase inhibitory activity of extracts from Palestinian indigenous flora in relation to the treatment of Alzheimer's disease. *Functional Foods in Health and Disease*, 4(9), 381-400. https://doi.org/10.31989/ffhd.v4i9.149

Al-Laith, A.A., Alkhuzai, J., Freije, A. (2019). Assessment of antioxidant activities of three wild medicinal plants from Bahrain. *Arabian Journal of Chemistry*, 12(8), 2365-2371. https://doi.org/10.1016/j.arabjc.2015.03.004

Allison, J.P. and Cook, M. (1981). Oxalate degradation by microbes of the large bowel of herbivores: the effect of dietary oxalate. *Science*, 212, 675-676.

Al-Qahtani, S.M. (2019). Effect of soil properties on the diversity and distribution of weeds in citrus farms in Arid region. *Applied Ecology and Environmental Research*, 17(1), 723-732. https://doi.org/10.15666/aeer/1701_723732

Alsherif, E.A. (2020). Cereal weeds variation in middle Egypt: Role of crop family in weed composition. *Saudi Journal of Biological Sciences*, 27(9), 2245-2250. https://doi.org/10.1016/j.sjbs.2020.07.001

Aly Ahmed, F., Sharaf, M.A., Sofy, M.R., Elhaw, M.H. (2014). Phytochemical Analysis and Biological Activity of *Convolvulus althaeoides* and *Convolvulus stachydifolius* var. villosus. *Report and Opinion*, 6(7), 36-44.

Anderson, M., Markham, K.R. (2006). Flavonoids, chemistry, biochemistry, and applications. *Natural products from plants*. Edition Taylor et Francis Group, LLC. *Annual Review of Nutrition*, 22, 19-44.

Antunes, R., Franco, J.C., Moreira, I., Silva, E.B., Soares, C., Sousa Bastos, M.E., Vasconcelos, M.T. (2005). Flora and weed management in citrus orchards. Actas Reunión de la SEMh 2005. Lisbon, Portugal.

AOAC (1996). Official methods of analysis of the AOAC. Association of Official Analytical Chemists. Arlington. VA, USA.

Apak, R., Güçlü, K., Özyürek, M., Esin Karademir, S., Erçğ, E. (2006). The cupric ion reducing antioxidant capacity and polyphenolic content of some herbal teas. *International Journal of Food Sciences and Nutrition*, 57(5-6), 292-304. https://doi.org/10.1080/09637480600798132

Ashenafi, M., Dalga, D. (2014). Effect of herbicides on weed dynamics and yield and yield attribute of bread wheat (*Triticum aestivum* L.) in south eastern part of Ethiopia. *International Journal of Technology Enhancements and Emerging Engineering Research*, 2(4), 2347-4289.

Astley, S.B. (2003). Dietary antioxidants past, present and future. *Trends in Food Science and Technology*, 14, 93-98.

Austin, M.B., Noel, J.P. (2003). The chalcone synthase superfamily of type III polyketide synthases. *Natural Product Reports*, 20, 79-110.

Babali, B., Hasnaoui, A., Medjati, N., Bouazza, M. (2013). Note on the vegetation of the mounts of Tlemcen (Western Algeria): Floristic and phytoecological aspects. *Open Journal of Ecology*, 3(5), 370-381. https://doi.org/10.4236/oje.2013.35042

Badshah, L., Hussain, F. (2011). Farmers preferences and use of local fodder flora in Tank District, Pakistan. *African Journal of Biotechnology*, 10(32), 6062-6071. https://doi.org/10.5897/AJB10.1154

Bagnouls, F., Gaussen, H. (1953). Saison sèche et indice xérothermique. Doc. Carte porte. Vég. Art. 8. Toulouse. 47pp.

Bahloul, N., Bellili, S., Aazza, S., Chérif, A., Faleiro, M. L. (2020). Aqueous Extracts from Tunisian *Diplotaxis*: Phenol Content, Antioxidant and Anti-Acetylcholinesterase Activities, and Impact of Exposure to Simulated Gastrointestinal Fluids. *Antioxidants*, 5(12), 1-15. https://doi.org/10.3390/antiox5020012

Baião, N., Lara, L. (2005). Oil and fat in broiler nutrition. *Revista Brasileira de Ciência Avícola*, 7(3), 129-141. https://doi.org/10.1590/s1516-635x2005000300001

Baillie, C., Kaufholdt, D., Meinen, R., Hu, B., Rennenberg, H. (2018). Surviving Volcanic Environments-Interaction of Soil Mineral Content and Plant Element Composition. *Frontiers in Environmental Science*, 6(52), 1-13. https://doi.org/10.3389/fenvs.2018.00052

Bakchiche, B., Gherib, A., Aazza, S., Gago, C. Miguel, M.G. (2013). Antioxidant activities of eight Algerian plant extracts and two essential oils. *Industrial Crops and Products*, 46, 85–-96.

Balamurugan, B., Ramamoorthy, M., Kumar Mandal, R.S., Keerthana, J., Gopalakrishnan, G., Kavya, K.M., Kharayat, N.S., Chaudhary, G.R., Katiyar, R. (2017). Mineral an important nutrient for efficient reproductive health in dairy cattle. *International Journal of Science, Environment and Technology*, 6(1), 694-701.

Balamurugan, B., Ramamoorthy, M., Kumar Mandal, R.V., Keerthana, J., Gopalakrishnan, G., Kavya, K.M., Kharayat, N.S., Chaudhary, G.R., Katiyar, R. (2017). Mineral an important nutrient for efficient reproductive health in dairy cattle. *International Journal of Science, Environment and Technology*, 6(1), 694-701.

Balkan, S., Aydoğdu, H., Balkan, S., Aşkin, B., Ersoy, H. (2019). Evaluation of antioxidant and antifungal activities of several plants against agents of postharvest citrus sour rot and green mould rot. *Romanian Biotechnological Letters*, 24(5), 798-806. https://doi.org/10.25083/rbl/24.5/798.806

Bardaweel, S.K., Hudaib, M.M., Tawaha, K.A., Bashatwah, R.M. (2015). Studies on the *in vitro* antiproliferative, antimicrobial, antioxidant, and acetylcholinesterase inhibition activities associated with *Chrysanthemum coronarium* L. essential oil. *Evidence-Based Complementary and Alternative Medicine*, 2015, 6, Article ID 79083. https://doi.org/10.1155/2015/790838

Bar-Eyal, M., Edna, S., Spiegel Yitzhak, S. (2006). Nematocidal activity of *Chrysanthemum coronarium*, *European Journal of Plant Pathology*, 114, 427-433.

Barralis, G. (1976). Méthodes d'étude des groupements adventices des cultures annuelles, application a la cote d'or. 5 e coll. Inter. Sur l'écologie et la biologie des mauvaises herbes. Tome I. Dijon, 55-68pp.

Barros, L., Carvalho, A.M., Ferreira, I. (2010). Leaves, flowers, immature fruits, and leafy flowered stems of *Malva sylvestris*: A comparative study of the nutraceutical potential and composition. *Food and Chemical Toxicology*, 48(6), 1466-1472. https://doi.org/10.1016/j.fct.2010.03.012

Bar-Shalom, R., Bergman, M., Grossman, S., Azzam, N., Sharvit, L., Fares, F. (2019). *Inula viscosa* L. extract inhibits growth of colorectal cancer cells *in vitro* and *in vivo* through induction of apoptosis. *Frontiers in Oncology*, 9, 227. https://doi.org/10.3389/fonc.2019.00227

Basile, A., Senatore, F., Gargano, R., Sorbo, S., Del Pezo, M., Lavitola, A., Ritieni, A., Bruno, M., Spatuzzi, D., Rigano, D., Vuotta, M.L. (2006). Antibacterial and antioxidant activities in *Sideritis italica* (Miller) Greuter et Burdet essential oils. *Journal of Ethnopharmacology*. 107(2), 240-248. https://doi.org//10.1016/j.jep.2006.03.019

Başyiğit, B., Alaşalvar, H., Doğan, N., Doğan, C., Berktaş, S., Çam, M. (2020). Wild mustard (*Sinapis arvensis* L.) parts: compositional analysis, antioxidant capacity and determination of individual phenolic fractions by LC–ESI–MS/MS. *Food Measure*, 14, 1671-1681. https://doi.org/10.1007/s11694-020-00415-2

Batovska, D., Parushev, S., Stamboliyska, B., Tsvetkova, I., Ninova Najdenski, H. (2009). Examination of growth inhibitory properties of synthetic chalcones for which antibacterial activity was predicted. *European journal of Medicinal chemistry*, 44(5), 2211-2218.

Bellakhdar, J. (1997). La pharmacopée marocaine traditionnelle : médecine arabe ancienne et savoirs populaires. Paris, France: Ibis Press, 676pp.

Bellakhdar, J. (2005). The Traditional Moroccan Pharmacopoeia, Medicine and Arabic.

Benabadji, N., Bouazza, M. (2000). Contribution à une étude bioclimatique de la steppe à *Artemisia herba-alba Asso*. Dans l'Oranie (Algérie occidentale). *Sécheresse*, 11(2), 117-123.

Benavente-Garcia, O., Castillo, J., Lorente, J. (2000). Antioxidant activity of phenolics extracted from *Olea europaea* L. leaves, *Food Chemistry*, 68, 457-62.

Bender, R.W., Cook, D.E., Combs, D.K. (2016). Comparison of in situ versus in vitro methods of fiber digestion at 120 and 288 hours to quantify the indigestible neutral detergent fiber fraction of corn silage samples. *Journal of Dairy Science*, 99, 5394-5400.

Benest, M. (1985). Evolution de la plateforme de l'ouest algérien et du nord-est marocain au cours du Jurassique supérieur et du début du Crétacé : Stratigraphie, milieu de dépôt et dynamique sédimentaire. Thes. Doc. Géologie, Univ. Lyon.

Benso, B., Franchin, M., Massarioli, A.P., Paschoal, J.A.R., Alencar, S.M., Franco, G.C.N., Rosalen, P.L. (2016). Anti-inflammatory, anti-osteoclastogenic and antioxidant effects of *Malva* *sylvestris* extract and fractions: *In vitro* and *in vivo* studies. *PLoS ONE*, 11(9), 1-19. https://doi.org/10.1371/journal.pone.0162728

Bérdy J. (2005). Bioactive microbial metabolites. *The Journal of Antibiotics*, 58(1), 1-26.

Berger, L.L. (1987). Salt and Trace Minerals for Livestock, Poultry and Other Animals. prior editions were written by Tony J. Cunha (1917-1992) Published by the Salt Institute 700 North Fairfax Street, Suite 600 Alexandria, Virginia 22314-2040.

Berthod, A., Carda-Broch, S., Garcia-Alvarez-Coque, M.C. (1999). Hydrophobicity of ionizable compounds. A theoretical study and measurements of diuretic octanol-water partition coefficients by countercurrent chromatography. *Analytical Chemistry*, 71(4), 879-888.

Bertrand, M., Doré, T. (2008). Comment intégrer la maîtrise de la flore adventice dans le cadre général d'un système de production intégrée. *Innovations Agronomiques*, 3, 1-13.

Bianco, V.V., Santamaría, P. (1998). Nutritional value and nitrate content in edible wild species used in southern Italy. Proc. 3rd IS on diversification of vegetable crops. Eds. VE, Rubatzky, Chen Hang, J.Y. Peron, *Acta Horticulturae*, 476, 71-87.

Bigot, L., Bodot, P. (1973). Contribution à l'étude biocénotique de la garrigue a *Quercus coccifera*-II. Composition biotique du peuplement des invertébrées. *Life and Environnent*, 23(2), 229-249.

Blois, M.S. (1958). Antioxidant determination by the use of stable free radical, *Nature*. 181.

Blumenthal, M. (2000). Herbal Medicine Expanded Commission E. Monographs. 1st Ed. American Botanical Council. Austin, 215-218.

Bnouham, M. (2012). Recent advances in pharmacologic study of anticancer natural products from medicinal plants in Morocco. *Tang [Humanitas Medicine]*, 2(3), 22.1-22.9. https://doi.org/10.5667/tang.2012.0024

Bonnefont-Rousselot, D., Raji, B., Walrand, S., Gardes-Albert, M., Jore, D., Legrand, A., Vasson, M. P. (2003). An intracellular modulation of free radical production could contribute to the beneficial effect of metformin towards oxidative stress. *Metabolism*, 52(5), 586-589.

Bonnier, G., De., L.G. (1948). Flore complète portative de la France, de la Suisse et de La Belgique (Paris : Librairie Générale de l'enseignement).

Bordoloi, M., Bordoloi, P. K., Dutta, P. P., Singh, V., Nath, S., Narzary, B., Bhuyan, P. D., Rao, P. G., Barua, I. C. (2016). Studies on some edible herbs: Antioxidant activity, phenolic content, mineral content, and antifungal properties. *Journal of Functional Foods*, 23, 220-229. https://doi.org/10.1016/j.jff.2016.02.028

Bouabdallah, H. (2008). Etude des différents milieux de la région de Tlemcen. Document pédagogique, Univ. Tlemcen.

Bouanani, A. (2004). Hydrologie, transport solide et modélisation. Etude de quelques sous bassins de la Tafna (NW Algérie). Thes. Doc., Univ Abou Bekr Belkaid, Tlemcen, Algérie, 250pp.

Boudy, P. (1955). Economie forestière nord-africaine : Description forestière de l'Algérie et de la Tunisie. Ed. Larose, Paris.

Bouhache, M., Boulet, C., Chougrani, A. (1994). Aspects floristico-agronomiques des mauvaises herbes de la région du Loukkos (Maroc). *Weed Research*, 34(2), 119-126. https://doi.org/10.1111/j.1365-3180.1994.tb01979.x

Bourhia, M., Laasri, F.E., Moussa, S.I., Ullah, R., Bari, A., Saeed Ali, S., Kaoutar, A., Haj Said, A.A., El Mzibri, M., Said, G., Khlil, N., Benbacer, L. (2019). Phytochemistry, antioxidant activity, antiproliferative effect, and acute toxicity testing of two Moroccan *Aristolochia* species. *Evidance-Based Complementary Alternative Medecine*, 2019, 8, Article ID 9710876. https://doi.org/10.1155/2019/9710876

Bouzidi, L. El, Larhsini, M., Markouk, M., Abbad, A., Bekkouche, K. (2011). Antioxidant and Antimicrobial Activities of *Withania frutescens*. *Natural Product Communications*, 6(10), 1447-1950. https://doi.org/10.1177/1934578X1100601009

Braun-Blanquet, J. (1951). Pflanzensoziologie. Grundzuge der vegetations (Autriche : Vienne Ed.).

Bribi, N., Algieri, F., Rodriguez-Nogales, A., Garrido-Mesa, J., Vezza, T., Maiza, F., Utrilla, M.P., Rodriguez-Cabezas, M.E., Galvez, J. (2015). Antinociceptive and anti-inflammatory effects of total alkaloid extract from *Fumaria capreolata* L. *Evidance-Based Complementary Alternative Medecine*, 2015, 7, Article ID 736895. https://doi.org/10.1155/2015/736895

Brouillard, R. (1986). The flavonoids Advances. *Research science*, 525-538.

Brunel, S., Tison, J. (2005). Study on invasive plants in the Mediterranean Basin. *Rencontre Environnement*, 59, 49 - 50.

Bruneton, J. (1993). Pharmacognosie : Phytochimie et plantes médicinales. 2ème édition, Lavoisier TEC et Doc, Paris, 268-277.

Bruneton, J. (1999). Pharmacognosie, phytochimie, plantes médicinales. 3ème Ed : Lavoisier, Paris, 1120pp.

Bruneton, J. (2009). Pharmacognosie, phytochimie, plantes médicinales. Lavoisier Technique et Documentation, 4ème Edition. Paris.

Cabiddu, A., Addis, M., Pinna, G., Spada, S., Fiori, M., Sitzia, M., Pirisi, A., Piredda, G., Molle, G. (2006). The inclusion of a daisy plant (*Chrysanthemum coronarium*) in dairy sheep diet. 1: Effect on milk and cheese fatty acid composition with particular reference to C18:2 cis-9, trans-11. *Livestock Science*, 101(1-3), 57-67. https://doi.org/10.1016/j.livprodsci.2005.09.014

Carpino, S., Licitra, G., Van Soest, P.J. (2003). Selection of forage species by dairy cattle on complex Sicilian pasture. *Animal Feed Science and Technology*, 105(1-4), 205-214. https://doi.org/10.1016/S0377-8401(03)00054-3

Castro, P., González, A. Prada, D. (1990). Determinación simultánea de nitrógeno y fósforo en muestras de pradera. Actas de la XXX Reunión Científica de la SEEP. 200-207pp.

