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Application of a New Binder in the Treatment of Liquid Effluents

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DEDICATION

I dedicate this thesis to myself, for the perseverance, effort, and resilience I've shown throughout this journey.

To my parents, for their unconditional love, constant support, and the sacrifices they've made to help me succeed.

And to my project partner, for being part of this academic experience.

BELOUATEK Marouane

DEDICATION

First and foremost, I thank Allah (SWT), the Most Merciful, for granting me the strength, patience, and guidance to complete this journey. Without His blessings, none of this would have been possible.

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Abstract

This thesis aims to evaluate the effectiveness of ceramic membranes made from local materials in the treatment of liquid effluents. The work is divided into four chapters. The first chapter discusses the characteristics of industrial effluents, their impact on the environment and human health, and existing treatment methods. The second chapter focuses on membrane technology, with particular emphasis on ceramic membranes: their operating principles, manufacturing process, physicochemical properties, and advantages over polymeric membranes. The third chapter presents the materials used, the experimental methods, the crossflow filtration system, and the analyses performed (pH, conductivity, TDS, COD, UV-visible spectrophotometry). The fourth chapter presents and discusses the experimental results obtained from the treatment of dyes (malachite green, methylene blue), heavy metals (lead nitrate, cadmium nitrate), and real wastewater from the Tayal textile factory. The tested membranes showed good retention capacity and a significant reduction in conductivity, TDS, and COD, confirming their effectiveness in effluent decontamination. This study demonstrates the potential of local ceramic membranes as a sustainable, economical, and efficient solution for treating industrial wastewater.

Résumé

L'objectif de ce mémoire est d'évaluer l'efficacité de membranes céramiques élaborées à partir de matériaux locaux dans le traitement des effluents liquides. Le travail est divisé en quatre chapitres. Le premier aborde les caractéristiques des effluents industriels, leurs impacts sur l'environnement et la santé humaine, ainsi que les différentes méthodes de traitement existantes. Le deuxième chapitre est dédié à la technologie des membranes, avec un accent particulier sur les membranes céramiques : leurs principes de fonctionnement, leur fabrication, leurs propriétés physico-chimiques et leurs avantages par rapport aux membranes polymériques. Le troisième chapitre décrit les matériaux utilisés, les méthodes expérimentales, le dispositif de filtration en flux tangentiel, et les analyses effectuées (pH, conductivité, TDS, DCO, spectrophotométrie UV-visible). Le quatrième chapitre présente et discute les résultats expérimentaux obtenus lors du traitement de colorants (vert malachite, bleu de méthylène), de métaux lourds (nitrate de plomb, nitrate de cadmium), ainsi que des eaux usées réelles de l'usine textile Tayal. Les membranes testées ont montré une bonne capacité de rétention, une réduction significative de la conductivité, du TDS, et de la DCO, confirmant leur efficacité pour la décontamination des effluents. Ce travail démontre le potentiel des membranes céramiques locales comme solution durable, économique et efficace pour le traitement des eaux industrielles polluées.

ملخص

يهدف هذا البحث إلى تقييم فعالية الأغشية السيراميكية المصنوعة من مواد محلية في معالجة المياه العادمة. ينقسم العمل إلى أربعة فصول. يتناول الفصل الأول خصائص المياه الصناعية المستعملة، وتأثيرها على البيئة وصحة الإنسان، وطرق المعالجة التقليدية. يركز الفصل الثاني على تقنية الأغشية، وبشكل خاص الأغشية السيراميكية من حيث مبدأ عملها، طرق تصنيعها، خصائصها الفيزيائية والكيميائية، ومميزاتها مقارنةً بالأغشية البوليميرية. يعرض الفصل الثالث المواد المستخدمة، الطرق التجريبية، نظام الترشيح العرضي، والتحليل المنجز (الرقم الهيدروجيني، التوصيلية، المواد الصلبة الذائبة الكلية، الطلب الكيميائي للأوكسجين، والتحليل الطيفي بالأشعة فوق البنفسجية والمرئية). أما الفصل الرابع فيستعرض ويناقش النتائج التجريبية لمعالجة الأصباغ (المالakit الأخضر، الأزرق الميثيليني)، والمعادن الثقيلة (نترات الرصاص، نترات الكاديوم)، ومياه الصرف الحقيقية من مصنع تايل للنسيج. أظهرت الأغشية المختبرة قدرة جيدة على الاحتفاظ بالملوثات وتقليل التوصيلية مما يؤكد فعاليتها في إزالة التلوث. يبرز هذا العمل إمكانيات الأغشية السيراميكية المحلية كحل مستدام، COD والـ TDS والـ واقتصادي وفعال لمعالجة مياه الصرف الصناعية.

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GENERAL INTRODUCTION

Water is a vital resource for life, human health, and sustainable development. However, with the rapid growth of population, industry, and urbanization, water quality is increasingly threatened by pollution. One of the most significant contributors to this problem is the direct or poorly treated discharge of wastewater into the environment. These discharges often contaminate the surface and groundwater, disrupt ecosystems, and pose serious risks to both human health and biodiversity [1].

Among the various industrial sectors, the textile industry is one of the most water-intensive and polluting. Processes such as dyeing, printing, and finishing release large volumes of wastewater loaded with synthetic dyes, inorganic salts, and sometimes heavy metals. These pollutants are often resistant to conventional biological treatment methods, making their removal a complex challenge. In addition, industrial effluents like those from the Tayal textile factory are known to contain a mixture of colorants, chemicals, and dissolved solids that require advanced treatment techniques.

To address these challenges, researchers have explored a wide range of treatment methods, including chemical precipitation, adsorption, coagulation–flocculation, and oxidation processes. More recently, membrane filtration has emerged as a promising solution due to its efficiency, simplicity, and ability to treat highly contaminated effluents without the need for chemical additives. Among membrane types, ceramic membranes stand out for their exceptional resistance to heat, chemicals, and mechanical stress, along with a longer operational lifespan [2].

Despite their advantages, commercial ceramic membranes remain expensive. This has led to increased interest in developing alternative membranes from natural materials such as local Algerian clays, which are abundant, low-cost, and environmentally friendly. These clay-based membranes show great potential for treating industrial effluents in a more sustainable and economical way [3].

In this study, we evaluate the performance of locally fabricated ceramic membranes in treating three types of contaminants:

- Synthetic dyes (Malachite Green and Methylene Blue),
- Heavy metals (Lead and Cadmium), and
- Real industrial wastewater from the Tayal textile factory.

This thesis is structured into three main parts:

- **The first part** provides a general overview of wastewater, its sources, classifications, and traditional treatment approaches.
- **The second part** focuses on membrane technologies, particularly ceramic membranes: their properties, fabrication methods, and separation mechanisms.
- **The third part** presents the experimental results of treating dyes, metal-contaminated solutions, and real Tayal wastewater using the prepared ceramic membranes, with performance analyzed based on retention rate, pH, conductivity, total dissolved solids (TDS), and chemical oxygen demand (COD).