Ćetković, G.S., Djilas, S.M., Čanadanović-Brunet, J.M., Tumbas, V.T. (2004). Antioxidant properties of marigold extracts. *Food Research International*, 37(7), 643-650. https://doi.org/10.1016/j.foodres.2004.01.010

Chafic, Z., Berrichi, A., Bouali, A., Taleb, A. (2013a). Systematic, phytoecological and agronomic survey of citrus fruits weed flora in the North-eastern Morocco. *Moroccan Journal of Biology*. 10, 20-29.

Chafik Z., Taleb A., Bouhache M., Berrichi A. (2013b). Flore adventice des agrosystèmes du Maroc Oriental : cas du périmètre de la Moulouya. *Revue Marocaine de Protection des Plantes*, 4, 27-44.

Chafik, Z., Bouchache, M., Taleb, A., Berrichi, A. (2014). Actualisation de la flore adventice des cépages du périmètre irrigué de la Moulouya. *Revue Marocaine de Protection des Plantes*, 5, 47-65.

Chahmi, N., Anissi, J., Jennan, S., Farah, A., Sendide, K., El Hassouni, M. (2015). Antioxidant activities and total phenol content of *Inula viscosa* extracts selected from three regions of Morocco. *Asian Pacific Journal of Tropical Biomedicine*, 5(3), 228-233. https://doi.org/10.1016/S2221-1691(15)30010-1

Chandel, B.S., Singh, A. (2017). Biopotency of *Acorus calamus*, *Withania somnifera* and *Azadirachta indica* in suppressing oviposition and adult emergence, caused by of *Callasobruchus* *chinensis* (L.) infesting stored chickpea seeds in U.P. *International Journal of Zoological Studies*, 2(6), 161-166. http://www.zoologyjournals.com/download/202/2-6-60-237.pdf

Chauvel, B., Tschudy, C., Munier-Jolain, N. (2011). Gestion intégrée de la flore adventice dans les systèmes de culture sans labour. *Cahiers Agricultures*, 20(3), 194-203.

Chebbani, R. (1996). Etude à différentes échelles des risques d'érosion dans le bassin versant d'Isser (Tlemcen). Thes. Mag., I.N.A. El-Harrach, Alger, 135pp.

Chemouri, S., Ainad Tabet, M., Kechairi, R., Tefiani, C., Megharbi, A., Roca-Fernandez, A. I., Larid, M. (2019). Impact of mechanical and chemical weeding on the floristic diversity of citrus orchards in Tlemcen (Northwestern Algeria). *International Journal of Environmental Studies*, 77(4), 602-612. https://doi.org/10.1080/00207233.2019.1690279

Chiaravalli, M., Rapetti, L., Graziosi, A. R., Galassi, G., Crovetto, G. M., Colombini, S. (2019). Comparison of Faecal versus Rumen Inocula for the Estimation of NDF Digestibility. *Animals*, 9(928), 1-10.

Chopra, Ananda, S. (2003). Medicine across cultures: history and practice of medicine in non-western cultures. Norwell, *Kluwer Academic Publishers*, 75-83. https://doi.org/10.1007/0-306-48094-8

Chung, S.K., Osawa, T., Kawakishi, S. (1997). Hydroxyl radical scavenging effects of species and scavengers from brown mustrad (*Brassica nigra*). *Bioscience, Biotechnology, and Biochemistry*, 61, 118-123.

Church D.C. (1988). The ruminant digestive physiology and nutrition. A division of Simon and Schuster, Englewood Cliffs, Prentice Hall, New Jersey, USA, 275pp.

Civelek, C., Balkaya, A. (2013). The Nutrient Content of Some Wild Plant Species Used as Vegetables in Bafra Plain Located in the Black Sea Region of Turkey. *The European Journal of Plant Science and Biotechnology*, 7(1), 62-65.

Classen, B., Amelunxen, F., Blaschek, W. (2001). Ultrastructural observations on the rust fungus *Puccinia malvacearum* in *Malva sylvestris* ssp. mauritiana. *Plant Biology*. 3, 437-442. https://doi.org/10.1055/s-2001-16456

Clifford, MN. Astringency. In F.A. Tomas-Barberan and R.J. (1997). Phytochemistry of Fruits and Vegetables. Robins (eds), Clarendon Press, Oxford, 87-107pp.

Cohen, R.D.H. (1976). Phosphorus nutrition of ruminants. Department of Agriculture, Agricultural Research Station, Grafton, N.S.W., 120-127.

Colbach, N., Biju-Duval, L., Gardarin, A., Granger, S., Guyot, S.H.M., Meziere, D., Munier-Jolain, N.M., Petit, S. (2014). The role of models for multicriteria evaluation and multi-objective design of cropping systems for managing weeds. *Weed Research*, 54(6), 541-555. https://doi.org/10.1111/wre.12112

Combs, D. (2014). Using In Vitro Total-Tract NDF Digestibility in Forage Evaluation. *Focus on forage*. 15(2), 1-3.

Combs, D. K. (2016). Relationship Between NDF Digestibility and Animal Performance. *WCDS Advances in Dairy Technology*, 28, 83-96.

COMMISSION REGULATION (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed. Official Journal of the European Union.

Compean, K.L., Ynalvez, R.A. (2014). Antimicrobial Activity of Plant Secondary Metabolites: A Review. *Research Journal of Medicinal Plant*, 8(5), 204-213. https://doi.org/10.3923/rjmp.2014.

Conforti, F., Ioele, G., Statti, G.A., Marrelli, M., Ragno, G., Menichini, F. (2008). Antiproliferative activity against human tumor cell lines and toxicity test on Mediterranean dietary plants. *Food and Chemical Toxicology*, 46(10), 3325-3332. https://doi.org/10.1016/j.fct.2008.08.004

Conforti, F.S., Marrelli, S., Menichini, M., Statti, F., Uzunov, G.A., Tubaro, D., Menichini, A. (2009). The protective ability of Mediterranean dietary plants against the oxidative damage: the role of radical oxygen species in inflammation and the polyphenol, flavonoid and sterol contents. *Food Chem*istry, 112(3), 587-594. https://doi.org/10.1016/j.foodchem.2008.06.013

Cordell, G.A. (1981). Introduction to Alkaloids-A Biogenetic approach and references therein, John Wiley, New York, 426-427pp.

Cotelle, A., Bernier, J.L., Catteau, J.P., Pommery, J., Wallet, J.C., Gaydou, E.M. (1996). Antioxidant properties of hydroxyl- flavones. *Free Radical Biology and Medicine*, 20, 35-43.

Couplan, F. (2003). Guide des plantes sauvages comestibles et toxiques. Ed. Delachaux et Niestle. Collection. Règne végétal.

Cowan, M. (1999). Plant products as antimicrobial agents, *Clinical Microbiology Reviews*,12(4), 564-582.

Cristaudo, A., Restuccia, A., Onofri, A., Lo Giudice, V., Gresta, F. (2015). Species–area relationships and minimum area in citrus grove weed communities. *Plant Biosystems*, 149(2), 337-345. https://doi.org/10.1080/11263504.2013.825344

Cruz-Garcia, Gisella S., Lisa L. Price. (2012). Weeds as important vegetables for farmers. Acta Societatis Botanicorum Poloniae, 81(4), 397-403.

Cseke, LJ., Kirakosyan, A., Kaufman, PB., Warber, SL., Duke, JA., Brielmann, HL. (2006). Natural products from plants. 2ème Edition. Taylor et Francis Group, LLC.

Cummings, J.H., Stephen, A.M. (2007). Carbohydrate terminology and classification: Review. *European Journal of Clinical Nutrition*, 61(1), 5–18. https://doi.org/10.1038/sj.ejcn.1602936

Cushnie, T.P.T., Hamilton, V.E.S., Chapman, D.G., Taylor, P.W., Agneau, A.J. (2007). Agregation of *Staphyloc* Addis *occus aureus* following treatment with the antibacterial flavonol galangin. *Journal of applied microbiology*, 103(5), 1562-1567.

Dai, J., Mumper, R. (2010). Plant phenolics: extraction, analysis and their antioxidant and anticancer properties. *Molecules*, 15, 7313-7352.

Daniewski, W.M., Skibicki, P., Gumuka, M., Drozdz, B., Grabarczyk, H., Boszyk, E. (1988). Sesquiterpene lactones. XXXV. Constituents of *Reichardia tingitana* L. Roth. and their anti-feedant activity. *Acta Societatis Botanicorum Poloniae*, 57(4), 539-545. https://doi.org/10.5586/asbp.1988.051

De Bello, F. (2007). Grazing effect on the species-arearelationship. Variation along aclimatic gradient in NE Spain. *Journal of Vegetation Science*. 25-34.

De Clavijo, R. (2005). The reproductive strategies of the heterocarpic annual *Calendula arvensis* (Asteraceae). *Acta Oecologica*, 28(2), 119-126. https://doi.org/10.1016/j.actao.2005.03.004

De Oliveira, H.A.B., Anunciação, P.C., da Silva, B.P., de Souza, Â.M.N., Pinheiro, S.S., Lucia, C.M. Della, Cardoso, L. de M., Castro, L.C.V., Pinheiro-Sant'ana, H.M. (2019). Nutritional value of non-conventional vegetables prepared by family farmers in rural communities. *Ciencia Rural*, 49(8), 1-10. https://doi.org/10.1590/0103-8478cr20180918

Defraigne, J.O., Pincemail, J. (2009). Stress oxydant et antioxydants : mythes et réalités. *Revue Medical de Liège*, 63, 10-19.

Deliorman Orhan, D., Özçelik, B., Hoşbaş, S., Vural, M. (2012). Assessment of antioxidant, antibacterial, antimycobacterial, and antifungal activities of some plants used as folk remedies in

Turkey against dermatophytes and yeast-like fungi. *Turkich Journal of Biology*, 36(6), 672-686. https://doi.org/10.3906/biy-1203-33

Delpech, R. (1980). Informations apportées par les mauvaises herbes pour l'élaboration d'un diagnostic phytoécologique stationnel. Presented at the 6ème Colloque International Ecologie et Biologie des mauvaises herbes, Montpellier.

Demain, AL. (1999). Pharmaceutically active secondary metabolites of microorganisms. *Applied Microbiology and Biotechnology*, 52(4), 455-463.

Derouiche, K., Zellagui, A., Gherraf, N., Belbout, A. (2014). Evaluation of Antioxidant and Antimicrobial Activities of Flowers Extracts of *Chrysanthemum Segetum* L. *World Journal of Environmental Biosciences*, 7(4), 106-111.

Diab, N., Deghiche, L. (2013). Floristic diversity of weed in the oasis of Ziban (Biskra, Algeria), abundance and importance. Conference poster in 22e Conférence du COLUMA. Journées Internationales sur la Lutte contre les Mauvaises Herbes, Dijon, France, 10-12 décembre 2013.

Disciglio, G., Tarantino, A., Frabboni, L., Gagliardi, A., Giuliani, M.M., Tarantino, E., Gatta, G. (2017). Qualitative characterization of cultivated and wild edible plants: Mineral elements, phenols content and antioxidant capacity. *Italian Journal of Agronomy*, 12(4), 383-394. https://doi.org/10.4081/ija.2017.1036

Disciglio, G., Tarantino, A., Frabboni, L., Gagliardi, A., Giuliani, M.M., Tarantino, E., Gatta, G. (2017). Qualitative characterization of cultivated and wild edible plants: Mineral elements, phenols content and antioxidant capacity. *Italian Journal of Agronomy*, 12(4), 383-394. https://doi.org/10.4081/ija.2017.1036

Doreau, M., Chilliard, Y. (1997). Digestion and metabolism of dietary fat in farm animals. *British Journal of Nutrition*, 78(1), 15-35. https://doi.org/10.1079/bjn19970132

Dorman, H.J.D., Deans, S.G., Noble, R.C., Surai, P. (1995). Evaluation in vitro of plant essential oils as natural antioxidants. *Journal of Essential Oil Research*, 7, 645-651.

Dormann, C., King, R. (2004). Comparing the palatability of native and non-native Mediterranean plants. *Ecología Mediterránea*, 2, 39-46. https://www.biom.uni-freiburg.de/mitarbeiter/dormann/publications-dormann/Dormann2004EcolMedit

Dougall, H.W., Bogdan, A.V. (1966). The Chemical Composition of Some Leguminous Plants Grown in the Herbage Nursery at Kitale, Kenya. *East African Agricultural and Forestry Journal*, 32(1), 45-49. https://doi.org/10.1080/00128325.1966.11662090

DSA (2016). Direction of Agricultural Services, Tlemcen, Algeria.

Duke, J.A. (1992). Handbook of edible weeds. CRC, Boca Raton, Florida, 246pp.

Eddoud, A., Buisson, E., Achour, L., Guediri, K., Bissati, S., Abdelkrim, H. (2018). Changes in weed species composition in irrigated agriculture in Saharan Algeria. *Weed Research*, 58(6), 424-436. https://doi.org/10.1111/wre.12328

Edenharder, R., Grünhage, D. (2003). Free radical scavenging abilities of flavonoids as mechanism of protection against mutagenicity induced by tert-butyl hydroperoxyde of cumene hydroperoxyde in *Salmonella typhimurium* TA102. *Mutation Research/Genetic Toxicology and Environmental Mutagenesis*, 540(1), 1-18.

Edrah, S. M., Omeman, A., Belhage, S., Hassan, S. A. (2018). Studies on physiochemical, qualitative, and quantitative phytochemical, analysis, and antibacterial activity of Oxalis pes-caprae and Erodium glaucophyllum utilized as a traditional folk treatment from Al-Khums Libya. *International Journal of Chemistry Studies*, 2(4), 01-09.

Edrah, S. M., Omeman, A., Belhage, S., Hassan, S. A. (2018). Studies on physiochemical, qualitative, and quantitative phytochemical, analysis, and antibacterial activity of *Oxalis pes-*

caprae and *Erodium glaucophyllum* utilized as a traditional folk treatment from Al-Khums Libya. *International Journal of Chemistry Studies*, 2(4), 1-9. http://www.chemistryjournal.in/download/56/2-5-13-292.pdf

El Bous, M., Abd el hamid, H. (2018). Species distribution patterns of the weed flora in mango orchards of Ismailia Governorate, Egypt: implications for conservation. *Taeckholmia*, 38(1), 184-200. https://doi.org/10.21608/taec.2019.5781.1001

El Bouzidi, L., Mahiou-Leddet, V., Bun, S.S., Larhsini, M., Abbad, A., Markouk, M., Fathi, M., Boudon, M., Ollivier, E., Bekkouche, K. (2013). Cytotoxic withanolides from the leaves of Moroccan *Withania frutescens*. *Pharmaceutical Biology*, 51(8), 1040-1046. https://doi.org/10.3109/13880209.2013.775162

El Moussaouia, A., Jawharia, F.Z., Almehdi, A.M., Elmsellem, H., Fikri Benbrahim, K., Bousta, D., Bari, A. (2019). Antibacterial, antifungal and antioxidant activity of total polyphenols of *Withania frutescens* L. *Bioorganic Chemistry*, *93*, 103337. https://doi.org/10.1016/j.bioorg.2019.103337

El-Gawad, M.A., Mashaly, I.A., Al-Nafie, R.I. (2015). Antioxidant activity and allelopathic potential of five wild antioxidant activity and allelopathic potential of five wild plants on germination and growth of *Biden spilosa* L. *International Journal of Current Research*, 7(10), 21019-21024.

El-Haci, I.A., Bekkara, F.A., Mazari, W., Gherib, M. (2013). Phenolics content and antioxidant activity of some organic extracts of endemic medicinal plant *Anabasis aretioides* Coss. and Moq. from Algerian Sahara. *Pharmacognosy Journal*, 5, 108-112.

Elong, M.I., Larid, M., Taibi, K. (2018). Functional analysis of competition for resources between durum Wheat (*Triticum turgidum* L. var. durum) and two species of weeds *Galiuma parine*, *Fumaria officinalis* in Tiaret, Algeria. *International Journal of Biosciences (IJB)*, 12(2), 169-176. https://doi.org/10.12692/ijb/12.2.169-176

El-Shazly, A., Sarg, T., Ateya, A., Abdel Aziz, A., El-Dahmy, S., Witte, L., Wink, M. (1996). Pyrrolizidine alkaloids from *Echium setosum* and *Echium vulgare*. *Journal of Natural Products*, 59(3), 310-313. https://doi.org/10.1021/np9600661

El-Shesheny, M.A., Hendawy, S.H., Ahmed, K.M. (2014). Assessment of productivity, botanical composition, and nutritive value of some plant communities at Sidi-Barrani in North Western Coast of Egypt. *Annals of Agricultural Sciences*, 59(2), 155-163. https://doi.org/10.1016/j.aoas.2014.11.001

Eltayep Ahmed, E.E.E. (2016). Weed survey on potato crop in Khartoum state, Sudan. Graduation Research Project. Sudan Univ. of Sciences and Technology, College of Agricultural Studies, Department of Plant Protection, 31pp.