EFFLUENTS

INTRODUCTION

Effluents, or wastewater discharges, represent a critical interface between human activities and environmental health. They are the liquid byproducts of industrial, agricultural, and urban processes, carrying a complex mixture of contaminants into aquatic ecosystems. Among these, industrial wastewater stands out as a major concern. It is the varied effluent from factories and facilities, released after being used in production, cleaning, cooling, and other industrial activities. Even rainwater can become part of the problem when it washes over contaminated surfaces, picking up pollutants like hydrocarbons, heavy metals, or nitrogenous compounds from factory roofs or abandoned industrial sites.

The discharge of untreated or inadequately treated effluents, whether from cities, farms, or factories, poses a significant threat to rivers, lakes, and coastal waters. These waters, which should sustain life, often end up saturated with pollutants, organic waste, toxic chemicals, or metallic salts, each posing distinct environmental risks. The nature of industrial wastewater alone can vary wildly depending on its source, making the challenge of treatment and prevention even more complex. Understanding where these effluents come from, what they carry, and how they harm ecosystems is not just scientific work — it's a necessary step toward protecting water, preserving biodiversity, and ensuring that communities and industries can thrive without sacrificing the health of our planet.

DEFINITION:

Water pollution refers to any detrimental modification in the chemical, physical, or biological properties of water by human influence, either directly or indirectly [4]. These changes disadvantage the quality of water and make it unsuitable for its proposed use, for instance, drinking, agriculture, or aquatic life [5,25].

Wastewater comprises all water that is contaminated in domestic, agricultural, and industrial processes and transports toxic substances such as chemicals, heavy metals, and

organic contaminants into sewerage systems [6]. Rainwater that becomes contaminated when it flows over urban and industrial surfaces, collecting contaminants such as oils, pesticides, and litter, also comes under this category [7].

When discharged into rivers, lakes or the ocean, wastewater and contaminants thereof lead to:

- Disturbances to ecosystems [8]
- Loss of biodiversity [9]
- Risk to human health due to exposure to pathogens or harmful chemicals [10]

To prevent such adverse effects, wastewater treatment and proper handling are essential [11,25].

Holistic solutions require:

- Advanced wastewater treatment technologies [12]
- Stricter regulations [13]
- Public awareness of water pollution consequences [14]

1. Effluent Classifications:

Effluents are classified based on various factors, such as their origin, nature of pollutants, and physical and chemical characteristics. Proper classification is essential for determining appropriate treatment methods and assessing their potential environmental and health impacts.

1.1. Industrial Effluents:

Industrial effluents are typically characterized by high levels of toxic chemicals, heavy metals, and organic compounds. These effluents are generated from manufacturing processes, power plants, and other industrial activities. Common pollutants in industrial effluents include:

Heavy metals such as lead (Pb), mercury (Hg), cadmium (Cd), and chromium (Cr) [15]

Organic chemicals including solvents, oils, and plastics [15]

Acidic and basic compounds depending on the nature of industrial processes [15]

The treatment of industrial effluents requires specific methods such as chemical precipitation, filtration, and adsorption to remove contaminants effectively [16].

1.2. Agricultural Effluents:

Agricultural effluents are produced by runoff from farmland and include nutrients like nitrogen and phosphorus, pesticides, and herbicides. These effluents can cause eutrophication in water bodies, leading to excessive algal growth and depletion of oxygen levels. Key pollutants in agricultural effluents are:

Nitrates and phosphates from fertilizers [17]

Pesticides and herbicides used in crop protection [17]

Sediments from soil erosion [17]

Agricultural runoff is often managed through methods such as buffer zones, constructed wetlands, and nutrient management practices to reduce its environmental impact [18].

1.3. Domestic Effluents:

Domestic effluents consist mainly of wastewater from residential areas, including water from kitchens, toilets, and laundry. These effluents contain organic matter, nutrients, and microorganisms, including pathogens. Key components include:

Organic matter such as food waste and detergents [19]

Human waste, which contains bacteria, viruses, and nutrients like nitrogen and phosphorus [19]

Chemical residues from cleaning agents and pharmaceuticals [19]

The treatment of domestic effluents generally involves primary, secondary, and tertiary treatments, with methods like biological filtration, chemical disinfection, and nutrient removal [20].

1.4. Hospital and Pharmaceutical Effluents:

Hospital and pharmaceutical effluents are unique due to the presence of pharmaceutical residues, antibiotics, and personal care products. These effluents can be hazardous to aquatic life and human health if not properly treated. Common contaminants include:

Pharmaceutical compounds such as antibiotics, hormones, and painkillers [21]

Disinfectants and antiseptics used in medical settings [21]

Pathogenic microorganisms from hospital waste [21]

Due to the complexity of these effluents, advanced treatment methods like ozonation and activated carbon filtration are often employed [22].

2. Physico-Chemical and Biological Characteristics of Effluents:

Effluents are characterized by a variety of physico-chemical and biological parameters that influence their treatment and environmental impact.

2.1. Physico-Chemical Parameters:

2.1.1. pH:

The pH of an effluent determines its acidity or alkalinity. It can affect the solubility of heavy metals and other pollutants in water, thereby influencing their toxicity to aquatic organisms. Acidic effluents can corrode infrastructure, while alkaline effluents can disrupt biological treatment processes [23].

2.1.2. Chemical Oxygen Demand (COD):

COD measures the amount of oxygen required to chemically oxidize organic matter in effluents. It is a key indicator of the organic load and is used to assess the pollution level of the effluent [24].

2.1.3. Biological Oxygen Demand (BOD):

BOD indicates the amount of oxygen consumed by microorganisms to biodegrade organic material in the effluent over a period of five days (BOD₅). It is an important parameter for assessing the biological degradability of wastewater [19].

2.1.4. Heavy Metals (Pb, Hg, Cd, Cr, Zn):

Heavy metals, such as lead, mercury, and cadmium, are highly toxic and can bioaccumulate in aquatic organisms. These metals are commonly found in industrial effluents and require specialized treatment methods, such as ion exchange or membrane filtration, for removal [20].

2.1.5. Suspended Solids (SS):

Suspended solids are solid particles suspended in the effluent. These solids can clog pipes and obstruct light penetration in water bodies, affecting photosynthesis in aquatic plants [19].

2.2. Biological Contaminants:

Effluents can contain a variety of biological contaminants, including bacteria, viruses, and parasites, which pose health risks.

2.2.1. Pathogenic Bacteria:

Pathogenic bacteria, such as *Salmonella* and *Escherichia coli*, are common in effluents from domestic, industrial, and hospital sources. These microorganisms can cause waterborne diseases, particularly if the effluent is not properly treated before discharge [23].

2.2.2. Viruses:

Viruses, including those causing hepatitis A and rotavirus, can also be present in effluents. They are often resistant to conventional treatment methods, requiring advanced disinfection techniques like UV treatment or ozonation [22].

2.2.3. Parasites:

Parasites such as Giardia and Cryptosporidium can be present in effluents, particularly those from hospitals and domestic wastewater. These parasites are resistant to chlorination and require specific treatment methods for removal [18].

3. Effects of Effluents on the Environment and Human Health:

Effluents have a significant impact on the environment and human health, affecting aquatic ecosystems, soils, and air quality.

3.1. Effects on Water Bodies:**3.1.1. Eutrophication:**

Eutrophication occurs when excessive nutrients, particularly nitrates and phosphates, accumulate in water bodies. This leads to algal blooms, which deplete oxygen levels and disrupt aquatic ecosystems [27].

3.1.2. Heavy Metal Contamination:

Heavy metals from industrial effluents can accumulate in aquatic organisms, affecting biodiversity and posing a risk to human health through the food chain [22].

3.1.3. Oxygen Depletion:

Effluents containing organic material consume oxygen as they decompose, leading to oxygen depletion in receiving waters. This results in hypoxic conditions, harming aquatic life [20].

3.2. Effects on Soil:

3.2.1. Acidification:

Acidic industrial effluents can lead to soil acidification, reducing soil fertility and affecting plant growth [28].

3.2.2. Toxic Accumulation:

Effluents containing toxic chemicals, such as pesticides and heavy metals, can accumulate in the soil, contaminating agricultural land and affecting crop production [27].

3.3. Effects on Air Quality:

3.3.1. Toxic Gas Emissions:

Some effluents, particularly from industrial and agricultural sources, release toxic gases such as ammonia and hydrogen sulfide into the air, posing risks to human and animal health [27].

3.3.2. Greenhouse Gas Emissions:

Effluents, especially from landfills and wastewater treatment plants, emit greenhouse gases such as methane and carbon dioxide, contributing to climate change [28].

3.4. Effects on Human Health:

3.4.1. Waterborne Diseases:

Effluents containing pathogenic microorganisms can cause diseases such as cholera, dysentery, and hepatitis A, especially if the effluent is untreated and used for drinking or recreational purposes [21].

3.4.2. Chemical Poisoning:

Heavy metals and toxic chemicals in effluents can lead to poisoning, cancer, and other serious health issues in humans exposed to contaminated water or air [19,25].

4. Effluent Treatment Methods:

Effluent treatment involves physical, chemical, and biological processes to reduce pollutants and make the water safe for reuse or discharge.

4.1. Physical Treatment:

Physical treatment processes include settling and filtration to remove suspended solids and particles from the effluent.

4.2. Chemical Treatment:

Chemical treatments, such as coagulation-flocculation and advanced oxidation, are used to remove dissolved pollutants and toxins.

4.3. Biological Treatment:

Biological treatments, using microorganisms to degrade organic matter, include methods like activated sludge and constructed wetlands.

5. Regulations and Effluent Discharge Standards:

Effluent management is strictly regulated by environmental standards to protect the environment, public health, and ensure the sustainability of natural resources. These regulations are implemented by national and international authorities, such as the World Health Organization (WHO) and the European Union (EU).

5.1. Effluent Discharge Standards:

Industrial and domestic effluents must comply with specific thresholds for water quality parameters, such as Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), heavy metals, nutrients (nitrates, phosphates), and pathogens. These standards are established to minimize the risk of water pollution in receiving bodies [26].

5.2. International and National Legislation:

Legislation varies by region and country. For example, in Europe, the EU Water Framework Directive (2000/60/EC) imposes strict requirements for surface water quality and effluent management methods. In Algeria, the 2005 Water Law and associated regulations set limits for industrial and agricultural effluents [27].

5.3. Discharge Permits and Environmental Controls:

Companies and industries responsible for effluent production must obtain discharge permits from the competent authorities. These permits define the quantities and types of pollutants allowed to be discharged into water or soil. Regular monitoring is conducted to ensure compliance with the established limits [28].

6. Towards Sustainable Effluent Management:

Sustainable effluent management involves adopting strategies aimed at minimizing pollutant production, maximizing resource reuse, and protecting aquatic and terrestrial ecosystems.

6.1. Source Reduction:

Reducing effluent production at the source is a fundamental approach to sustainable management. This includes cleaner production technologies, the use of less polluting chemicals, and the optimization of industrial processes to minimize effluent generation [29].

6.2. Recycling and Reuse of Effluents:

A key strategy in sustainable effluent management is the reuse of treated wastewater in agriculture, industry, and for non-potable uses. This reduces the demand for freshwater resources and limits the environmental impact of effluent discharge [30].

6.3. Advanced Treatment and Innovative Technologies:

The application of advanced technologies, such as reverse osmosis, ultrafiltration, and advanced oxidation, allows for effective effluent treatment and recovery of valuable resources they contain. These technologies not only eliminate pollutants that are difficult to degrade but also recover energy and reusable materials, contributing to a circular economy [31].

6.4. Awareness and Governance:

Sustainable effluent management also requires raising awareness among stakeholders, businesses, and consumers about the importance of reducing their environmental footprint.

Governments and international organizations play a key role in encouraging the implementation of policies and incentive strategies for businesses and communities [32].

MEMBRANE TREATMENT

INTRODUCTION

Ceramic membrane technology has emerged as a pivotal advancement in the field of filtration and separation processes, offering a robust alternative to traditional polymeric membranes. Composed of inorganic materials such as alumina, zirconia, and silicon carbide, ceramic membranes are renowned for their exceptional thermal and chemical stability, mechanical strength, and longevity [33]. These attributes make them particularly suitable for applications involving harsh operating conditions, including extreme temperatures, aggressive solvents, and high-pressure environments [34].

The superior durability of ceramic membranes allows effective operation in scenarios where polymeric membranes might degrade or fail. For instance, in water and wastewater treatment, ceramic membranes demonstrate remarkable resistance to fouling and can withstand rigorous cleaning protocols involving strong acids, alkalis, or oxidizing agents [35]. This resilience not only extends the operational lifespan of the membranes but also enhances process efficiency by reducing downtime and maintenance costs [36].

Moreover, ceramic membranes exhibit high hydrophilicity, which contributes to higher water fluxes and reduced membrane fouling compared to their polymeric counterparts. This characteristic is particularly advantageous in applications such as water purification, where maintaining high permeability and minimizing fouling are critical for system performance.

Despite their numerous advantages, the widespread adoption of ceramic membranes has been historically hindered by higher initial production costs relative to polymeric membranes. However, ongoing advancements in manufacturing techniques and materials science are progressively mitigating these cost concerns, making ceramic membranes increasingly viable for a broader range of industrial applications.

In summary, ceramic membrane technology represents a significant evolution in separation processes, offering enhanced performance, durability, and versatility. Its application across

various industries underscores its potential to address complex filtration challenges, paving the way for more efficient and sustainable operations.