El-Waer N.E.H. (2018). TUNWeeds: A Smart Identification Tool for Brassicaceae Weeds in Rapeseed (*Brassica napus*) Crop in Tunisia. *Tunisian Journal of plant Protection*, 13(SI), 191-198.

Elzaawely, A.A., Xuan, T.D., Tawata, S. (2007). Essential oils, kava pyrones and phenolic compounds from leaves and rhizomess of *Alpinia zerumbet* and their antioxidant activity. *Food Chemistry*, 103, 486-494.

Emberger, L. (1930). La végétation de la région méditerranéenne. Essai d'une classification dans groupement végétaux. *Revue Générale de Botanique*, 43, 641-662 and 705-729.

Emberger, L. (1952). Sur le Quotient Pluviothermique. C.R.A cade. Sci., 234, 2508-2510.

Emre Kitiş, Y., Kolören, O., Uygur F.N. (2017). The Effects of Mulch Textile Application on Weed Control and Mandarin Growing in Newly Established Mandarin Orchard. *Turkish Journal of Agriculture - Food Science and Technology*, 5(6), *568-580.*

Emre, I., Kurşat, M., Yılmaz, Ö., Erecevit, P. (2011). Some biological compounds, radical scavenging capacities and antimicrobial activities in the seeds of *Nepeta italica* L. and *Sideritis montana* L. subsp. *montana* from Turkey. *Grasas y aceites*, 62(1), 68-75. https://doi.org/10.3989/gya.033210

Ercetin, T., Senol, F.S., Erdogan Orhan, I., Toker, G. (2012). Comparative assessment of antioxidant and cholinesterase inhibitory properties of the marigold extracts from Calendula arvensis L. and Calendula officinalis L. *Industrial Crops and Products*, 36(1), 203-208. https://doi.org/10.1016/j.indcrop.2011.09.007

Erdogan, I., Şene, B., Musharraf, S.G. (2012). Experimental and toxicologic pathology antioxidant and hepatoprotective activity appraisal of four selected Fumaria species and their total phenol and flavonoid quantities. *Experimental and Toxicologic Pathology* 64(3), 205-209. https://doi.org/10.1016/j.etp.2010.08.007

Eruygur, N., Yilmaz, G., Üstün, O. (2012). Analgesic and antioxidant activity of some Echium species wild growing in Turkey. *Fabad Jornal of Pharmaceutical Sciences*, 37(3), 151-159. http://dergi.fabad.org.tr/pdf/volum37/issue3/151-159.pdf

Fahmy, A.G.E.D. (1997). Evaluation of the weed flora of Egypt from Predynastic to Graeco-Roman times. *Vegetation History and Archaeobotanical*, 6(4), 241-247. https://doi.org/10.1007/bf01370445

Farooq, M., Jabran, K., Cheema, Z. A., Hm, K. (2011). The role of allelopathy in agricultural pest management. *Pest Management Science*, 67, 493-506. https://doi.org/10.1002/ps.2091

Favier, A. (2003). Le stress oxydant. L'actualité chimique, 108p.

Fenni, M. (1993). Survey of the flora of cornfields in the high cereal plains of Setif (North-East Algeria). *Mededelingen van de Faculteit Landbouwwetenschappen*, 58, 1003-1011.

Ferreira, A., Proença, C., Serralheiro, M.L.M., Araújo, M.E.M. (2006). The *in vitro* screening for acetylcholinesterase inhibition and antioxidant activity of medicinal plants from Portugal. *Journal of Ethnopharmacology*, 108(1), 31-37. https://doi.org/10.1016/j.jep.2006.04.010

Fertout-Mouri, N. (2018). Étude phytoécologique de la flore adventice des agrosystèmes céréaliers de la région de Tessala (Algérie nord-occidentale). *Bulletin de la Société Royale des Sciences de Liège*, 87, 70-99.

Firuzi, O., Javidnia, K., Gholami, M., Soltani, M., Miri, R. (2010). Antioxidant activity and total phenolic content of 24 Lamiaceae species growing in Iran. *Natural Production Communicatioin*, 5(2), 261-264. https://doi.org/10.1177/1934578x1000500219

Flores, N., Jiménez, I.A., Giménez, A., Ruiz, G., Gutiérrez, D., Bourdy, G., Bazzocchi, I.L. (2009). Antiparasitic activity of prenylated benzoic acid derivatives from piper species. *Phytochemistry*, 70(5), 621-627.

Franke, A.C., Lotz, L.A.P., Van der Burg, W.J., Van Overbeek, L. (2009). The role of arable weed seeds for agroecosystem functioning. *Weed Research*, 49, 131-141. https://doi.org/10.1111/j.1365-3180.2009. 00692.x

Fried, G. (2010). Variations spatiales et temporelles des communautés adventices des cultures annuelles en France. *Acta Botanica Gallica*, 157(1), 183-192. https://doi.org/10.1080/ 12538078.2010.10516198

Fried, G., Chauvel, B., and Reboud, X. (2008). Évolution de la flore adventice des champs cultivés au cours des dernières décennies : Vers la sélection de groups d'espèces répondant aux systèmes de culture. *Innovations Agronomiques*, 3, 150-26.
Fried, G., Reboud, X. (2007). Évolution de la composition des communautés adventices des cultures de colza sous l'influence des systèmes de culture. *Oléagineux, Corps Gras, Lipides*, 14(2), 130-138. https://doi.org/10.1051/ocl.2007.0098
Friedman, M., Henika, P.R., Levin, C.E., Mandrell, R.E., Kozukue, N. (2006). Antimicrobial activities of tea catechins and the aflavins and tea extracs against *Bacillus cereus*. *Journal of Food Protection*, 69(2), 354-361.
Fujii, Y. (2001). Screening and Future Exploitation of Allelopathic Plants as Alternative Herbicides with Special Reference to Hairy Vetch, *Journal of Crop Production*, 4(2), 257-275, https://doi.org/10.1300/J144v04n02_09
Galal, T., Shehata, H. (2015). Impact of nutrients and heavy metals capture by weeds on the growth and production of rice (*Oryza sativa* L.) irrigated with different water sources. *Ecological Indicators*, 54(7), 108-115. https://doi.org/10.1016/j.ecolind.2015.02.024
Gamal, Z. (2017). Phytochemical Study and Tissue Culture of *Reichardia tingitana*. Th. Mast. Faculty of Pharmacy. Cairo Univ., 1-19pp.
Gaspar, M.C., Fonseca, D.A., Antunes, M.J., Frigerio, C., Gomes, N.G. M., Vieira, M., Santos, A.E., Cruz, M.T., Maria, D., Campos, M.G., Gaspar, M.C., Fonseca, D.A., Antunes, M.J., Frigerio, C., Gomes, N.G.M., Vieira, M., Santos, A.E., Cruz, M.T., Cotrim, M.D. (2018). Polyphenolic characterisation and bioactivity of an *Oxalis pes-caprae* L. leaf extract. *Natural Product Research*, 32(6), 732-738. https://doi.org/10.1080/14786419.2017.1335728
Gasparetto, J.C., Martins, C.A.F., Hayashi, S.S., Otuky, M.F., Pontarolo, R. (2012). Ethnobotanical and scientific aspects of Malva sylvestris L.: A millennial herbal medicine. *Journal of Pharmacy and Pharmacology*, 64(2), 172-189. https://doi.org/10.1111/j.2042-7158.2011.01383.x
Ghabru, A., Rana, N., Pradesh, H. (2019). Biological significance of secondary metabolites: A review. *International Journal of Research in Pharmacy and Pharmaceutical Sciences*, 4(2), 84-9.
Ghasemzadeh, A., Ghasemzadeh, N. (2011). Flavonoids and phenolic acids: Role and biochemical activity in plants and human. *Journal of Plant medicinal Research*, 5(31), 6697-6703. https://doi.org/10.5897/JMPR11.1404
Ghazanfar, S., Latif, A., Mirza, I.H., Nadeem, M.A. (2011). Macro-minerals concentration of major fodder tree leaves and shrubs of district Chakwal, Pakistan. *Pakistan Journal of Nutrition*, 10(5), 480-484.
Gilani, A.H., Bashir, S., Janbaz, K.H., Khan, A. (2005). Pharmacological basis for the use of *Fumaria indica* in constipation and diarrhea. *Journal of Ethnopharmacology*, 96(3), 585-589. https://doi.org/10.1016/j.jep.2004.10.010
Goering, H.K., Van Soest, P.J. (1970). Forage fiber analyses (apparatus, reagents, procedures, and some applications). Agriculture Handbook No. 379, Agric. Res. Serv., USDA, Washington, DC, USA, 20 pp.
Goeser, J.P., Combs, D.K. (2009). An alternative method to assess 24h ruminal in vitro neutral detergent fiber digestibility. *Journal of Dairy Science*, 92, 3833-3841.
Gökbulut, A., Özhan, O., Satilmis, B., Batçioglu, K., Günal, S., Şarer, E. (2013). Antioxidant and antimicrobial activities, and phenolic compounds of selected *Inula* species from Turkey. *Natural Product Communications,* 8(4), 475-478. https://doi.org/10.1177/1934578x1300800417

Gokulan, K., Khare, S., Cerniglia, C. (2014). Metabolic Pathways: Production of Secondary Metabolites of Bacteria. In: Batt, C.A., Tortorello, M.L. (Eds.), Encyclopedia of Food Microbiology, vol 2. Elsevier Ltd, Academic Press, 561-569pp. https://doi.org/10.1016/B978-0-12-384730-0.00203-2

Gomaa, N.H. (2017). Floristic Composition of Weed Vegetation in Citrus Orchards in Aljouf Region, Kingdom of Saudi Arabia. *Journal of Bio-Molecular Sciences (JBMS)*. 5(1), 15-23. https://www.directsciences.com/files/Gomaa.pdf

Gould, J.M. (1983). Probing the structure and dynamics of lignin in situ. *What's New in Plant Physiology*, 14, 25-91.

Grime, J.P. (1977). Evidence for the existence of three primary strategies in plants and its relevance to ecological and evolutionary theory. *The American Naturalist*, 111, 1169-1194. https://doi.org/10.1086/283244

Gross, G.G. (1992). Enzymes in the biosynthesis of hydrolysable tannins. In R.W. Heminway, P.E. Laks and S.J. Branham (eds), Plant Polyphenols. Plenum Press, New York, 43-60.

Gruenwald, J., Brendler, T., Jaenicke, C., Nontvale N.J. (2000). PDR for herbal medicines. *Journal of Medical Economics,* Montvale, NJ. 77, 284-285.

Guardia, P. (1975). Géodynamique de la marge alpine du continent africain d'après l'étude de l'Oranie Nord-occidentale. Relations structurales et paléogéographiques entre le Rif externe, le Tell et l'avant pays Atlassique. Thes. Doc., Univ. Nice.

Güçlütürk, I., Detsi, A., Weiss, K. (2012). Evaluation of antioxidant activity and identification of major polyphenolics of the invasive Weed *Oxalis pes-caprae*. *Phytochemical Analysis*, 23(6), 642-646. https://doi.org/10.1002/pca.2367

Guignard, J.L. (1996). Abrégé de biochimie végétale, Ed. Masson, Paris, 160pp.

Guil, J. L., Rodríguez-Garcí, I., Torija, E. (1997). Nutritional and toxic factors in selected wild edible plants. *Plant Foods for Human Nutrition*, 51(2), 99-107. https://doi.org/10.1023/A:1007988815888

Guinochet, M. (1973). Phytosociologie, Ed. Masson, Paris.

Gülcëin, Ç, Mshvildadze, V. Gepdiremen, A, Elias, R. (2004). Antioxidant Activity of Saponins Isolated from Ivy: alpha-hederin, Hederasaponin-C, Hederacolchiside-E and Hederacolchiside-F. Letter, *Planta Medica*, 70, 561-563 https://doi.org/10.1055/s-2004-827158

Gulcin, I. (2012). Antioxidant activity of food constituents: an overview. *Archives of Toxicology*, 86, 345-391.

Gulshan, A.B., Dasti, A.A., Hussain, S., Atta, M.I., Amin-ud-Din, A. (2012). Indigenous uses of medicinal plants in rural areas of Dera Ghazi Khan, Punjab, Pakistan. *Journal of Agricultural and Biological Science*, 7(9), 750-762.

Gutiérrez, D., Mendoza, S., Serrano, V., Bah, M., Pelz, R., Balderas, P., León, F. (2008). Proximate composition, mineral content, and antioxidant properties of 14 Mexican weeds used as fodder. *Weed Biology and Management*, 8, 291-296. https://doi.org/10.1111/j.1445-6664.2008.00307.x

Haag, J. R., (1951). Minerals for Livestock. Agricultural Experiment Station Bulletin 503. Oregon State College Corvallis, 12pp.

Hagerman, A. E., Riedl, K.M., Jones, G.A., Sovik, K.N., Ritchard, N.T., Hartzfeld, P.W., Richel, T.L. (1998). High molecular weight plant polyphenolics (tannins) as biological antioxidants. *Journal of Agricultural and Food Chemistry*, 46, 1887-92.

Hall, M.B., Mertens, D.R. (2008). *In vitro* fermentation vessel type and method alter fiber digestibility estimates. *Journal of Dairy Sci*ence, 91, 301-307.

Halliwell, B., Gutteribge, J. (1997). Lipid peroxidation in brain homogenates: the role of iron and hydroxyl radicals. *Journal of Neurochemistry*, 69 (3), 1330-1330.

Hammer *et al*., 2001

Hammer, Ø. (1999-2020). Reference manual. PAleontological STatistics (PAST), Version 4.03, 283pp.

Hanitet, K. (2012). Les groupements des adventices des cultures dans la région d'Oran. Thes. Mag., Department of Biology, Faculty of Sciences, Univ. of Oran, 71pp.

Hannachi, A. (2019). Ecological study for groups of citrus weeds in the Skikda region, Algeria. *Journal of Research in Weed Science*, 2, 216-223. https://doi.org/10.26655/jrweedsci

Hannachi, A. and Fenni, M. (2013). Étude floristique et écologique des mauvaises herbes des cultures de la région de Batna (Algérie). *Revue Agriculture*, 5, 24-36.

Haouas, D., Flamini, G., Halima-kamel, M. Ben, Habib, M., Hamouda, B. (2010). Feeding perturbation and toxic activity of five *Chrysanthemum* species crude extracts against *Spodoptera littoralis* (Boisduval) (Lepidoptera; Noctuidae). *Crop Protection*, 29(9), 992-997. https://doi.org/10.1016/j.cropro.2010.05.002

Harbone, J.B. (1967). Comparative biochimitry of the flavonoides. Academic press. New York, 1-130 pp.

Harborne, J.B. (1998). Phytochemical methods. A guide to modern techniques of plants analysis. 3rd Edition.

Harborne, J.B. (1999). Classes and functions of secondary products in N.J. Walton, D.E. Brown (Eds.), chemicals from plants, perspectives on secondary plant products, Imperial college Press, 1-25pp.

Harrold, R. L., Nalewaja, J. D. (1977). Proximate, mineral, and amino acid composition of 15 weed seeds. *Journal of Animal Science*, 44(3), 389-394.

Hartmann, T. (2007). From waste products to ecochemicals: Fifty years research of plant secondary metabolism. Review. *Phytochemistry*, 68, 2831-2846.

Hassine, M., Znati, M., Flamini, G., Ben, H. (2014). Chemical composition, antibacterial and cytotoxic activities of the essential oil from the flowers of Tunisian *Convolvulus althaeoides L. Natural Product Research: Formerly Natural Product Letters*, 28(11), 769-775. https://doi.org/10.1080/14786419.2013.879476

Hathaway, R., Pirelli, G. (2004). Forage Value. Chapter in Beef Cattle Nutrition Workbook, 19-26, 69pp.

Havsteen, B.H. (2002). The biochemistry and medical significance of the flavonoids. *Pharmacology and Therapeutics*, 96(2), 67-202.