1. FUNDAMENTALS OF MEMBRANE TECHNOLOGY

Membrane technology is a widely used separation method that relies on a selective barrier (membrane) to regulate the passage of substances based on size, charge, or solubility. It plays a crucial role in industrial applications such as water purification, food processing, pharmaceutical production, and chemical separation. Among membrane types, ceramic membranes stand out due to their superior durability, thermal and chemical resistance, and long lifespan compared to polymeric membranes [37].

1.1. Principles of Membrane Separation

Membrane separation processes work based on three primary mechanisms:

- **Sieving (Size Exclusion):** The most common mechanism where particles larger than the membrane's pore size are physically blocked, while smaller ones pass through. This is typical for microfiltration and ultrafiltration membranes used in water treatment and food industries [38].
- **Diffusion:** In this mechanism, molecules dissolve within the membrane material and move across it due to a concentration gradient. This process is used in gas separation membranes and reverse osmosis applications [39].
- **Electrostatic Interactions (Charge-Based Separation):** Certain membranes carry surface charges, which attract or repel ions and charged particles, aiding in separation. This principle is used in nanofiltration and ion-exchange membranes [40].

The performance of a membrane system depends on various factors, including pore size distribution, membrane material properties, operating pressure, and fouling behavior [41].

1.2. Types of Membranes: Ceramic vs. Polymeric

Membranes are classified into ceramic membranes and polymeric membranes based on their material composition. Each type has specific advantages and limitations depending on the application.

1.2.1. Ceramic Membranes

- Made from alumina (Al_2O_3), zirconia (ZrO_2), titania (TiO_2), and silicon carbide (SiC).
- Highly resistant to high temperatures (up to 1000°C) and harsh chemicals.
- Extremely durable and mechanically strong, making them ideal for challenging environments.
- Used in wastewater treatment, dairy processing, oil-water separation, and gas purification.
- High initial cost [42].

1.2.2. Polymeric Membranes

- Made from synthetic polymers like polyethersulfone (PES), polyvinylidene fluoride (PVDF), and cellulose acetate.
- Less expensive
- More prone to degradation under extreme temperatures and chemical exposure.
- Widely used in reverse osmosis (RO), nanofiltration (NF), and ultrafiltration (UF) for drinking water treatment [43].

1.3. Classification of Membrane Separation Processes:

Membrane separation processes can be categorized into various classes. Microfiltration, ultrafiltration, nanofiltration, and reverse osmosis are techniques that involve the separation of two liquid phases by selective permeation through semipermeable membranes [44]. Pervaporation and gas-phase permeation also utilize membranes; however, they differ from the aforementioned techniques as they involve gas-phase separation rather than liquid-phase separation. These methods serve as alternatives to distillation and azeotropic mixture dehydration [45]. Lastly, electro-membrane

technologies operate based on ion exchange principles and utilize electric current as the driving force for separation [46].

- **Microfiltration:** Microfiltration membranes were first developed around 1945 in Germany to facilitate rapid and frequent bacteriological analysis of drinking water. This technique quickly became a convenient tool not only for analysis but also for production [47]. Typically operated under low pressure (0.1 to 5 bar), microfiltration is commonly used in tangential flow filtration applications [48].
- **Ultrafiltration:** Ultrafiltration membranes are artificial porous membranes, either organic or inorganic, exhibiting an asymmetric (anisotropic) or composite structure. These membranes consist of a thin selective layer (typically around 10 micrometers thick), which contains mesopores and is supported by one or more porous and mechanically robust layers [49]. In organic anisotropic membranes, the supporting structure has larger pores that facilitate efficient drainage while maintaining mechanical integrity [50].
- **Nanofiltration:** Nanofiltration operates in an intermediate range between reverse osmosis and ultrafiltration. It allows for the separation of components with sizes around the nanometer scale (10^{-9} m), hence its name [51]. Monovalent ionized salts and small organic molecules with a molar mass below approximately 200–250 g/mol typically pass through nanofiltration membranes, while multivalent ions (such as calcium, magnesium, aluminum, and sulfates) and larger organic molecules (above 250 g/mol) are effectively retained [52]. The transfer mechanisms of nanofiltration are intermediate between those of ultrafiltration and reverse osmosis. This process is frequently employed in water softening applications [53]. A key advantage of nanofiltration is its ability to separate low-molecular-weight compounds at relatively low to moderate pressures [54].

Reverse Osmosis (fig II.1 ,fig II.2): Reverse osmosis membranes possess pore sizes ranging between 0.2 and 1 nm. This method is one of the most cost-effective solutions for removing 90–99% of contaminants from a fluid [55]. Osmosis is a naturally occurring phenomenon in

biological membranes, where a concentration gradient across a semipermeable membrane induces solvent flow from the lower-concentration side to the higher-concentration side due to osmotic pressure. Reverse osmosis applies an external pressure exceeding the osmotic pressure, forcing the solvent to move from the more concentrated solution to the less concentrated one [56].

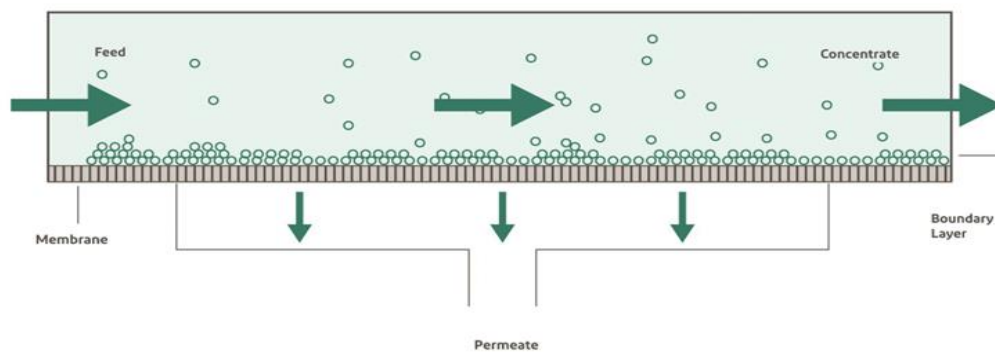


Figure 0.1 Crossflow membrane filtration (DuPont Water Solutions. (2020). FilmTec™ Reverse Osmosis Membranes Technical Manual.)