Haziyah Ishak, A., Husna Shafie, N., Mohd Esa, N., Bahari, H., Ismail, A. (2018). From Weed to Medicinal Plant: Antioxidant Capacities and Phytochemicals of Various Extracts of Mikania micrantha. *International Journal of Agriculture and Biology*, 20, 561-568. https://doi.org/10.17957/IJAB/15.0522

Hendawy, S.F., Abouziena, H.F., Abd El-Razika, T.M., Amera, H.M., Husseina, M.S. (2020). Winter weeds and its control in the medicinal plants in Egypt: a survey study. *Egyptian Pharmaceutical Journal*, 18, 16-26. https://doi.org/10.4103/epj.epj_13_18

Hennebelle, T., Sahpaz, S., Bailleul, F. (2004). Polyphénols végétaux, sources, utilisations et potentiel dans la lutte contre le stress oxydatif. *Phytothérapie*, 2(1), 3-6.

Herbert, E.W., Dittmer, K.E. (2017). Case Report Acute and chronic oxalate toxicity in Miniature Horses associated with soursob (*Oxalis pes-caprae*) ingestion. 29, 549-557. https://doi.org/10.1111/eve.12605

Hesse, M. (2002). Alkaloids: Nature's Curse or Blessing? Wiley- VCH, New York.
Hiçsönmez, Ü., Ereeş, F.S., Özdemir, C., Özdemir, A., Çam, S. (2009). Determination of major and minor elements in the malva sylvestris L. from turkey using ICP-OES techniques. *Biological Trace Element Research*, 128(3), 248-257. https://doi.org/10.1007/s12011-008-8270-0
Ho, S.C., Tang, Y.L., Lin, S.M., Liew, Y.F. (2010). Evaluation of peroxynitrite scavenging capacities of several commonly used fresh spices. *Food Chemistry*, 119, 1102-1107.
Hoffman, P.C., Shaver, R.D., Combs, D.K., Undersander, D.J., Bauman, L.M., Seeger, T.K. (2001). Understanding NDF Digestibility of Forages. *Focus on Forage*, 3(10), 3-5.
Hopkins, XG. (2003). Physiologie végétale. Edition De Boeck et Larcier. 515pp.
Hosni, K., Hassen, I., Sebei, H., Casabianca, H. (2013). Secondary metabolites from *Chrysanthemum coronarium* (Garland) flowerheads: Chemical composition and biological activities. *Industrial Crops and Products*, 44(2013), 263-271. https://doi.org/10.1016/j.indcrop.2012.11.033
Hughes, M., Mlambo, V., Lallo, C.H.O., Jennings, P.G.A. (2012). Potency of microbial inocula from bovine faeces and rumen fluid for in vitro digestion of different tropical forage substrates. *Grass Forage Science*, 67, 263-273.
Hussain, M., Farooq, S., Merfield, C., Jabran, K. (2018). Mechanical weed control. Chapter In *Non-Chemical Weed Control*. Elsevier Inc. https://doi.org/10.1016/B978-0-12-809881-3.00008-5
Idris, K.I., Elamin, S.E., Omer, S.O., Mohamed, E.S. (2011). Critical period of weed competition and yield losses of irrigated common bean (*Phaseolus vulgaris* L.) in Northern Sudan. Gezira Journal of Agricultural Science, 9(2), 1-8.
Irfan Emre, I., Kurs¸ M., Yılmaz, O., Erecevit, P. (2011). Some biological compounds, radical scavenging capacities and antimicrobial activities in the seeds of *Nepeta italica* L. and *Sideritis montana* L. subsp. montana from Turkey. *Grasas y Aceites*, 62(1), 68-75. https://doi.org/10.3989/gya.033210
Ishak, A.H., Shafie, N.H., Esa, N.M., Bahari, H., Ismail, A. (2018). From weed to medicinal plant: Antioxidant capacities and phytochemicals of various extracts of *Mikania micrantha*. *International Journal of Agricultural and Biology*, 20(3), 561-568. https://doi.org/10.17957/IJAB/15.0522
Isla, M.I., Craig, A., Ordoñez, R., Zampini, C., Sayago, J., Bedascarrasbure, E., Alvarez, A., Salomón, V., Maldonado, L. (2011). Physico chemical and bioactive properties of honeys from Northwestern Argentina. LWT-*Food Science and Technology*, 44(9), 1922-1930. https://doi.org/10.1016/j.lwt.2011.04.003
Ito, C., Itoigawa, M., Onoda, S., Hosokawa, A., Ruabgrungsi, N., Okuda, T., Tokuda, H., Nishino, H., Furukawa, H. (2005). Chemical constituents of *Murraya siamensis*: three coumarins and their anti-tumor promoting effect. *Phytochemistry*, 66(5), 567-572.
Ivana, B., Dragutin, Đ., Pavle, M., Leka, M. (2018). Mineral composition of plant extracts from. *Archives for Technical Sciences*, 19(1), 85-90. https://doi.org/10.7251/afts.2018.1019.085B
Jain, C. (2019). Bioactivity of secondary metabolites of various plants: A review. *International Journal of Pharmaceutical Sciences and Research*, 10(2), 494-504. https://doi.org/10.13040/IJPSR.0975-8232
James, L.F. (1978). Oxalate Poisoning in Livestock. In Effects of Poisonous Plants on Livestock (Issue 2). *Academic Press*, INC. https://doi.org/10.1016/b978-0-12-403250-7.50020-0
Jan, S., Wali, S., Ahmad, N., Ahamd, I., Hamayun, M. (2014). Pharmacognostic and pharmacological studies of leaf, stem, and fruit of *Emex spinosa* (L.) Campd. *International Journal of Biosciences*, 5(10), 37-45. https://doi.org/10.12692/ijb/5.10.37-45

Jan, S., Wali, S., Ahmad, N., Ahamd, I., Hamayun, M., Shah, N. (2014). Pharmacognostic and pharmacological studies of leaf, stem, and fruit of *Emex spinosa* (L.) Campd. *International Journal of Biosciences*, 5(10), 37-45. http://dx.doi.org/10.12692/ijb/5.10.37-45

Jannoyer, M.L., Le Bellec, F., Lavigne, C., Achard, R., Malézieux, E. (2011). Choosing cover crops to enhance ecological services in orchards: A multiple criteria and systemic approach applied to tropical areas. *Procedia Environmental Sciences*, 9, 104-112. https://doi.org/10.1016/j.proenv.2011.11.017

Jauzein, P. (1995). Flore des champs cultivés. INRA, Paris, 898pp.

Jauzein, P. (2011). Flore des champs cultivés, Versailles : Ed. Quae, 903pp.

Jbilou, R., Amri, H., Bouayad, N., Ghailani, N., Ennabili, A., Sayah, F. (2008). Insecticidal effects of extracts of seven plant species on larval development, α-amylase activity, and offspring production of *Tribolium castaneum* (Herbst) (Insecta: Coleoptera: Tenebrionidae). *Bioresource Technology*, 99(5), 959-964. https://doi.org/10.1016/j.biortech.2007.03.017

Joel, E.L., Bhimba, B.V. (2013). Biological activity of secondary metabolites isolated from mangrove fungi Neurospora crassa. *Journal of Environmental Biology*, 34(4), 729-32.

Jordan, M.A., Leslie, W. (2004). Microtubules as a target for anticancer drugs. *Nature Reviews Cancer*, 4(4), 253-265.

Kabera, J., Semana, E., Mussa, A., He, X. (2014). Plant Secondary Metabolites: Biosynthesis, Classification, Function and Pharmacological Classification, Function and Pharmacological Properties. *Journal of Pharmacy and Pharmacology*, 2(7), 377-392.

Kaid Slimane, L. (2000). Etude de la relation sol-végétation dans la région nord des Monts de Tlemcen. Thes. Mag. Dpt. Fac. Sci., Univ. Tlemcen, 120 pp.

Kamalak, A., Canbolat, O., Gurbuz, Y., Ozkan, C.O., Kizilsimsek, M. (2005). Determination of nutritive value of wild mustard, *Sinapis arvensis* harvested at different maturity stages using in situ and in vitro measurements. *Asian-Australasian Journal of Animal Sciences*, 18(9), 1249-1254. https://doi.org/10.5713/ajas.2005.1249

Karkour, L. (2012). La dynamique des mauvaises herbes sous l'effet des pratiques culturales dans la zone des plaines intérieures. Thes. Mag. Fac. Sciences of Nature and Life. Univ. Ferhat Abbas Sétif. 104 pp.

Karkour, L., Fenni, M. (2016). Effet des pratiques culturales sur la dynamique des flores adventices des terres cultivées dans la zone semi-aride (Algérie). *Revue Agriculture*, 1, 52-61.

Kazi Tani, C. (2010). Contribution à l'étude des communautés d'adventices des cultures du secteur phytogéographique oranais (Nord-Ouest algérien) : Aspects botanique, agronomique et phyto-écologique. Thes. Doc., 226pp.

Kazi Tani, C., Le Bourgeois, T., Munoz, F. (2010). Contribution à l'étude des communautés d'adventices des cultures du secteur phytogéographique Oranais (nord-ouest algérien) : Aspects botanique, agronomique et phyto-écologique. Flora Mediterranea, 20, 29-46.

Kelton, J.A., Price, A.J. (2000). Weed Science and Management, in soils, plant growth and crop production -Vol. III. Encyclopedia of Life Support Systems (EOLSS), 10 pp.

Kennouche, S., Bicha, S., Bentamene, A., Crèche, J., Benayache, F. (2016). In Vitro Antioxidant Activity, Phenolic and Flavonoid Contents of Different Polarity Extracts from *Chrysanthemum segetum* L. Growing in Algeria. *International Journal of Pharmacognosy and Phytochemical Research*, 8(9), 1522-1525

Khan, Z.I., Ashraf, M., Valeem, E.E. (2006). Forage mineral status evaluation: the influence of pastures. *Pakistan Journal of Botany*, 38(4), 1043-1054.

Khan R., Khan M. A., Sultan S., Mawat K. B., Khan I., Hassan G., Shah H.U. (2013). Nutritional quality of sixteen terrestrial weeds for the formulation of cost-effective animal feed. *The Journal of Animal and Plant Sciences*, 23(1), 75-79.

Kheddam, M. Adane, N. (1996). Contribution à l'étude phytoécologique des mauvaises herbes des cultures pérennes dans la plaine de la Mitidja. I. Aspect floristique. *Annales Agronomiques*, 17(1-2), 1-26.

Kim, H.S. (2019). Effect of *Calendula arvensis* extract on anti-melanogenesis and antimicrobial activity. *Journal of Applied Biological Chemistry*. 62(2), 137-141. https://doi.org/10.3839/jabc.2019.019

Kim, J.H., Campbell, B.C., Mahoney, N., Chan, K.L., Molyneux, R.J., Blajee, A. (2010). Augmenting the activity of antifungical agents against *Aspergilla* using structural analogues of benzoic acid as chemosensitizing agents. *Fungal Biology*, 114(10), 817-824.

Kim, J.Y., Lim, H.J., Lee, D.Y., Kim, D.H., Jeon, R., Ryu, J.H. (2009). In vitro anti-inflammatory activity of lignans isolated from *Mgnolia fargesii*. *Bioorganic and Medicinal chemistry letters*, 19(3), 937-940.

Kossel, A. (1891). Ueber abkömmlinge der phenylamidoessigsäure. Berichte der deutschen chemischen Gesellschaft, 24(2), 4145-4156.

Ksouri, R., Medgiche, W., Fellah, H., Trabelsi, N., Boulaaba, M., Smaoui, A., Abdelly, C. (2008). Influence of biological, environmental, and technical factors on phenolic content and antioxidant activities of Tunisian halophytes. *Comptes Rendus Biologies*, 331(11), 865-873.

Kubata, B.k., Nagamune, K., Murakami, N., Merkel, P., Kabututua, Z., Martin, S.K., Kalulug, T., Mustakuk, H., Hoshida, M., Ohnishi-Kameyama, M., Kinoshita, T., Duszenko, M., Uradea, Y. (2005). *Kola acuminate* proanthocyanidins: a class of anti-trypanosomal compounds effective against *Trypanosome brucei*. *International Journal for Parasitology*, 35(1), 91-103.

Kumar, A., Singh, S.P., Bhakuni, R.S. (2014). Secondary metabolites of Chrysanthemum genus and their biological activities. *Current Science*, 89(9), 1489-1501.

Kumarasamy, Y., Byres, M., Cox, P.J., Jaspars, M., Nahar, L., Sarker, S.D. (2007). Screening seeds of some Scottish plants for free radical scavenging activity. Phytotherapy Research, 21(7), 615-621. https://doi.org/10.1002/ptr

kyanova, L.D., Storozheva, Z.I., Proshin, A.T. (2007). Corrective effect of flavonoid containing preparation extralife on the development of Parkinson's syndrome. *Bulletin of Experimental Biology and Medicine*, 144, 42-45.

Labreuche, J., Laurent, F., Roger-Estrade, J. (2014). Faut-il travailler le sol ? Acquis et innovations pour une agriculture durable, Versailles, Quae Ed, 192 pp.

Lardy, G. (2018). Forage Nutrition for Rumin*ants*. North Dakota State Univ. Fargo, North Dakota. *AS 1250*, 1-16.

Le Bellec, F. (2015). Caractérisation des communautés adventices des vergers d'agrumes de la Réunion et détermination d'espèces favorables à la mise en place de la lutte biologique par conservation. 1-51.

Lenzi, A., Orlandini, A., Bulgari, R., Ferrante, A., Bruschi, P. (2019). Antioxidant and mineral composition of three wild leafy species: A comparison between microgreens and baby greens. *Foods*, 8(10), 1-19. https://doi.org/10.3390/foods8100487

Lien, E.J., Ren, S., Bui, H.H., Wang, R. (1999). Quantitative structure-activity relationship analysis of phenolic antioxidants, *Free Radical Biology and Medecine*, 26, 285-294.

Loisel, R., Gamila, H. (1993). Traduction des effets du débroussaillement sur les écosystèmes forestiers et pré-forestiers par un indice de perturbation. Annales de la Société des Sciences Naturel et d'Archéologiede Toulon et du Var 45, 123-132

Longo, L., Scardino, A., Vasapollo, G. (2007). Identification and quantification of anthocyanins in the berries of *Pistacia lentiscus* L., *Phillyrea latifolia* L. and *Rubia peregrina* L. *Innovative Food Science and Emerging Technologies*, 8(3), 360-364. https://doi.org/10.1016/j.ifset.2007.03.010

Lopes, F., Ruh, K., Combs, D.K. (2015). Validation of an approach to predict total-tract fiber digestibility using a standardized in vitro technique for different diets fed to high-producing dairy cows. *Journal of Dairy Sciences*, 98, 2596-2602.

López, V., Akerreta, S., Casanova, E., Garcia-Mina, J.M., Cavero, R.Y., Calvo, M.I. (2008). Screening of Spanish medicinal plants for antioxidant and antifungal activities. *Pharmaceutical Biology*, 46(9), 602–609. https://doi.org/10.1080/13880200802179634

Lopez-Alonso, M. (2012). Trace Minerals and Livestock: Not Too Much Not Too Little. *International Scholarly Research Network Veterinary Science*, Article ID 704825, 18pp. https://doi.org/10.5402/2012/704825

Lowell, J.R. (1848). A fable for critics. In: The Complete Poetical Works of James Russell Lowell. Riverside Press, Cambridge, MA, 117-148 pp.

Lucio, M., Nunes, C., Gaspar, D., Ferreira, H., Lima, J.L.F.C., Reis, S. (2009). Antioxidant activity of vitamin E and Trolox: understanding of the factors that govern lipid peroxidation studies *in vitro*. *Food Biophysics*, 4, 312-320.

Lutsenko, Y., Bylka, W., Matławska, I., Darmohray, R. (2010). *Hedera helix* as a medicinal plant. *Herba Polonica*. 56(1), 83-96.

Mader. M., Amberg-Fisher, V. (1982). Role of peroxidase in lignifications of tobacco cells. Oxidation of nicotinimide adenine dinucleotide and formation of hydrogen peroxide by cell wall peroxidises. *Plant physiology*, 70, 1128-1131.

Mahmoudi, H., Hosni, K., Zaouali, W., Amri, I., Zargouni, H., Hamida, N. Ben Kaddour, R., Hamrouni, L., Nasri, M. Ben, Ouerghi, Z. (2016). Comprehensive phytochemical analysis, antioxidant, and antifungal activities of *Inula viscosa* Aiton. leaves. *Journal of Food Safety*, 36(1), 77-88. https://doi.org/10.1111/jfs.12215

Maillet, J. (1981). Evolution de la flore adventice dans le montpelliérains sous la pression des techniques culturales. Thes. Doc., Univ. des Sciences et Techniques du Languedoc, Montpellier.

Maiza-Benabdesselam, F., Chibane, M., Madani, K., Max, H., Adach, S. (2007). Determination of isoquinoline alkaloids contents in two Algerian species of *Fumaria* (*Fumaria capreolata* and *Fumaria bastardi*). *African Journal of Biotechnology*, 6(21), 2487-2492. https://doi.org/10.5897/ajb2007.000-2394

Majda, F., Al suhbi, Mohamed A. Al aib, Salem A. El shatshat. (2017). Allelopathic potential of common mallow (*Malva sylvestris* L.). *Imperial Journal of Interdisciplinary Research*, 3(2), 1742-1748.

Makni, S., Tounsi, S., Rezgui, F., Trigui, M., Bouassida, K.Z. (2019). *Emex spinosa* (L.) Campd. ethyl acetate fractions effects on inflammation and oxidative stress markers in carrageenan induced paw oedema in mice. *Journal of Ethnopharmacology*, 234, 216-224. https://doi.org/10.1016/j.jep.2018.12.015

Mamyrbékova-Békro, J.A., Konan, K.M., Békro, Y.A., Djié Bi, M.G., Zomi Bi, T. J., Mambo, V., Boua Boua, B. (2008). Phytocompounds of the Extracts of Four Medicinal Plants of Côte d'Ivoire and assessment of their potential antioxidant by thin layer chromatography. *European Journal of Scientific Research*, 24, 219-228.

Mangan, J.L. (1982). The nitrogenous constituents of fresh forages. Proc Brit Society of Animal Production. Occasional Publication No 6. England, UK. Sept 6th - 10th, 25-40 pp.

Marten, G.C., Andersen, R.N. (1975). Forage Nutritive Value and Palatability of 12 Common Annual Weeds. *Crop Science*, 15, 821-827.

Mas, M.T., Verdú, A.M.C (2005). Biodiversidad de la flora arvense en cultivos de mandarino según el manejo del suelo en las interfilas. *Boletín de sanidad vegetal. Plagas*, 31, 231-241.

Mashaly, I.A., Awad, E.R. (2003). Weed flora of orchards in the Nile Delta, Egypt: Floristic Features. *Asian Journal of Plant Sciences*, 2(3), 314-324.

Masquelier, J., Dumon, M., Dumas, J. (1979). Stabilisation des collagènes par des oligomères procyanidoliques. *Acta thérapeutiquen*, 1, 101-104.

Masuda, T., Yonemori, S., Oyama, Y., Tkeda, Y., Tanaka, T., Andoh, T. (1999). Evaluation of the antioxidant activity of environmental plants: Activity of the leaf extracts from seashore plants. *Journal of Agricultural and Food Chemistry*, 47(4), 1749-1754.

Mauricio, R.M., Owen, E., Mould, F.L., Givens, I., Theodorou, M.K., France, J., Davis, D.R., Dhanoa, M.S. (2001). Comparison of bovine rumen liquor and bovine faeces as inoculum for an in vitro gas production technique for evaluating forages. *Animal Feed Science and Technology*, 89, 33-48.

Maxia, A., Frau, M.A., Foddis, C., Lancioni, M.C., Kasture, V., Kasture, S. (2012). Ethanolic extract of *Rubia peregrina* L. (Rubiaceae) inhibits haloperidol-induced catalepsy and reserpine-induced orofacial dyskinesia. *Natural Product Research.* 26(5), 438-445. https://doi.org/10.1080/14786419.2010.511015

McDowell, L.R., Arthington, J.D. (2005). Minerals for Grazing Ruminants in Tropical Regions. Extension Bulletin Animal Science Department, Univ. of Florida, 86 pp. http://ufdc.ufl.edu/UF00054813/00001

McMurry, J.E. (2015). Organic chemistry with biological applications. In: Secondary Metabolites: An Introduction to Natural Products Chemistry. Stamford, USA: Cengage Learning Ltd, 1016-1046 pp.

Meena, A.K., Pal, B., Panda, P., Sannd, R., Rao, M.M. (2010). A review on *Rubia cordifolia*: its phyto constituents and therapeutic uses. Drug Invention Today, 2(5), 244-246.

Mekkioui, A. (1989). Etude bioclimatique des méditerranéennes occidentales et de L'ouest algerien. D.E.S. Thes. Dep. Bio. Fac.Sci.Univ. Tlemcen, 111 pp.

Melakhessou, Z., Demnati, F., Boubaker, Z. (2020). Diagnostic de la diversité des plantes adventices dans les agrosystèmes : cas des champs de blé dans les Aurès. *Bulletin de la Société Royale des Sciences de Liège*, 89, 39-54.

Mesli, K. (2001). Contribution à une étude syntaxonomique et écologique Des Mattorals de la région de Tlemcen. Thes. Mag. en écologie végétale. Univ de Tlemcen.

Michal, G., Schomburg, D. (2013). Biochemical Pathways: An Atlas of Biochemistry and Molecular Biology. 2nd ed. Wiley, 416 pp.

Middleton, E., Kandaswami, C., Theoharides, T.C. (2000). The effects of plant flavonoids on mammalian cells: implications for inflammation, heart disease, and cancer. *Pharmacological Reviews*, 52(4), 673 - 751.

Miguel, M.G. (2010). Antioxidant and Anti-Inflammatory Activities of Essential Oils: A Short Review. *Molecules*, 15, 9252-9287.

Miguel, M.G., Nunes, S., Dandlen, S.A., Cavaco, A.M., Dulce, M. (2014). Phenols, flavonoids, and antioxidant activity of aqueous and methanolic extracts of propolis (*Apis mellifera* L.) from Algarve, South Portugal. *Food Science and Technology*, 34(1), 16-23.

Miller, N.J., Sampson, J. Candeias L.P. (1996). Antioxidant activities of carotenes and xanthophylls, *FEBS Letter*, 384, 240-2.

Mills, S., Bone, K. (2000). Principles and Practice of Phytotherapy–Modern Herbal Medicine. New York: Churchill Livingstone, 31-34 pp.

Millspaugh, C.F. (1887). American Medicinal Plants: An illustrated and descriptive guide to the medicinal plants used in homeopathic remedies. Boericke and Tafel, Philadelphia, USA, 494 pp.

Minson, D.J. (1990). Protein In: Forage in Ruminant Nutrition. Minson D.J. (Ed.). Academic Press Inc. San Diego, California, USA. 162-207 pp.

Miser-Salihoglu, E., Akaydin, G., Caliskan-Can, E., Yardim-Akaydin, S. (2013). Evalution of antioxidant activity of various herbal folk medicines. *Journal of Nutrition and Food Science.* 3(5), 1-9. https://doi.org/10.4172/2155-9600.1000222

Mohajer, S., Taha, R.M., Ramli, R. Bin, Mohajer, M. (2016). Phytochemical constituents and radical scavenging properties of *Borago officinalis* and *Malva sylvestris*. *Industrial Crops and Products.* 94, 673-681. https://doi.org/10.1016/j.indcrop.2016.09.045

Mohammed, C.B., Chahid, B., Fatima, B., Fatima-Zohra, S., Meriem, B., Farid, C. (2014). Antioxidant activity, phenolic and flavonoid content in leaves, flowers, stems and seeds of mallow (*Malva sylvestris* L.) from North Western of Algeria. *African Journal of Biotechnology*, 13(3), 486-491. https://doi.org/10.5897/ajb2013.12833

Mohiuddin, A.K. (2019). Chemistry of Secondary Metabolites. *Annals of Clinical Toxicology*, 2(1), 1014.

Moon, J.K., Shibamoto, T. (2009). Antioxidant Assays for Plant and Food Components. *Journal of Agricultural Food Chemistry*, 57, 1655-1666.

Mukherjee, P.K., Kumar, V., Mal, M., Houghton, P.J. (2007). Acetylcholinesterase inhibitors from plants. *Phytomedicine*, 14(4), 289-300. https://doi.org/10.1016/j.phymed.2007.02.002

Munda, N. (2010). Identification de polyphénols, évaluation de leur activité antioxydante et étude de leurs propriétés biologiques. Thes. Doc., Ecole doctorale SESAMES, France. 160 pp.

Murray, R.D.H., Mendez, J., Brown, S.A. (1982). The natural coumarins, Wiley, New York, 714 pp.

Murex, L. (2010). Phytochemical Screening for active compounds in Pedalium. *Science and Technology*, 2(1), 110-114.

Nashiki, M., Narita, H., Higashiyama, Y. (2005). Herbage mass, nutritive value, and palatability of five grass weeds for cattle in the northern Tohoku region in Japan. *Weed Biology and Management*, 5, 110-117.

Nasr, N., Miyandeh, H.B. (2013). Weeds identification in west of Mazandaran Province citrus orchards (Iran). *American Journal of Research Communication*, 1(6), 27-38.

Newman, Y.C., Lambert, B., Muir, J.P. (2009). Defining forage quality. *EDIS Publication SS-AGR*, 1-13 pp. http://soilcrop.tamu.edu/publications/FORAGE/PUB_forage_Defining_Forage Quality.pdf

Nikolakaki, A., Christodoulakis, N.S. (2004). Leaf structure and cytochemical investigation of secretory tissues in *Inula viscosa*. *Botanical Journal of the Linnean Society*, 144(4), 437–448. https://doi.org/10.1111/j.1095-8339.2003.00264.x

Nikolova, M., Evstatieva, L., Nguyen, T.D. (2011). Screening of plant extracts for antioxidant properties. *Botanica Serbica*, 35(1), 43-48.

Nirmaljit, K., Rattanpal, H.S. (2017). Weed management in citrus orchards. *Rashtriyakrishi*, 12(1), 122-122.

Norris, R.F., Kogan, M. (2005). Ecology of interactions between weeds and arthropods. *Annual Review of Entomology*, 50, 479-503. https://doi.org/10.1146/annurev.ento.49.061802.123218

NRC (National Research Council), (1981). Nutrient requirements of goats: angora, dairy, and meat goats in temperate and tropical countries. National Academy Press. Washington, USA, 91 pp.

NRC (National Research Council), (1985). Nutrient requirements of sheep. 6th Revised Ed. National Academy Press, Washington, USA, D.C.

NRC (National Research Council), (2000). Nutrient requirements of beef cattle. 7th Ed. National Academies Press, Washington, USA, D. C.

NRC (National Research Council), (2001). Nutrient Requirements of Dairy Cattle. 7th Ed. National Academy Press. Washington, USA, 381 pp.

Ochocka, R.J., Rajzer, D., Kowalski, P., Lamparczyk, H. (1995). Coumarins from *Chrysanthemum*. *Electrophoresis Journal of Chromatography A.*, 709 (1), 197-202.

Oelberg, K. (1956). Factors affecting the nutritive value of range forages. Journal of range Management, 9, 220-225.

Ohloff, G., Riechstoffe und Geruchssinn. (1990). Scent and Fragrances: The Fascination of Odors and Their Chemical Perspectives. New York: Springer, Berlin, 1994.

Okamura, H., Mimura, A., Yakou, Y., Niwano, M., Takahara, Y. (1993). Antioxydant activity of tannins and flavonoids in *Eucalyptus rostrata*. *Phytochemistry*, 33(3), 557-561.

Okunade, A.L., Elwin-Lewis, M.P.F., Lewis, W.H. (2004). Natural antimycobacterial metabolites: Current status. *Phytochemistry*, 65, 1017-1032.

Olesen, J.E., Hansen, P.K., Berntsen, J., Christensen, S. (2004). Simulation of above-ground suppression of competing species and competition tolerance in winter wheat varieties. *Field Crops Research*, 89, 263-280. https://doi.org/10.1016/j.fcr.2004.02.005

Olivier, L., Muracciole, M., Reduron, J.P. (1995). Premiers bilans sur la flore des îles de la Méditerranée. État des connaissances et conservation. *Ecologia Mediterranea*, 21, 355-372.

Omezine, A. (2011). Introduction récente de certaines espèces adventices des cultures en Tunisie. *Bulletin OEPP/EPPO Bulletin*, 41, 77-84.

Omezine, A., Teixeira, J.A. (2012). Floristic biodiversity of weed communities in relation to conventional and organic farming. Bioremediation, *Biodiversity and Bioavailability*, 6(1), 61-69.

Omezzine, F., Rinez, A., Ladhari, A., Farooq M., Haouala, R. (2011). Allelopathic potential of *Inula viscosa* against crops and weeds. *International Journal of Agriculture and Biology*, 13(6), 841-849.

Ondua, M., Njoya, E.M., Abdalla, M.A., Mcgaw, L.J. (2019). Anti-inflammatory and antioxidant properties of leaf extracts of eleven South African medicinal plants used traditionally to treat inflammation. *Journal of Ethnopharmacology*. 234, 27–35. https://doi.org/10.1016/j.jep.2018.12.030

Onen, H., Akdeniz, M., Farooq, S., Hussain, M., Ozaslan, C. (2018). Weed flora of citrus orchards and factors affecting its distribution in western Mediterranean region of Turkey. *Planta Daninha*, 36(54), 1-14. https://doi.org/10.1590/S0100-83582018360100036

Oran, S.A., Al-Eisawi, D.M. (1998). Checklist of medicinal plants in Jordan. In: *Dirasat, Medical and Biological Sciences*, 25(2), 84-112.

Orhan, N., Gökbulut, A., Deliorman Orhan, D. (2017). Antioxidant potential and carbohydrate digestive enzyme inhibitory effects of five *Inula* species and their major compounds. *South African Journal of Botany*, 111, 86-92. https://doi.org/10.1016/j.sajb.2017.03.040

Oyaizu, M. (1986). Studies on products of browning reaction prepared from glucose amine. *Japanese Journal of nutrition*, 44, 307-315.

Ozenda, P. (1991). Flore et végétation du Sahara. 2éme éd., (Paris : CNRS).

Özer, N. (2004). Çerkeş (Çankırı) yöresinde kullanılan halk ilaçları. *Hacettepe Eczacılık Fakültesi dergisi*, 24(2), 67-80.

Özgen, U., Houghton, P.J., Ogundipe, Y., Coskun, M. (2003). Antioxidant and antimicrobial activities of *Onosma argentatum* and *Rubia peregrina*. *Fitoterapia*. 74(7-8), 682-685. https://doi.org/10.1016/S0367-326X(03)00161-8

Ozkan, E., Karakas, F.P., Yildirim, A.B., Tas, I., Eker, I., Yavuz, M.Z., Turker, A.U. (2019). Promising medicinal plant *Inula viscosa* L.: Antiproliferative, antioxidant, antibacterial and phenolic profiles. *Progress in Nutrition*, 21(3), 652-661. https://doi.org/10.23751/pn.v21i3.7186

Pagare, S., Bhatia, M., Tripathi, N., Pagare, S., Bansal, Y.K. (2015). Secondary metabolites of plants and their role: Overview. *Current Trends in Biotechnology and Pharmacy*, 9(3), 293-304.

Palmquist, D.L. (1976). A Kinetic Concept of Lipid Transport in Ruminants. A Review. *Journal of Dairy Science*, 59(3), 355-363. https://doi.org/10.3168/jds.S0022-0302(76)84212-9

Păltinean, R., Mocan, A., Vlase, L., Gheldiu, A. M., Crişan, G., Ielciu, I., Voştinaru, O., Crişan, O. (2017). Evaluation of polyphenolic content, antioxidant, and diuretic activities of six *Fumaria* species. *Molecules*, 22(4), 1-14. https://doi.org/10.3390/molecules22040639

Panglossi, H.V. (2006). Antioxidants: new research; Ed: NOVA, 3-13 pp.

Passalacqua, N., Guarrera, P., De Fine, G. (2007). Contribution to the knowledge of the folk plant medicine in Calabria region (Southern Italy). *Fitoterapia*, 78(1), 52-68.

Péreza, M.P., Navas-Cortésb, J.A., Pascual-Villalobosa, M.J., Castillob, P. (2003). Nematicidal activity of essential oils and organic amendments from Asteraceae against root-knot nematodes, *Plant Pathology*, 52, 395-401.

Perkins, A.E. (1957). The effect of rations excessively high and extremely low in protein content on dairy cows. Ohio Agric. Exp. Stn. Res. Bull. 799 pp.

Piluzza, G., Bullitta, S. (2011). Correlations between phenolic content and antioxidant properties in twenty-four plant species of traditional ethnoveterinary use in the Mediterranean area. *Pharmaceutical Biol*ogy, 49(3), 240-247. https://doi.org/10.3109/13880209.2010.501083

Pirie, N.W. (1955). Protections. In: Peach, K. and Tracey, M.W. (eds.): Moderne Methoden der Pflanzenanalyse, Band 4, Berlin.