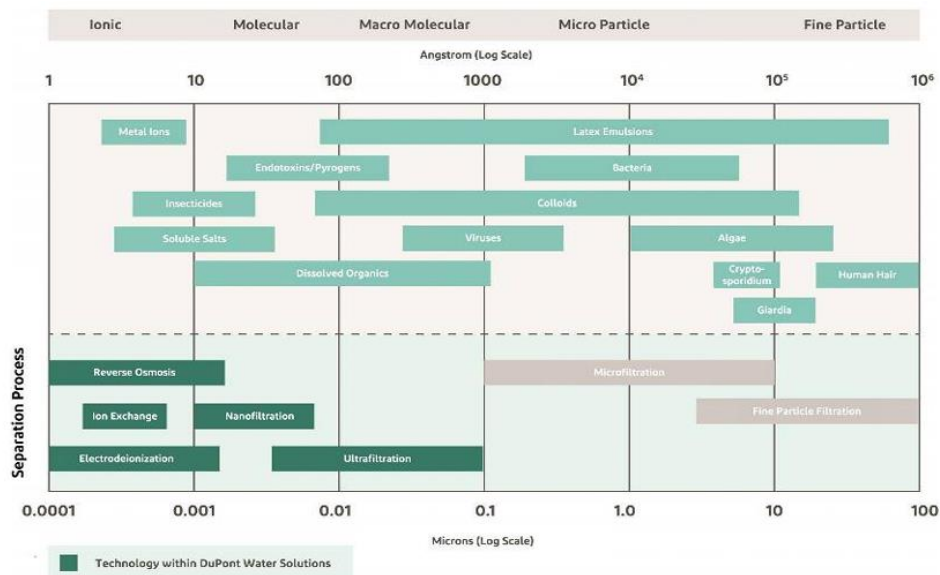


Figure 0.2 Ranges of filtration processes (DuPont Water Solutions. (2020). FilmTec™ Reverse Osmosis Membranes Technical Manual.)

1.3.1. Structure and Properties of Ceramic Membranes:

A. Composition and Materials:

Ceramic membranes are composed of inorganic oxides that provide exceptional stability under extreme operating conditions. The most commonly used materials include:

- **Alumina (Al_2O_3):** Offers excellent mechanical strength and chemical resistance, making it widely used in microfiltration and ultrafiltration applications [57].
- **Zirconia (ZrO_2):** Known for its superior resistance to thermal shock and corrosion, commonly used in high-temperature applications [58].
- **Titania (TiO_2):** Provides strong photocatalytic properties, making it suitable for advanced oxidation processes and self-cleaning membranes [59].
- **Silicon Carbide (SiC):** Exhibits exceptional resistance to acids, alkalis, and extreme temperatures, making it ideal for aggressive chemical filtration [60].
- **Mixed-Oxide Ceramics:** Combining different oxides (e.g., alumina-titania) enhances specific properties like permeability and fouling resistance [61].

Ceramic membranes can also be coated with hydrophilic or hydrophobic layers to modify their surface properties, improving selectivity and reducing fouling [62].

B. Membrane Morphology and Pore Structure:

Ceramic membranes typically have an asymmetric structure, composed of multiple layers with varying pore sizes. This structure enhances filtration efficiency by providing high permeability while maintaining mechanical strength. The key morphological characteristics include:

- **Support Layer (Macroporous, 1–10 μm pores):** Provides mechanical strength and stability.
- **Intermediate Layer (Mesoporous, 5–50 nm pores):** Controls permeability and supports the selective layer.
- **Top (Active) Layer (Microporous, <2 nm pores):** Determines selectivity based on molecular size and charge interactions [63].

The pore size distribution influences separation performance, with microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) membranes differing based on pore diameter [64].

C. Chemical, Mechanical, and Thermal Properties:

Ceramic membranes are superior to polymeric membranes in terms of durability and resistance to extreme conditions. Their key properties include:

1. Chemical Stability:

- Resistant to extreme pH (0–14), making them suitable for acidic and alkaline wastewater treatment.
- Inert to organic solvents, enabling applications in chemical and pharmaceutical industries [65].
- Less susceptible to biofouling due to their smooth, non-porous surfaces [66].

2. Mechanical Strength:

- Withstands high pressures (up to 10 MPa or 100 bar) without structural damage.
- Highly resistant to abrasion, ensuring longer lifespan compared to polymeric membranes [67].
- Can be used in high-flux filtration systems with minimal structural deformation.

3. Thermal Resistance:

- Operates at temperatures up to 800–1000°C, making them ideal for hot gas filtration and thermal desalination.
- Unlike polymeric membranes, which degrade above 100°C, ceramic membranes maintain their integrity under extreme conditions [68].
- Additionally, surface modifications such as plasma treatment can further enhance hydrophilicity, reducing fouling and improving filtration performance [69].

D. Transport Mechanisms in Ceramic Membranes:

The movement of solutes and solvents through ceramic membranes depends on several transport mechanisms, including:

1. Poiseuille Flow (Convective Transport):

Governs the movement of liquids in microfiltration and ultrafiltration membranes.

Fluid moves through pores due to applied pressure.

2. Knudsen Diffusion:

This occurs when gas molecules travel through nanoporous membranes, frequently used in gas separation applications.

Transport depends on molecule-wall collisions rather than direct pore flow [70].

3. Solution-Diffusion Mechanism:

Used in nanofiltration (NF) and reverse osmosis (RO), where solutes dissolve in the membrane and move across due to a concentration gradient.

Selective based on molecular size and polarity [71].

4. Surface Diffusion:

Molecules adsorb onto the membrane surface and migrate along it before desorption.

Important in pervaporation and catalytic membrane reactors.

5. Capillary Condensation:

Occurs in membranes with mesoporous structures where vapor condenses into liquid inside the pores, affecting filtration performance.[72]

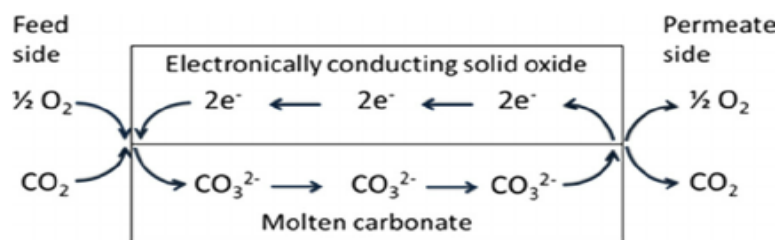


Figure 0.3 Papaioannou, E. I., Qi, H., & Metcalfe, I. S. (2015).
Membrane transport mechanism. ResearchGate

1.3.2. Manufacture process of ceramic membrane:

The manufacturing process of ceramic membranes involves several critical steps that determine their final properties, including porosity, mechanical strength, and chemical resistance. The key stages in this process include raw material selection, shaping, drying, sintering, and surface modification.

A. Raw Material Selection

The first step in the manufacturing process is the selection of raw materials, which typically include metal oxides such as alumina (Al_2O_3), zirconia (ZrO_2), titania (TiO_2), and silicon carbide (SiC). These materials are chosen based on their thermal stability, chemical resistance, and mechanical properties [73]. Additives such as binders, dispersants, and pore formers are also used to enhance processing characteristics and control the final membrane structure.

B. Shaping Methods

Once the raw materials are prepared, they are shaped into the desired membrane form. Several shaping techniques are commonly used:

- **Extrusion:** A paste-like mixture is forced through a die to create tubular or monolithic structures, commonly used for water filtration applications [74].
- **Tape Casting:** A slurry is spread into thin layers to produce flat-sheet membranes, often used in gas separation and pervaporation [75].
- **Slip Casting:** A suspension of ceramic particles is poured into a mold, allowing liquid to drain and leaving a consolidated layer behind [76].
- **Pressing:** Powdered ceramic materials are compressed under high pressure to form dense structures suitable for high-strength applications [77].