Platt, B.S., Heard, C.R.C., Stewart, R.J. (1964). Experimental protein-calorie deficiency, 445 pp. In H. N. Munro, Mammalian protein metabolism, vol. 2. Academic Press, New York.

Pop, C.E., Pârvu, M., Arsene, A.L., Pârvu, A.E., Vodnar, D.C., Tarcea, M., Toiu, A.M., Vlase, L. (2017). Investigation of antioxidant and antimicrobial potential of some extracts from *Hedera helix* L. *Farmacia*, 65(4), 624-629.

Porqueddu, C. (2001). Screening germplasm and varieties for forage quality: Constraints and potentials in annual medics. In: Delgado I. (ed.), Lloveras J. (ed.). Quality in lucerne and medics for animal production. Zaragoza : CIHEAM, 2001. p. 89-98 (Options Méditerranéennes : Série A. Séminaires Méditerranéens ; n. 45)

Protopapadakis, E. (1985). Changement de la flore adventice des vergers d'agrumes en Crète sous la pression du désherbage chimique. Agronomie, *EDP Sciences*, 5(9), 833-840. https://doi.org/10.1051/agro:19850910

Quennesson, S. Oste, S. (2017). Gestion des adventices par la couverture végétale du sol en agriculture et espaces verts : Panorama des techniques utilisées. Presented at the 6ème Conférence sur les moyens alternatifs de protection pour une production intégrée, Lille, 21-23 Mars.

Quézel, P., Barbero, M., Bonin, G., Loisel, R. (1991). Pratiques Agricoles et couvert forestier en région méditerranéenne humide et sub-humide (Paris : CNRS).

Quézel, P., Santa, S. (1962-1963). Nouvelle Flore de l'Algérie et des Régions Désertiques Méridionales. Éd. C.N.R.S, Paris, Tome 1 (1962), 565 pp, Tome 2 (1963), 571-1170 pp.

Quézel, P., Santa, S. (1963). Nouvelle flore de l'Algérie et des régions désertiques méridionales (Paris : CNRS).

Rached. W, Benamar, H., Bennaceur, M., Marouf, A. (2010). Screening of the antioxidant potential of some Algerian indigenous plants. *Journal of Biological Sciences*. 10(4), 316-324.

Rad, J.S., Alfatemi, M.H., Rad, M.S., Jyoti, D. (2013). Phytochemical and antimicrobial evaluation of the essential oils and antioxidant activity of aqueous extracts from flower and stem of *Sinapis arvensis* L. *American Journal of Advanced Drug Delivery*. 1(1), 001-010.

Radojević, I.D., Stankovic, M., Topuzovic, M. (2012). Antioxidative and antimicrobial properties of different extracts from *Sideritis montana* L. *Romanian Biotechnological Letters.* 17(2), 7014-7022.

Ramin, M., Lerose, D., Tagliapietra, F., Huhtanen, P. (2015). Comparison of rumen fluid inoculum *vs.* faecal inoculum on predicted methane production using a fully automated in vitro gas production system. *Livestock Sciences,* 181, 65-71.

Ramirez Lozano, R. (2015). Nutrition of grass carbohydrates. In: Grass Nutrition. Edited by R. Ramirez Lozano. Palibrio Press, Bloomington, Indiana USA. 29-56 pp.

Raunkiaer, C.C. (1905). Types biologiques pour la géographie botanique. *Kongelige Danske Videnskabernes Selskabs Forhandlinger*, 5, 347-438.

Raunkiaer, C.C. (1934). The Life Forms of Plants and Statistical Plant Geography; Being the Collected Papers of C Raunkiaer (Oxford: Oxford Univ. Press).

Re, R., Pellegrini, N., Proteggente, A., Pannala, A., Yang, M., Rice-Evans, C. (1999). Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radical Biology and Medicine*, 26, 1231-1237.

Rebaz, Z., Chahid-Shaukat, S., Siddiqui, I.A. (2001). Allelopathic potential of *Anagallis arvensis*: A cosmopolitan weed. *Pakistan Journal of Biological Sciences*, 4(4), 446-450.

Reyburn, H., Mtove, G., Hendriksen, I., Von Seidlein, L. (2009). Oral qui-nine for the treatment of uncomplicated malaria. *British Medical Journal (Clinical Research Edition),* 339, b2066. https://doi.org/10.1136/bmj.b2066

Rice-Evans, C.A., Packer, L. (2003). Flavonoids in health and disease. New York: Marcel Dekker Inc., 504 pp.

Rimani, M., Mzabri, I., Chafik, Z., Berrichi, A. (2019). Weeds flora associated with Saffron (*Crocus sativus* L.) in Morocco. *Materials Today: Proceedings*, 13, 1108-1114. https://doi.org/10.1016/j.matpr.2019.04.078

Robert, M.F., Wink, M. (1999). Alkaloids- Biochemistry, Ecology, and Medicinal Applications. Book Reviews. *Phytochemistry*, 52, 1177-1180.

Romojaro, A., Botella, M.Á., Obón, C., Pretel, M.T. (2013). Nutritional and antioxidant properties of wild edible plants and their use as potential ingredients in the modern diet. *International Journal of Food Sciences and Nutrition*, 64(8), 944-952. https://doi.org/10.3109/09637486.2013.821695

Rueffer, M. (1988). Biosynthetic studies of Protober berine and related Alkaloids use plant cell cultures" in National Product chemistry III (Eds. Atta-ur-Rehman and P.W. Le Quesne), 247-256. Springer Verlag, Berlin.

Ryan, M.F. (1991). The role of magnesium in clinical biochemistry: an overview. *Annals of Clinical Biochemistry:* 28, 19-26.

Salama, F., Abd El-Ghani, M., El-Tayeh, N., Amro, A., Abdrabbu, H. (2016). Weed flora of common crops in desert reclaimed arable lands of southern Egypt. *Taeckholmia*, 36(1), 58-76. https://doi.org/10.21608/taec.2016.11940

Salim, H., Rimawi, W.H., Mjahed, A. (2017). Analysis of extracts from Palestinian *Inula viscosa* for their phenolic, flavonoid and lipid contents, antioxidant, antibacterial activity. *Journal of Chemistry and Biochemistry*, 5(1), 12-23. https://doi.org/10.15640/jcb.v5n1a

Salma, U., Khan, T., Shah, A.J. (2018). Antihypertensive efficacy of extract of Hedera helix in high salt-induced hypertensive Sprague-Dawley rats. *Asian Pacific Journal of Tropical Medicine*, 11(8), 473-479. https://doi.org/10.4103/1995-7645.240083

Sarikurkcu, C., Targan, S., Ozer, M.S., Tepe, B. (2017). Fatty acid composition, enzyme inhibitory, and antioxidant activities of the ethanol extracts of selected wild edible plants consumed as vegetables in the Aegean region of Turkey. *International Journal of Food Properties*, 20(3), 560-572. https://doi.org/10.1080/10942912.2016.1168837

Sarni-Manchado, P., Cheynier, V. (2006). Les polyphénols en agroalimentaires, Ed. Lavoisier (Tec et Doc), Paris, 300-398 pp.

Sathish Kumar (2003). Management of infertility due to mineral deficiency in dairy animals. In: Proceedings of ICAR summer school on "Advance diagnostic techniques and therapeutic approaches to metabolic and deficiency diseases in dairy animals". Held at IVRI, Izatnagar, UP (15th July to 4th Aug.). 128-137 pp.

Sayed, I., Cheloufi, H., Halilat, M.T., Eddoud, O. (2014). Contribution à l'étude quantitative des messicoles associées aux céréales conduites sous centre pivots dans la région de Ouargla (cas des périmètres céréaliers de Hassi Ben Abdallah). Revue Agriculture, 08, 04-09.

Sayed, O.H. (1993). Ecophysiological characteristics, distribution, and nutritional value of desert range plants in Qatar. *Qatar University Science Journal*, 13(2), 243-246.

Schmidt, S., Niklova, I., Pokorny, J., Farkas, P., Sekretar, S. (2003). Antioxidant activity of evening primrose phenolics in sunflower and rapeseed oils. *European Journal of Lipid Science and Technology*, 105, 427-435. https://doi.org/10.1002/ejlt.200300842

Schonewille, J.T. (2014). Magnesium in dairy cow nutrition: An overview Magnesium in dairy cow nutrition: an overview. Plant Soil, 368, 167-178. https://doi.org/10.1007/s11104-013- 1665-5

Schwarz, H., Liebhard, P., Ehrendorfer, K., Ruckenbauer, P. (2007). Potential growth and biomass productivity of *Miscanthus_giganteus* as affected by plant density and N-fertilization in central Greece. *Biomass and Bioenergyi*, 31,145 - 152.

Seca, L., Pinto, D.C. (2019). Biological Potential and Medical Use of Secondary Metabolites. *Medicines*, 6(2), 66. https://doi.org/10.3390/medicines6020066

Serag, M., Khedr, A., Rabei, S., Elkomy, A. (2015). Ecological Study on Weed Flora Growing in the Orchards of New Damietta. *Journal of Plant Production*, 6(12), 2003–2018. https://doi.org/10.21608/jpp.2015.52128

Serghini, K., Perez De Lugue, A., Castejon, M.M., Garcia, T.L., Jorrin, J.V. (2001). Sunflower (*Helianthus annuus* L.) response to broomraoe (*Orobanche cernua* loefl.) parasitism: induced synthesis and excretion of 7-hydroxylated simple coumarins. *Journal of Experimental Botany*, 52, 227-234.

Shahat, A.A., Ibrahim, A.Y., Ezzeldin, E., Alsaid, M.S. (2015). Acetylcholinesterase inhibition and antioxidant activity of some medicinal plants for treating neuro degenerative disease. *African Journal of Traditional, Complementary and Alternative Medicines*, 12(3), 97-103. https://doi.org/10.4314/ajtcam.v12i3.12

Shakoor, A., Zaib, G., Ur Rahman, A. (2018). Biological activities of three medicinal plants from district Mirpur, AJK, Pakistan. *Pakistan Journal of Pharmaceutical Sciences*, 31(6), 2341-2346.

Shaltout, K., El Keblawy, A., Mousa, M. (2008). Evaluation of the range plants quality and palatability for camel grazing in the United Arab Emirates. *Journal of Camelid Science*, 1, 1-13.

Sharifi-Rad, J., Hoseini-Alfatemi, S., Miri, A., Sharifi-Rad, M., Sharifi-Rad, M., Hoseini, M., Sharifi-Rad, M. (2016). Exploration of Phytochemical and Antibacterial Potentiality of *Anagallis arvensis* L. Extract against Methicillin-Resistant Staphylococcus aureus (MRSA). *British Biotechnology Journal*, 10(2), 1-8. https://doi.org/10.9734/bbj/2016/20505

Sharma, M.C., Joshi, C., Das, G. and Hussain, K. (2007). Mineral nutrition and reproductive performance of the dairy animals: a review. *Indian Journal of Animal Sci*ence, 77, 599-608.

Sharma, S.D., Singh, M. (2006). Gramoxone Inteon- a New Formulation for Weed Management in Citrus. *Proceedings of the Florida State Horticultural Society*, 119, 172-176.

Sheetal, S.K., Choudhary, S.K. and Sengupta, D. (2014). Mineral deficiency predisposes occurrence of retention of placenta in crossbred, *Veterinary World*, 7(12), 1140-1143.

Shills, M.E. (1997). Magnesium in dairy cow nutrition: an overview, *Plant Soil*, 368, 167-178. https://doi.org/10.1007/s11104-013-1665-5

Shweta, Rana, G.S., Duhan, A., Kaur, A. (2018). Evaluation of different herbicides on weed control, yield, and quality of sweet orange. *Chemical Science Review and Letters*, 7(26), 528-532.

Simmonds, H., Holst, P., Bourke, C. (2000). The palatability, and potential toxicity of Australian weeds to goats. National Library of Australia, Cataloguing in Publication entry: Simmonds, Helen. Australia, 156 pp.

Singh, S.N., Sengar, O.P.S. (1970). Investigation on milk and meat potentialities of Indian goats, 1965-70. Final Tech. Report Project A7-AH18. Raja Balwant Singh College, Bichpuri (Agra), India.

Singleton, V.L., Rossi, J.R. (1965). Colorimetry of total phenols with phosphomolybdic-phosphotungstic acid reagents. *American Journal of Enology and Viticulture*. 16, 144-158.

Siqueira, J.O., Nair, M.G., Hammerschmidt, R., Safir, G.R. (1991). Significance of phenolic compounds in plant-soil-microbial systems. *Critical Reviews in Plant Sciences*, 10, 63-121.

Siyahpoosh, A., Fathi, G., Zand, E., Siadat, S., Bakhshande, A., Gharineh, M.H. (2012). Competitiveness of different densities of two wheat cultivars with wild mustard weed species (Sinapis arvensis L.) in different densities. *World Applied Sciences Journal*, 20(5), 748-752.

Smyth, T., Ramachandran, V.N., Smyth, W.F. (2009). A study of antimicrobial activity of selected naturally occurring and synthetic coumarins. *International journal of antimicrobial agents*, 33(5), 421-426.

Soares, J.R.A.S. (1996). Constituição Polifenólica e Actividade Antioxidante de Extractos de *Thymus zygis*. Thes. Mast., Univ. de Coimbra, Coimbra, Portugal.

Song, M.C., Hong, Y.H., Kim, D.H., Kim, D.K., Chung, I.S., Lee, Y.H., Kim, S.H., Park, M. H., Kwon, B.M., Baek, N.I. (2003). Development of biologically active compounds from edible plant sources. Isolation of sterol compounds from aerial parts of Garland (*Chrysanthemum coronarium* L.). *Journal of the Korean Society of Agricultural Chemistry*, 46, 376-379.

Sorensen, T. (1948). A method of establishing groups of equal amplitude in plant sociology based on similarity of species content. *Det Kongelige Danske Videnskabernes Selskab. Biologiske Skrifter*, 5(4), 1-34.

Sriti Eljazi, J., Selmi, S., Zarroug, Y., Wesleti, I., Aouini, B., Jallouli, S., Limam, F. (2018). Essential oil composition, phenolic compound, and antioxidant potential of *Inula viscosa* as affected by extraction process. *International Journal of Food Properties*. 21(1), 2309-2319. https://doi.org/10.1080/10942912.2018.1517782

Stalikas, C.D. (2007). Extraction, separation and detection methods for phenolic acids and flavonoids. *Journal of Separation Science*, 30, 3268-3295.

Stalińska, K., Guzdek, A., Rokicki, M. and Koj, A. (2005). Transcription factors as targets of the anti-inflammatory treatment. A cell culture study with extracts from some Mediterranean diet plants. *Journal of Physiology and Pharmacology*. 56(1), 157-169.

Stöckigt, J., Sheludko, Y., Unger, M., Gerasimenko, I., Warzecha, H., Stöckigt, D. (2002). High-performance liquid chromatographic, capillary electrophoretic and capillary electrophoretic-electrospray ionization mass spectrometric analysis of selected alkaloid groups. *Journal of chromatography* A, 967, 85-113.

Sulas L., Re G.A., Molle G., Ligios S. (1999). *Chrysanthemum coronarium* L.: A new pasture species for Mediterranean forage systems. In: Etienne M. (ed.). Dynamics and sustainability of Mediterranean pastoral systems. Zaragoza: CIHEAM., *Cahiers Options Mediterraneennes*, 39, 83-86.

Sundaram, E.N., Singh, K.P., Reddy, K.P., Reddy, P.U., Nair, K.R.J., Raveendard, C.H., Nayak, C. (2010). A preliminary study to evaluate analgesic and behavioural activities of the homoeopathic drug, *Anagallis arvensis* in rats. *Indian Journal of Research in Homoeopathy*, 4(2), 41-44.

Suttle, N. (2010). Mineral Nutrition of Livestock. 4th Ed. Commonwealth Agricultural, Bureau International, Oxfordshire, UK.

Tabaraki, R., Ghadiri, F. (2013). *In vitro* antioxidant activities of aqueous and methanolic extracts of *Smyrnium cordifolium* Boiss. and *Sinapis arvensis* L. *International Food Research Journal*, 20(5), 2111-2115.

Tabaraki, R., Yosefi, Z., Asadi Gharneh, H.A. (2012). Chemical composition and antioxidant properties of *Malva sylvestris* L. *Journal of Research and Agricultural Science*. 8(114), 59–68.

Takaqi, N., Akamatsu, S.U. (1985). Effect of spring weed cover on 15 N uptake by citrus trees under orchard conditions. *Journal of Japanese Society for Horticultural Science*, 54, 307-140.

Taleb, A., Maillet, J. (1994). Mauvaises herbes des céréales de la Chaouia (Maroc). I. Aspect floristique. *Weed Research*, 34(5), 345-352. https://doi.org/10.1111/j.1365-3180.1994.tb02003.x

Tanji, A. (2001). Adventices de la fève non irriguée dans la province de Settat. *Al Awamia*, 103, 71-81.

Tanji, A., Elgharous, M. (1998). A survey of mineral composition of weeds seeds. *Weed Research*, 38, 79-86.

Tawaha, K., Alali, F.Q., Gharaibeh, M., Mohammad, M., El-Elimat, T. (2007). Antioxidant activity and total phenolic content of selected Jordanian plant species. *Food Chemistry*. 104(4), 1372-1378. https://doi.org/10.1016/j.foodchem.2007.01.064

Tefiani, C., Riazi, A., Belbachir, B., Lahmar, H., Aazza, S., Figueiredo, A. C., Miguel, M.G. (2016). *Ammoides pusilla (Brot.) Breistr.* from Algeria: Effect of harvesting place and plant part (leaves and flowers) on the essential oil's chemical composition and antioxidant activity. *Open Chemistry Journal*. 14(1), 343-350. https://doi.org/10.1515/chem-2016-0037

The Local Food-Nutraceuticals Consortium, (2005). Understanding local Mediterranean diets: A multidisciplinary pharmacological and ethnobotanical approach. *Pharmacological research*, 52(4), 353-366. https://doi.org/10.1016/j.phrs.2005.06.005

Thirumurugan, D., Cholarajan, A., Raja, S.S.S., Vijayakumar, R. (2018). An Introductory Chapter: Secondary Metabolites. Secondary Metabolites - Sources and Applications. https://10.5772/intechopen.79766

Tinthoin, R. (1948). Les aspects physiques du tell oranais. Ed. L. Fouque, Oran, 638pp.

Tomas-Barberan, F., Espin, J.C. (2001). Phenolic compounds and related enzymes as determinants of quality of fruits and vegetables. *Journal of the Science of Food and Agriculture*, 81, 853-876.

Torun, B., Biyik, H.H., Ercin, Z., Coban, E. (2018). Antifungal activities of *Urtica dioica* L., *Sinapis arvensis* L. and *Apium graveolens* Mill. leaves on *Botrytis cinerea* Pers. *Annals Phytomedicine,* 7(2), 94-97. https://doi.org/10.21276/ap.2018.7.2.15

Tóth, B., Kúsz, N., Forgo, P., Bózsity, N., Zupkó, I., Pinke, G., Hohmann, J., Vasas, A. (2017). Abietane diterpenoids from *Sideritis montana* L. and their antiproliferative activity. *Fitoterapia* 122, 90-94. https://doi.org/10.1016/j.fitote.2017.08.016

Touré, A., Rodenburg, J., Marnotte, P., Dieng, I., Huat, J. (2014). Identifying the problem weeds of rice-based systems along the inland-valley catena in the southern Guinea Savanna, Africa. *Weed Biology and Management*, 14(2), 121-132. https://doi.org/10.1111/wbm.12040

Tunçtürk, M., Tunçtürk, R., Eryiğit, T., Kaya, A.R. (2018). Mineral and Heavy Metal Constituents of Three Edible Wild Plants Growing in Van Province. *Journal of the Institute of Science and Technology*, 8(2), 293-298. https://doi.org/10.21597/jist.429008

Uddin, G., Rauf, A., Qaisar, M., Rehman, T.U., Latif, A., Ali, M. (2011). Preliminary phytochemical screening and antimicrobial activity of *Hedera Helix* L. *Middle East Journal of Scientific Research*. 8(1), 198-202.

Ullah, R., Ibrar, M., Shah, S., Hameed, I. (2012). Phytotoxic, cytotoxic, and insecticidal activities of *Calendula arvensis* L. *E3 Journal of Biotechnology and Pharmaceutical Research*, 3(6), 104-111.

University of Pittsburgh (2020). African Studies and African Country Resources. Retrieved from https://pitt.libguides.com/c.php?g=12378&p=65815

Usai, M. (2004). Natural dyes from plants: an ancient resource for modern purposes. *Chimica e l'Industria*, 86, 66-70.

Usman, K., Khalil, S.K., Khan, M.A. (2010). Impact of tillage and herbicides on weed density and some physiological traits of wheat under rice-wheat cropping system. *Sarhad Journal of Agriculture*, 26(4), 475-487.

Valente, M.E., Borreani, G., Caredda, S., Cavallarin, L., Sulas, L. (2003). Ensiling forage garland (*Chrysanthemum coronarium* L.) at two stages of maturity and at different wilting levels. *Animal Feed Science and Technology*, 108(1-4), 181-190. https://doi.org/10.1016/S0377-8401(03)00123-8

Van Soest, P.J. (1982). Nutritional Ecology of the Ruminant. Cornell University Press, 1994, 476 pp.

Venditti, A., Bianco, A., Frezza, C., Serafini, M., Giacomello, G., Giuliani, C., Bramucci, M., Quassinti, L., Lupidi, G., Lucarini, D., Papa, F., Maggi, F. (2016). Secondary metabolites, glandular trichomes and biological activity of *Sideritis montana* L. subsp. montana from Central Italy. *Chemistry and Biodiversity*, 13(10), 1380-1390. https://doi.org/10.1002/cbdv.201600082

Verdú, A.M., Mas, M.T. (2007). Mulching as an alternative technique for weed management in mandarin orchard tree rows. *Agronomy for Sustainable. Development.*, 27, 367-375. https://doi.org/10.1051/agro:2007028

Verpoorte, R. (1998). Exploration of Nature's Chemodiversity: The Role of Secondary Metabolites as Leads in Drug Development. *Drug Discovery Today*, 3(5), 232-8.

Verpoorte, R., Alfermann, A.W. (2000). Metabolic engineering of plant secondary metabolism. Ed: Kluwer Academic, 1- 23.

Vijayakumar, R., Arts, G. (2018). *An Introductory Chapter: Secondary Metabolites*. In *Open access peer-reviewed chapter.* https://doi.org/10.5772/intechopen.79766

Vincken, J.P., Heng, L., De Groot, A., Gruppen, H. (2007). Review Saponins, classification and occurrence in the plant kingdom. *Phytochemistry*, 68, 275-297.

Vukajlović, F., Pešić, S., Tanasković, S., Predojević, D., Gvozdenac, S., Prvulović, D., Bursić, V. (2019). Efficacy of *Echium* spp. water extracts as post-harvest grain protectants against *Plodia interpunctella*. *Romanian Biotechnological Lett*ers, 24(5), 761-69. https://doi.org/10.25083/rbl/24.5/761.769

Waller, G.R. (1989). Allelochemical action of some natural products. ln: Chou CH, Waller GR, eds. Phytochemical Ecology: Alleochemicals, Mycotoxins and lnsect Pheromones and Allomones. Academia Sinica Monograph Series, n° 9. Taipei: institute of Botany, 29-54 pp.

Wang B.J., Lien Y.H. Yu Z.R. (2004). Supercritical fluid extractive fractionation study of the antioxidant activities of propolis. *Food Chemistry*, 86, 237-243.

Ward, G.M. (1966). Potassium metabolism of domestic ruminants. A review. *Journal of Dairy Sci*ences, 49, 268-276.

Watt, J.M., Breyer-Brandwijk, M.G. (1962). The medicinal and poisonous plants of Southern and Eastern Africa. Second ed. Livingstone, Edinburgh.

Wills, R.B.H., Wong, A.W.K., Scriven, F.M., Greenfield, H. (1984). Nutrient Composition of Chinese Vegetables. *Journal of Agricultural and Food Chemistry*, 32(2), 413-416. https://doi.org/10.1021/jf00122a059

Wink, M. (2010). Biochemistry of Plant Secondary Metabolism, Annual Plant Reviews 40, Ed: Wiley-Blackwell, 1-23 pp.

Wu, T.S., Damu, A.G., Su, C.R., Kuo, P.C. (2004). Terpenoids of *Aristolochia* and their biological activities. *Natural Product Reports.* 21(5), 594-624. https://doi.org/10.1039/b401950d

Wyk, B. Van. (2011). The potential of South African plants in the development of new food and beverage products. *South African Journal of Botany*, 77(4), 857-868. https://doi.org/10.1016/j.sajb.2011.08.003

Zadeh, J.B., Kor, Z.M. (2013). Utilization and addition sugars to ruminants' rations. *International Journal of Advanced Biological and Biomedical Research*, 1(10), 1236-1245.

Zeggwagh, N.A., Ouahidi, M.L., Lemhadri, A., Eddouks, M. (2006). Study of hypoglycaemic and hypolidemic effects of *Inula viscosa* L. aqueous extract in normal and diabetic rats. *Journal of Ethnopharmacologyi*, 108(2), 223-227. https://doi.org/10.1016/j.jep.2006.05.005

Zenk, M.H., Jueng, M. (2007). Evolution and current status of the phytochemistry of nitrogenous compounds. *Phytochemistry*, 68, 2757-2772.

Zhang, S.Z., Li, Y.H., Kong, C.H., Xu, X.H. (2016). Interference of allelopathic wheat with different weeds. *Pest Management Science*, 72(1), 172-178. https://doi.org/10.1002/ps.3985

Zhao H., Dong J., Lu J., Chen J., Li Y. Shan L. (2006). Effect of extraction solvent mixtures on antioxidant activity evaluation and their extraction capacity and selectivity for free phenolic compounds in barley (*Hordeum vulgare* L.). *Journal of Agricultural and Food Chemistry*, 54(19), 7277-7286.

Zhou, H.C., Lin, Y.M., Wei, S.D., Tam, NF.Y. (2011). Structural diversity and antioxidant activity of condensed tannins fractionated from mangosteen pericarp. *Food Chemistry*, 129(4), 1710-1720.

Zidane, L., Salhi, S., Fadli, M., El Antri, M., Taleb, A., Douira, L. (2010). Étude des groupements d'adventices dans le Maroc occidental. *Biotechnology, Agronomy and Society and Environment*, 14(1), 153-166.

Zima, T., Fialova, L., Mestek, O., Janebova, M., Crkovska, J., Malbohan, I., Stipek, S., Mikulikova, L., Popov, P. (2001). Oxidative stress, metabolism of ethanol and alcohol related diseases. *Journal of Biomedical Science*, 8, 59-70.

Žugić, A., Dordević, S., Arsić, I., Marković, G., Živković, J., Jovanović, S., Tadić, V. (2014). Antioxidant activity and phenolic compounds in 10 selected herbs from Vrujci Spa, Serbia. *Industrial Crops and Products.* 52, 519-527. https://doi.org/10.1016/j.indcrop.2013.11.027

List of scientific communications

- **Oral communications in Conferences**

  Chemouri, S., Roca-Fernandez, A. I., Ainad Tabet, M., Miguel, M.G., Tefiani, C., Larid, M. (2019). Allelopathic effect of two weeds from Algeria (*Chrysanthemum coronariumn* L. and *Calendula arvensis* L.) on the germination and growth of barley (*Hordeum vulgare* L.), 1st Congress of Youth Researchers in Sustainable Animal Production and Safety and Quality Food, 27-30 November, Lugo, Galicia, Spain.

  Chemouri, S., Miguel, M.G., Tefiani, C., Roca-Fernandez, A. I., Ainad Tabet, M., Larid, M. (2019). Antioxidant activity, phenol, and flavonoid contents of 16 weed species from northwest of Algeria, Spain. 1st Congress of Youth Researchers in Sustainable Animal Production and Safety and Quality Food, 27-30 November, Lugo, Galicia, Spain.

  Chemouri, S., Ained Tabet, M. (2017). Biodiversity of citrus groves in the agricultural basin of Tlemcen (North-West Algeria). 4th International Congress of Plants Biodiversity, 5-8 October, Marrakech, Morocco.

- **Posters in Conferences**

  Chemouri, S., Belmir, M., Kazi Tani, Ch., Taibi, A., Larid, M. (2018). Ornithochory and inventory of apophytes at citrus groves in the agricultural basin of Tlemcen (Northwestern Algeria). The 6th Franco-Tunisian Congress of Zoology, The 6th Franco-Maghreb Congress of Zoology, The 119th Annual Days of the SZF, 21-23 November, Paris, France.

  Chemouri, S., Ained Tabet, M., Belmir, M., Tefiani, Ch., Larid, M. (2018). Impact of soil maintenance on the distribution of weeds in citrus groves in the Tlemcen region. National Seminar on the Future of Agriculture and Processing of Agricultural Products in Algeria, 30 April, Tlemcen, Algeria.

  Chemouri, S., Tefiani, Ch., Miguel, M.G., Ained Tabet, M., Taibi, A., Larid, M. (2018). Total phenolic content, flavonoids, and antioxidant activity of *Chrysanthemum coronarium* L. and *Calendula arvensis* L. from the Northwest of Algeria. 5th International Workshop MGIBR. Stakeholders in the equine sector: characteristics, critical points, and potential. April 27-28, 2019 conference hall, Palace of Culture, Imama, Tlemcen, Algeria.

  Chemouri, S., Kazi Tani, Ch. (2017). Ornithochory and the role of birds in the introduction of perennial apophytes in Tlemcen citrus orchards. 4th International Congress of Plants Biodiversity, 5-8 October, Marrakech, Morocco.

- **Conference papers**

  Chemouri, S., Ained Tabet, M., Tefiani, Ch., Larid, M. (2018). Inventory of weed species of citrus groves in the region of Tlemcen (Northwestern Algeria). International Scientific Agricultural Symposium, 04-07 October, Jahorina Mountain, Bosnia, and Herzegovina.

Annexes

Annex 1: Floristic list of the 88 weed species inventoried in the citrus orchards studied (Tlemcen, NW of Algeria).

Codes	Scientific nomenclatures	Botanical families	Areas of repartition	Ethological types
ALLNI	*Allium nigrum* L.	Liliaceae	Mediterranean	G.
AMARE	*Amaranthus retroflexus* L.	Amaranthaceae	Cosmopolitan	Th.
ANAAR	*Anagallis arvensis* L.	Primulaceae	Sub-cosmopolitan	Th.
APIGR	*Apium graveolens* L.	Apiaceae	North tropical	Th. /H2
ARIVU	*Arisarum vulgare* Targ. -Tozz.	Araceae	Mediterranean Circum	G.
ARIBA	*Aristolochia baetica* L.	Aristolochiaceae	Ibero-Moroccan	G.
ASPAC	*Asparagus acutifolius*	Liliaceae	Mediterranean	G.
ASPMI	*Asphodelus microcarpus* Salzm. and Viv.	Liliaceae	Mediterranean	G.
ASTPE	*Astragalus perigrinus* Vhal.	Fabaceae	Eastern Mediterranean	Th.
AVESA	*Avena sativa* L.	Poaceae	Temp.-subtropical	Th.
AVEST	*Avena sterilis* L.	Poaceae	Macar. - Mediterranean. -Irano. -Tour.	Th.
BETVU	*Beta vulgaris*	Chenopodiaceae	Eurasiatic - Mediterranean	Th. /H2
BRASY	*Brachypodium sylvaticum* (Huds.) P.B.	Poaceae	Paleotemp.	Th.
BROMA	*Bromus madritensis* L.	Poaceae	European - Mediterranean	Th.
BRORI	*Bromus rigidus* Roth	Poaceae	Paleo-sub-trop.	Th.
BRORU	*Bromus rubens* L.	Poaceae	Paleo-sub-trop.	Th.
CALAR	*Calendula arvensis* L.	Asteraceae	Sub- Mediterranean	Th.
CALSU	*Calendula suffruticosa* Vahl	Asteraceae	Esp. N. A.	Th.
CALVI	*Calycotome Intermédia* (Salzm.) Maire	Papilionaceae	Mediterranean	Nph.
CENSO	*Centaurea solstitialis* L.	Asteraceae	Med. AS.	Th.
CHAHU	*Chamaerops humilis* L.	Palmaceae	W. Mediterranean	Ch.
CHEAL	*Chenopodium album* L.	Chenopodiaceae	Cosmopolitan	Th.
CHESP	*Chenopodium* sp.	Chenopodiaceae	Mediterranean	Th.
CHRCO	*Chrysanthemum coronarium* L.	Asteraceae	Mediterranean	Th.
CHRSE	*Chrysanthemum segetum* L.	Asteraceae	Sub-cosmopolitan	Th.