C. Drying and Binder Removal

After shaping, the membranes undergo a drying process to remove excess moisture. This step is crucial to prevent defects such as cracking or warping. Organic additives (binders) used during shaping are then eliminated by controlled heating at moderate temperatures (~200-600°C) in a process known as debinding [78].

D. Sintering

Sintering is the most critical step in the manufacturing process. The shaped membranes are heated to high temperatures (800-1600°C) to fuse ceramic particles together, creating a strong and porous structure. The sintering conditions, such as temperature, time, and atmosphere, significantly influence the final membrane properties, including pore size and mechanical strength [78].

E. Surface Modification and Coating

To improve selectivity and performance, ceramic membranes often undergo surface modification. This can include:

- **Sol-Gel Coating:** A thin layer of nanostructured material is applied to reduce pore size and enhance selectivity [79].

- **Chemical Vapor Deposition (CVD):** A gaseous precursor reacts on the membrane surface, forming a uniform coating that enhances chemical resistance [80].
- **Hydrophilic or Hydrophobic Treatment:** Modifications such as silanization or polymer grafting can alter membrane wettability, affecting filtration efficiency [81].

F. Final Testing and Quality Control

Before deployment, ceramic membranes undergo rigorous quality control testing to ensure performance consistency. Standard tests include:

- Pore Size Distribution Analysis (e.g., mercury porosimetry, BET surface analysis) [82].
- Mechanical Strength Testing (e.g., compressive and flexural strength tests) [83].
- Chemical and Thermal Stability Assessments to verify resistance to extreme conditions [84].

2. Classification of ceramic membranes

Membranes are also categorized based on their pore size and separation mechanism into porous and dense membranes [85].

2.1. Porous Membranes

Porous membranes function similarly to conventional filters but at a much smaller scale, with pore sizes ranging from 0.01 μm to 1 μm . They allow selective separation based on size exclusion, where larger particles are retained while smaller particles pass through [86].

The classification based on pore diameter includes:

- **Microporous membranes:** Pore size < 2 nm (e.g., gas separation membranes) [87].
- **Mesoporous membranes:** Pore size 2–50 nm (e.g., ultrafiltration membranes) [88].
- **Macroporous membranes:** Pore size > 50 nm (e.g., microfiltration membranes).
- **Reverse Osmosis (RO):** Water desalination and wastewater treatment [89].
- **Gas Separation:** Hydrogen recovery, oxygen/nitrogen separation [90].
- **Pervaporation:** Separation of organic solvents from water [91].

3. Classification by geometry:

Ceramic membranes are designed in various configurations to optimize their performance for specific applications. The choice of configuration depends on factors such as available space, filtration efficiency, mechanical strength, and ease of cleaning. The most commonly used ceramic membrane configurations include tubular, flat-sheet, monolithic, and hollow fiber membranes.

3.1. Tubular Membranes

Tubular ceramic membranes consist of cylindrical tubes with an internal porous structure through which the feed solution flows. Filtration occurs either from inside-out or outside-in, depending on the application.

3.2. Flat-Sheet Membranes

Flat-sheet ceramic membranes are thin, rigid plates made from ceramic materials. They are typically used in crossflow filtration systems where the feed solution moves parallel to the membrane surface.

3.3. Monolithic Membranes

Monolithic ceramic membranes consist of a single porous ceramic block with multiple flow channels. They provide high mechanical strength and large filtration areas within a compact structure.

3.4. Hollow Fiber Membranes:

Hollow fiber ceramic membranes consist of multiple tiny, hollow cylindrical fibers bundled together. The feed solution is filtered as it passes through the fiber walls.

4. Operating Principles of Ceramic Membrane Treatment

Ceramic membranes operate based on different separation mechanisms that define their selectivity and efficiency. These mechanisms influence the retention of specific particles or molecules, making ceramic membranes suitable for various applications, including water treatment, food processing, and pharmaceutical separations. The key filtration mechanisms

involved in ceramic membrane treatment are sieving (size exclusion), adsorption, charge interactions, solution-diffusion, and hydrophobic-hydrophilic interactions.

4.1. Filtration Mechanisms:

a. Sieving (Size Exclusion):

The sieving mechanism, also referred to as size exclusion, is the primary separation principle in microfiltration (MF) and ultrafiltration (UF) processes. In this mechanism, particles and molecules are separated based on their size relative to the pore size of the membrane. When a feed solution is passed through a membrane, particles larger than the membrane pores are retained, while smaller particles permeate through the structure [92]. The separation efficiency depends on the pore size distribution, membrane material, and operating conditions such as pressure and crossflow velocity [93].

Microfiltration membranes typically have pore sizes ranging from 0.1 to 10 μm , allowing for the retention of suspended solids, bacteria, and some large macromolecules. Ultrafiltration membranes, on the other hand, have smaller pore sizes in the range of 2 to 100 nm, making them effective for protein separation, virus removal, and concentration of macromolecules [94].

b. Adsorption:

Adsorption is a filtration mechanism where solutes interact with the membrane surface and become selectively retained due to physical or chemical interactions. This mechanism is particularly significant in nanofiltration (NF) and ultrafiltration (UF), where organic compounds, proteins, and dissolved contaminants interact with the membrane material [95].

The efficiency of adsorption depends on various factors:

- Membrane material and surface chemistry (hydrophobic or hydrophilic properties).
- pH and ionic strength of the solution, which affect the electrostatic interactions between solutes and the membrane.

- Membrane fouling, as adsorbed molecules may accumulate on the surface, leading to performance decline over time [96].
- maieutical contaminants) from water [97].

4.2. Filtration Configurations:

The configuration of a filtration system significantly impacts the efficiency, membrane lifespan, and overall performance of a ceramic membrane process. Two primary filtration modes exist in membrane technology: dead-end filtration and crossflow filtration. The choice between these configurations depends on factors such as fouling behavior, energy consumption, and the required separation efficiency [98].

4.2.1. Dead-End Filtration:

Definition and Mechanism:

In dead-end filtration, the feed solution is forced perpendicularly against the membrane surface, with no tangential flow component. As a result, the permeate passes through the membrane, while the retained particles accumulate on the surface, forming a filter cake layer.

Advantages:

- Lower energy consumption due to simpler system design [99].
- Higher initial permeate flux compared to crossflow filtration.
- Straightforward operation and maintenance, making it suitable for batch processes.

Disadvantages:

- Severe fouling and rapid clogging due to retained particles accumulating on the membrane surface.
- Frequent cleaning requirements, increasing downtime and chemical usage [100].
- Limited suitability for high-solid-content solutions, as accumulation reduces membrane efficiency.

4.2.2. Crossflow Filtration

Definition and Mechanism:

Unlike dead-end filtration, crossflow filtration involves a tangential flow of feed solution across the membrane surface, which helps sweep away retained particles. This reduces cake formation and maintains a more stable filtration rate over time. The feed solution is continuously recirculated, allowing for higher efficiency and reduced fouling [101].