Codes	**Scientific nomenclatures**	**Botanical families**	**Areas of repartition**	**Ethological types**
CONAL	*Convolvulus althaeoïdes* L.	Convolvulaceae	Macar. - Mediterranean	G. /H.
CONAR	*Convolvulus arvensis* L.	Convolvulaceae	Eurasiatic	G.
CORSC	*Coronilla scorpioides* Koch.	Fabaceae	Mediterranean	Th.
CYNDA	*Cynodon dactylon* (L.) Pers.	Poaceae	Thermocosm.	G.
CYPCO	*Cyperus conglomeratus* (Rottb.)	Cyperaceae	Trop.-Subtrop.	G.
CYPLO	*Cyperus longus* L.	Cyperaceae	Paleo. -Sub.-trop.	G.
CYPRO	*Cyperus rotundus* L.	Cyperaceae	Mediterranean	G.
DACGL	*Dactylis glomerata* L.	Poaceae	Paleo-Temp.	H.
DAUCA	*Daucus carota* L.	Apiaceae	Eur.- Mediterranean	Th. /H2
ECHVU	*Echium vulgare* L.	Boraginaceae	Mediterranean	H.
EMESP	*Emex spinosa* (L.) Campb.	Polygonaceae	Mediterranean	Th.
EROMO	*Erodium moschatum* (Burm.) l'Her.	Geraniaceae	Mediterranean	Th. /H.
ERYCO	*Eryngium compestre* L.	Apiaceae	European - Mediterranean	H. /G.
FERCO	*Ferula communis* L.	Apiacae	Mediterranean	H.
FICCA	*Ficus carica* L.	Moraceae	Mediterranean	Ph.
FOEVU	*Fœniculum vulgare* (Miller) Gaertner	Apiaceae	Mediterranean	Th. / H.
FUMCA	*Fumaria capreolata* L.	Fumariaceae	Mediterranean	Th.
GLASE	*Gladiolus segetum* Ker. -Gawl.	Iridaceae	Mediterranean	G.
GLYFL	*Glyceria fluitans* (L.) R. Br.	Poaceae	Sub-cosmopolitan	H.
GLYMA	*Glyceria maxima* (Hartm.) Holmb.	Poaceae	European. -As.-N. Am.	H.
HEDHE	*Hedera helix* L.	Araliaceae	European. - Mediterranean	NPh.
HEDNA	*Hedysarum naudinianum* Coss.	Fabaceae	End.	Th.
HELNO	*Helosciaduim nodiflorum* Lag.	Apiaceae	Atl. Mediterranean	Th.
HORMU	*Hordeum murinum* L.	Poaceae	Circumbor.	Th.
INUCR	*Inula crithmoides* L.	Asteraceae	Atl.- Mediterranean	Th.
INUMO	*Inula montana*	Asteraceae	W. Mediterranean. - Sub.-Atl.	Th.

Codes	Scientific nomenclatures	Botanical families	Areas of repartition	Ethological types
INUVI	*Inula viscosa* (L.) Ait.	Asteraceae	Circummed.	Th. / Ch.
KOEPH	*Koeleria phleoides* Pers.	Poaceae	Mediterranean. -Ir.-Tour.	Th.
LAMAU	*Lamarckia aurea* (L.) Moench.	Poaceae	Mediterranean. -Ir.-Tour.	Th.
MALSY	*Malava sylvetris* L.	Malvaceae	Mediterranean	Th. /H.
MALPA	*Malva parviflora* L.	Malvaceae	Mediterranean	Th.
MEDRU	*Medicago rugosa* Desr.	Fabaceae	Eastern Mediterranean	Th.
OLEEU	*Olea europea* L.	Oleaceae	Mediterranean	Ph.
OPHFU	*Ophrys fusca* Link.	Orchideae	Mediterranean	G.
ORCPU	*Orchis italica* Poiret.	Orchideae	Eurasiatic	G.
ORNUM	*Ornithogalum umbellatum* L.	Liliaceae	Atl.- Mediterranean	G.
ORYCA	*Oryzopsis caerulescens*	Poaceae	Mediterranean	H.
ORYMI	*Oryzopsis miliacea* (L.) Asch. et Schw.	Poaceae	Mediterranean. -Ir.-Tour.	H.
ORYPA	*Oryzopsis paradoxa* (L.) Nutt.	Poaceae	W. Mediterranean	H.
OXAPE	*Oxalis pescapreae* L.	Oxalidaceae	South African	G.
PALSP	*Palllenis spinosa* (L.) Cass.	Asteraceae	European Mediterranean	Th. / H2
PANMI	*Panicum miliaceum* L.	Poacaeae	Mediterranean	Th.
PICEC	*Picris echioides* L.	Asteraceae	Mediterranean. -Ir.-Tour.	Th. /H.
PISAT	*Pistacia atlantica* Desf.	Anacardiaceae	Endemic North African	Ph.
PISLE	*Pistacia lentiscus* L.	Anacardiaceae	Mediterranean	Ph.
POROL	*Portulaca oleracea* L.	Portulacaceae	Cosmopolitan	Th.
REITI	*Reichardia tingetana* L. Roth.	Asteraceae	Mediterranean	Th. /H.
RUBPE	*Rubia peregrina* L.	Rubiaceae	Atlantic-Mediterranean	H.
RUMCO	*Rumex conglomeratus* Murr.	Polygonaceae	Cosmopolitan	Th. / H.
SCOHI	*Scolymus hispanicus* L.	Asteraceae	Mediterranean	Th. / H2
SCOMU	*Scorpiurus muricatus* L.	Fabaceae	Mediterranean	Th.
SETVE	*Setaria verticillata* (L.) P.B.	Poaceae	Thermo-cosmopolite	Th.
SETVI	*Setaria viridis* (L.) P. B.	Poaceae	Temp.-Subtrop.	Th.

Codes	Scientific nomenclatures	Botanical families	Areas of repartition	Ethological types
SIDMO	*Sideritis montana* L.	Labiateae	Mediterranean	Th.
SINAR	*Sinapis arvensis* L.	Brassicaceae	Paleo-Temp.	Th.
SMYOL	*Smyrnium olusatrum* L.	Apiaceae	Mediterranean	H.
SOLNI	*Solanum nigrum* L.	Solanaceae	Cosmopolitan	Th.
SONAS	*Sonchus asper* (L.) Vill.	Asteraceae	Cosmopolitan	Th.
STAOM	*Stachys ocymastrum* (L.) Briq.	Labiateae	Mediterranean	Th.
TAROF	*Taraxacum officinalis* F.H. Wigg.	Asteraceae	European. -Sib.- Mediterranean.	H.
THAGA	*Thapsia garganica* L.	Apiaceae	Mediterranean	H.
TORNO	*Torilis nodosa* Gaertn.	Apiaceae	Eurasiatic	Th.
WITFR	*Withania frutescens* Panquy	Solanaceae	Ibero-Mar.	Ch.

Legend: Th.: Therophyte, Ch.: Chamephyte, H.: Hemicryptophyte, G.: Geophyte, Ph.: Phanerophyte, NPh.: Nanophanerophyte, H2.: Biannual, End.: Endemic, N.Af.: North African, S.Af.: South African, Ibero-Maur.: Ibero-Mauritanian, Ir.-Tour.: Irano-Torranian, Am. : American, As.: Asiatic, Atl.: Atlantic, Cosm. : Cosmopolite, Mar.: Moroccan, Macar. : Macaronesian, Sib.: Siberian, Med. : Mediterranean, Circummed.: Circummediterranean W.-Med.: West-Mediterranean, Circumbor.: Circumboreal, Eur.: European, Euras.: Eurasiatic, Paleo-Temp.: Paleo-Temperate, Paleo-Trop.: Paleo-Tropical, Subcosm.: Subcosmopolite, Thermocosm.: Thermocosmopolite, Trop.: Tropical, Subtrop.: Subtropical.

Abstract: The present thesis deals with the impact of mechanical and chemical weeding on the composition of weed flora in citrus orchards as well as the valorization of 20 % of this flora. The first part of this study was grounded on 168 surveys before and after each weeding (mechanical and chemical) in comparison to a control without weeding. A total of 88 species, belonging to 71 genera and 30 botanical families were identified. The Mediterranean elements (64%), therophytes (51%) and dicotyledons (64%) were dominant. the ANOVA revealed the existence of a significant spatial-temporal difference in the mean species richness between the stations (control *vs.* chemical and mechanical). In addition, the species richness before weeding tended to be higher than that after it. The second part was interested in the evaluation of the antioxidant activities (DPPH, ABTS and chelating power) and the phenols and flavonoids content of 19 weed species (representing 20 % of the total flora) that aimed at valorizing citrus weeds. Four species (*I. viscosa* L., *S. montana* L., *R. peregrina* L. and *R. tingitana* L.) were characterized by their richness in phenols and flavonoids and as good DPPH scavengers. Principal component analysis permitted to find two main groups: that in which *I. viscosa* L., *R. tingitana* L. and *S. montana* L. possessed relative high amounts of total phenols and good capacity for scavenging ABTS free radicals, whereas *M. sylvestris* L., *E. spinosa* (L.) Campd., *H. helix* L. and *R. peregrina* L. were good scavengers of DPPH free radicals and chelators of ferrous ions. This can be the first step for possible mixtures of these extracts in order to evaluate the possible synergistic effect among them, acting simultaneously as free radical scavengers and chelating agents. The third part treated the evaluation of the nutritive value and the mineral composition of 19 weeds which aimed to valorize these species to be used as fodder for animal nutrition. The results showed that *O. pes-caprae* was the species exhibiting the best NDF-Digestibility. As for the mineral composition, *Ch. coronarium* showed the highest K and Ca levels while *E. spinosa* presented the higher Mg and Zn concentrations. For Fe, Ch. segetum was the richest species. Also, *I. viscosa* had the best Mn content and *H. helix* recorded the higher P level. These results suppose the potential use of weeds in pharmaceutical, industrial, and cosmetic industries but also for animal feeding.

Keywords: Weeds, weeding techniques, valorization, antioxidant activity, nutritive value.

Résumé : La présente thèse traite l'impact du désherbage mécanique et chimique sur la composition de la flore adventice dans les vergers d'agrumes ainsi que la valorisation de 20% de cette flore. La première partie de cette étude porte sur 168 relevés floristiques réalisés avant et après chaque mode de désherbage en comparaison avec un témoin sans désherbage. Au total, 88 espèces appartenant à 71 genres et 30 familles botaniques ont été recensés et identifiés. Les éléments méditerranéens (64%), les thérophytes (51%) et les dicotylédones (64%) prédominent. L'ANOVA a révélé l'existence d'une différence spatio-temporelle significative dans la richesse moyenne en espèces entre les stations (témoin *vs.* chimique et mécanique). De plus, la richesse en espèces a tendance à être plus élevée avant qu'après le désherbage. La seconde porte sur l'évaluation des activités antioxydantes (DPPH, ABTS et le pouvoir chélatant) et à la teneur en phénols et flavonoïdes de 19 espèces d'adventices (représentant 20% de la flore totale) visant à valoriser les adventices d'agrumes. Quatre espèces (*I. viscosa* L., *S. montana* L., *R. peregrina* L. et *R. tingitana* L.) ont été caractérisées par leur richesse en phénols et flavonoïdes et comme de bons piégeurs de DPPH. L'analyse des composants principaux a permis de trouver deux groupes principaux : celui dans lequel *I. viscosa*, *R. tingitana* et *S. montana* possédant des quantités relativement élevées de phénols totaux et une bonne capacité à piéger les radicaux libres ABTS, tandis que *M. sylvestris* L., *E. spinosa* (L.) Campd., *H. helix* L. et *R. peregrina* se sont révélés de bons piégeurs de radicaux libres DPPH et chélateurs d'ions ferreux. Cela peut être la première étape pour d'éventuels mélanges de ces extraits afin d'évaluer le possible effet synergique entre eux, agissant à la fois comme piégeurs de radicaux libres et agents chélateurs. La troisième partie a traité l'évaluation de la valeur nutritive et de la composition minérale de 19 adventices qui visait à valoriser ces espèces pour être utilisées comme fourrage dans la nutrition animale. Les résultats ont montré que *O. pes-caprae* L. est l'espèce qui représente la meilleure digestibilité des NDF. Quant à la composition minérale, *Ch. coronarium* a montré les niveaux les plus élevés de K et du Ca tandis que *E. spinosa* a présenté des concentrations plus élevées de Mg et de Zn. Pour Fe, *Ch. Segetum* L. s'est avérée l'espèce la plus riche. En outre, *I. viscosa* possède la meilleure teneur en Mn et *H. helix* a enregistré le niveau de P le plus élevé. Ces résultats supposent une utilisation potentielle des adventices dans les domaines pharmaceutiques et biotechnologiques, la phytoremédiation et aussi dans l'alimentation animale, etc.

Mots-clés : Adventices, techniques de désherbage, valorisation, activité antioxydante, valeur nutritive.

ملخص : تتناول الأطروحة الحالية تأثير إزالة الأعشاب الضارة الميكانيكية والكيميائية على تكوين نباتات الحشائش في بساتين الحمضيات بالإضافة إلى تثمين 20٪ من هذه النباتات. يتعلق الجزء الأول من هذه الدراسة بـ 168 مسحا نباتيا تم إجراؤها قبل وبعد كل طريقة لإزالة الأعشاب الضارة مقارنة بشاهد بدون إزالة الأعشاب الضارة. في المجموع ، تم تحديد 88 نوعًا تنتمي إلى 71 جنسًا و 30 عائلة نباتية. تسود عناصر البحر الأبيض المتوسط (64٪) والنباتات الحرارية (51٪) وثنائية الفلقة (64٪) في بستان الحمضيات. كشف التحليل الاحصاءي باستعمال تحليل التباين (ANOVA) عن وجود اختلاف مكاني وزماني كبير في متوسط ثراء الأنواع بين المحطات (الشاهد مقابل الكيميائية والميكانيكية). بالإضافة إلى ذلك ، فإن ثراء الأنواع يميل إلى أن يكون أعلى من قبل إزالة الأعشاب الضارة. يتعلق الجزء الثاني بتقييم الأنشطة المضادة للأكسدة (DPPH و ABTS والقوة المخلبة) ومحتوى الفينولات والفلافونويد في 19 نوعًا من الحشائش (تمثل 20٪ من إجمالي النباتات) التي تهدف إلى تعزيز أعشاب الحمضيات. . أربعة أنواع (*R. peregrina* L. and *R. tingitana* L., *S. montana* L., *I. viscosa* L.) تميزت بغناها بالفينولات والفلافونويدات وباعتبارها كاسحات جيدة في إزالة الجذور الحرة لـ DPPH. سمح تحليل المكونات الرئيسية بالعثور على مجموعتين رئيسيتين: تلك التي تمتلك فيها كميات عالية نسبيًا من إجمالي الفينولات وقدرة جيدة على تنظيف الجذور الحرة ABTS (*I.viscosa*, *R. tingitana*, *S. montana*) بينما قد ثبت أن (*M. sylvestris* L., *E. spinosa* (L.) Campd., *H. helix* L., *R. peregrina*) كانوا كاسحات جيدة للجذور الحرة DPPH ومخلبات الأيونات الحديدية. يمكن أن تكون هذه هي الخطوة الأولى للخلطات المحتملة من هذه المقتطفات من أجل تقييم التأثير التآزري المحتمل بينها ، حيث تعمل ككاسحات للجذور الحرة وعوامل مخلبية. وتناول الجزء الثالث تقييم القيمة الغذائية والتركيب المعدني لـ 19 حشائشاً ، والتي تهدف إلى تعزيز هذه الأنواع لاستخدامها كعلف في تغذية الحيوانات. أظهرت النتائج أن *O. pes-caprae* L. هو النوع الذي يمثل أفضل قابلية هضم لـ NDF. أما بالنسبة للتركيب المعدني ، فقد أظهرت *Ch. coronarium* أعلى مستويات K و Ca بينما أظهرت *E. spinosa* تركيزات أعلى من Mg و Zn. بالنسبة لـ Fe ، تبين أن *Ch. segetum* L. كانت أغنى الأنواع. بالإضافة إلى ذلك ، كان لدى *I. viscosa* أفضل محتوى من Mn وسجلت *H. helix* أعلى مستوى P. تفترض هذه النتائج استخدامًا محتملاً للأعشاب الضارة في مجالات الأدوية والتكنولوجيا الحيوية ، والمعالجة النباتية وأيضًا في علف الحيوانات ، إلخ.

الكلمات المفتاحية: الحشائش الضارة ، تقنيات التطهير ، التثمين ، النشاط المضاد للأكسدة ، القيمة الغذائية.