Advantages:

- Minimization of fouling and cake layer formation, leading to extended membrane lifespan [102].
- Steady-state operation, providing consistent filtration performance over time.
- Higher processing capacity, particularly for suspensions with high solid loads.
- Reduced frequency of chemical cleaning, decreasing operational costs [103].

4.2.3. Comparison of Dead-End and Crossflow Filtration

The choice between dead-end and crossflow filtration depends on the application, feed characteristics, and operational constraints. Table 1 summarizes the key differences.

Feature	Dead-End Filtration	Crossflow Filtration
Flow Direction	Perpendicular to membrane	Tangential to membrane
Fouling Tendency	High	Low
Energy Consumption	Low	High
Suitability for High-Solid Feeds	Low	High
Cleaning Frequency	High	Low
Application	Drinking water, sterile filtration	Wastewater, food, processing

4.2.4. Advantages of Crossflow Filtration in Reducing Fouling

Fouling remains one of the major limitations in membrane processes, leading to reduced permeability, increased energy costs, and shorter membrane lifespan [104]. Crossflow filtration significantly mitigates these effects through:

A. Shear Force Reduction of Cake Layer Formation

- The tangential flow prevents excessive buildup of retained particles, delaying clogging and preserving filtration efficiency [105].

B. Hydrodynamic Back-Transport Mechanisms

- In crossflow mode, concentration polarization is reduced, minimizing the risk of biofouling, organic fouling, and scaling [106].

C. Better Control Over Process Parameters

- Adjusting crossflow velocity and transmembrane pressure (TMP) enables optimized flux and selectivity, reducing membrane degradation over time [107].

D. Reduced Cleaning and Maintenance Costs

- Since fouling is lower in crossflow filtration, chemical cleaning is required less frequently, leading to improved membrane longevity and lower operational expenses [108].

4.3. Operating Conditions

The performance of ceramic membrane filtration is significantly influenced by key operating parameters, including temperature, pH, pressure, and flow rate. Optimizing these conditions is essential for enhancing filtration efficiency, minimizing fouling, and ensuring membrane longevity.

A. Temperature Tolerance:

Ceramic membranes have excellent thermal stability, enabling them to operate at significantly higher temperatures than polymeric membranes. This property makes them suitable for applications requiring sterilization or high-temperature filtration.

- Typical operating temperature range: up to 800°C, depending on the membrane material [109]
- High temperatures can enhance permeate flux by lowering fluid viscosity but may also increase the risk of scaling due to salt precipitation [110]

B. pH Stability:

Ceramic membranes exhibit outstanding chemical resistance across a broad pH range, allowing them to handle highly acidic and alkaline solutions without degradation.

- Typical pH range: 0–14, depending on membrane composition [111]
- Unlike polymeric membranes, ceramics can withstand aggressive chemical cleaning agents without losing performance [112]

C. Pressure Requirements:

The driving force for membrane filtration is pressure, which determines the flux and efficiency of separation. Different membrane processes operate within distinct pressure ranges:

Filtration type	Operating pressure (bar)
Microfiltration	0.1-2
Ultrafiltration	1-5
Nanofiltration	4-30
Reverse osmosis	30-80

Higher pressure increases flux, but also leads to higher energy consumption and potential membrane compaction [113]

D. Flow Rate and Crossflow Velocity:

The flow rate and crossflow velocity determine shear forces on the membrane surface, impacting performance and fouling control:

- Higher crossflow velocity reduces the formation of fouling layers, enhancing membrane lifespan [114]
- Typical crossflow velocity: 1–6 m/s, depending on application [115]

Materials & Methods

1. Filtration test:

Membrane filtration has become an essential method for water treatment. In this context, the development of ceramic membranes from clay materials modified with metal oxides offers high-performance solutions. With an optimized geometric design, these membranes exhibit a structure that ensures both good porosity and high mechanical strength. This study highlights the use of a microporous alumina-based membrane for the treatment of liquid effluents. [116, 117]

2. Characterization of ceramic Membranes:

The performance of a membrane depends largely on its characteristics:

- **Separation efficiency:** A well-defined cut-off threshold (for ultrafiltration) and known pore diameter (for microfiltration).
- **High permeation flux:** Achieved by increasing porosity and reducing membrane thickness, which helps minimize pressure losses.
- **High mechanical, chemical, and thermal resistance:** Allowing for effective, economical, and easy cleaning processes.
- The average pore size and its distribution are critical parameters that directly impact membrane performance [118].

3. The crossflow filtration unit:

The filtration setup typically includes a feed tank, the membrane, a pump, and two pressure gauges located upstream and downstream (2) of the membrane housing.

The liquid to be filtered is pumped tangentially across the membrane at a pressure of 3 Bar. Due to the applied pressure, part of the solution passes through the membrane (the permeate) and is collected for analysis. The portion retained by the membrane (the retentate) is recirculated back into the feed tank.

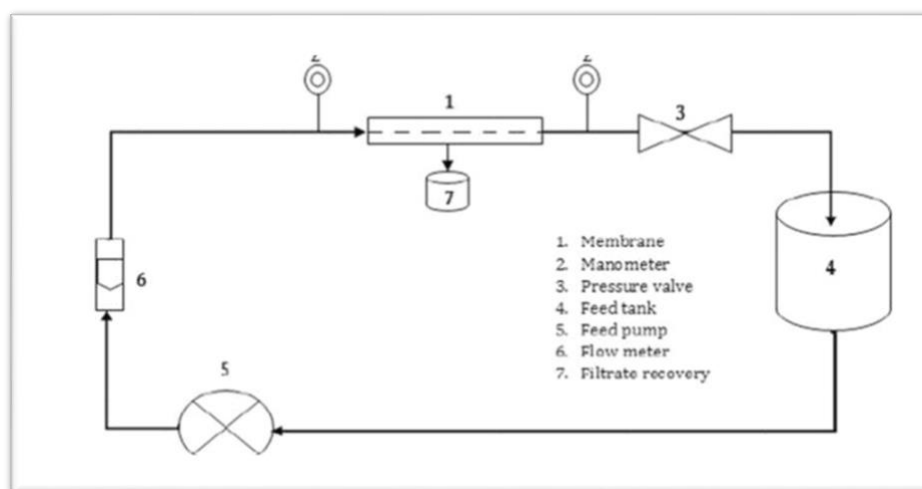


Figure 0.1 Schematic Presentation of the crossflow filtration unit



Figure 0.2 The crossflow filtration unit

4. Liquid Effluents:

The solutions used in this study were prepared in the laboratory.

The experiments were conducted on the following liquid effluents:

Solutions with concentrations of 10^{-4} M and 10^{-5} M:

- Malachite green
- Methylene blue
- Lead (III) nitrate $\text{Pb}(\text{NO}_3)_2$
- Cadmium nitrate $\text{Cd}(\text{NO}_3)_2$
- Wastewater from the Tayal factory.

5. Measurement of pH, Conductivity, and TDS:

5.1 pH:

The pH measurement, or hydrogen potential, indicates the concentration of H^+ ions in the solution. This parameter plays a key role in many physicochemical equilibria and is influenced by various factors. pH values were measured using a pH meter, ADWA AD1000.

5.2 Conductivity:

Conductivity measures the ability of water to conduct electrical current between two electrodes. Most dissolved substances in water exist as electrically charged ions. Therefore, measuring conductivity gives an indication of the total amount of dissolved salts in the water. Conductivity is also temperature-dependent: it increases as the water temperature rises. For this reason, measurement results are typically reported as conductivity values normalized to 20 or 25 °C. Most field instruments automatically perform this temperature correction.

Just like temperature, variations in conductivity can reveal pollution, mixing zones, or infiltration areas. Conductivity is also a useful tool for validating the physicochemical analysis of water [119].

5.3 Total Dissolved Solids (TDS):

Total Dissolved Solids (TDS) refer to the combined amount of organic and inorganic substances dissolved in water, including minerals, salts, and small quantities of organic matter, usually measured in milligrams per liter (mg/L) [120]. These substances are small enough to pass through standard filtration systems and can significantly influence water

quality, taste, and suitability for various uses, including drinking, agriculture, and industrial processes [121].

TDS is closely related to conductivity because both are influenced by the presence of dissolved ions. However, while conductivity reflects the water's ability to conduct electricity, TDS provides a direct estimate of the total mass of dissolved components. Many modern water quality instruments estimate TDS by converting conductivity readings using a specific conversion factor, which varies depending on the ionic composition of the water [122].

Fluctuations in TDS levels can signal events such as contamination, dilution, or evaporation. Monitoring TDS is therefore crucial for identifying pollution sources, evaluating the effectiveness of treatment systems, and verifying results from more detailed chemical analyses [123].

6. UV-Visible Spectrophotometry:

UV-Visible spectrophotometry is an analytical technique used to quantify substances in solution, provided that these compounds absorb light in the wavelength range of 200 to 800 nm. In this study, it is applied to determine the concentration of three dyes used as models for organic pollutants.

The instrument used, a SPECORD® 210 PLUS spectrophotometer, operates with a double-beam system. A white light source emits a beam that passes through a monochromator, which isolates a specific wavelength. This beam is then split into two paths: one passes through a reference cuvette containing only the solvent, while the other passes through the sample solution.

Two photodetectors measure the light intensity of each beam. The absorbance (A) of the sample is calculated using the following equation:

$$A = -\log_{10}(I / I_0)$$

where I_0 is the intensity of the reference beam and I is the intensity transmitted through the sample.

The dye concentration is then determined using the Beer-Lambert law:

$$A = \varepsilon \times L \times C$$

where ε is the molar extinction coefficient ($\text{L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$), L is the cuvette path length (cm), and C is the concentration of the substance ($\text{mol} \cdot \text{L}^{-1}$).

Before each measurement series, a calibration curve is created to identify the maximum absorption wavelength (λ_{max}) specific to each compound. If the absorbance exceeds the instrument's detection range, the sample must be diluted to ensure accurate readings.

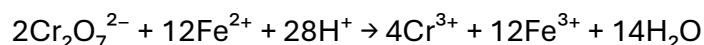
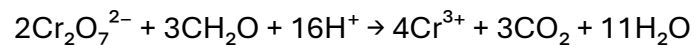
7. Determination of Chemical Oxygen Demand (COD):

Chemical Oxygen Demand (COD) is a key parameter used to assess the amount of oxygen required to chemically oxidize organic and inorganic compounds present in water. It is crucial for evaluating the pollution load of both raw and treated wastewater.

Method: Oxidation with Potassium Dichromate

The method involves oxidizing oxidizable matter using an excess of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in a strongly acidic medium (sulfuric acid) under reflux for two hours. After the reaction, the residual dichromate is titrated with a ferrous ammonium sulfate solution (Mohr's salt). COD is determined by calculating the amount of dichromate consumed. A catalyst (Ag^+) and a chloride complexing agent (Hg^{2+}) are used to prevent interferences.

Main redox reactions:



Reagent Preparation:

- Diluted sulfuric acid (4 mol/L): Add 220 mL of concentrated H_2SO_4 to 500 mL of distilled water, cool, and make up to 1 L.
- Sulfuric acid–silver sulfate mixture: Dissolve 10 g AgSO_4 in 40 mL water, add 960 mL concentrated H_2SO_4 . Let rest 1–2 days.

- Mohr's salt (0.12 mol/L): Dissolve 47 g in water, add 20 mL H₂SO₄, then dilute to 1 L.
- Potassium dichromate (0.04 mol/L) with mercury sulfate: Dissolve 80 g HgSO₄ in 800 mL water, add 100 mL H₂SO₄, cool, then add 11.767 g of dried K₂Cr₂O₇.
- Ferroin indicator: Dissolve 0.7 g FeSO₄ and 1.5 g 1,10-phenanthroline monohydrate in 100 mL water.

COD Calculation:

The COD value in g O₂/L is calculated as follows:

$$COD = \frac{8000 \times (VT - VE)n \times T}{V}$$

where:

VT = volume of ferrous solution used in the blank (mL)

VE = volume used in the sample (mL)

T = concentration of the ferrous solution (mol/L)

V = volume of the sample (mL)

If dilution was performed, multiply the result by the inverse of the dilution factor. A blank test with distilled water must always be run in parallel.

Results & Discussions

1. Calibration Curves of the Dyes (Fig 1 & Fig 2)

From a stock solution, a series of diluted (daughter) solutions were prepared at varying concentrations. These solutions, corresponding to the two selected dyes, were analyzed using UV-Visible spectrophotometry in the visible range.

Each dye was measured at its maximum absorbance wavelength:

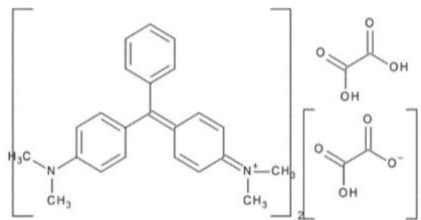
- **Methylene blue at 665 nm**
- **Malachite green at 619 nm**

The absorbance values obtained at these wavelengths were plotted against the corresponding concentrations. The resulting calibration curves are shown in the following figures.

Malachite green:

Structural Model: Cationic dye

- Maximum Absorbance ($\lambda_{\max}$): 619 nm
- Molecular Formula: $C_{52}H_{54}N_4O_{12}$
- Molecular Weight (M): 927.02 g/mol
- Melting Point: $\sim 159^\circ\text{C}$
- Solubility: 110 g/L in water at 24°C



Description:

Malachite green is a triphenylmethane dye that is water-soluble. It is commonly used for staining bacteria and plant tissues infected by fungi, as well as for spore visualization in microbiological and botanical studies.

a. Calibration curve of Malachite green :

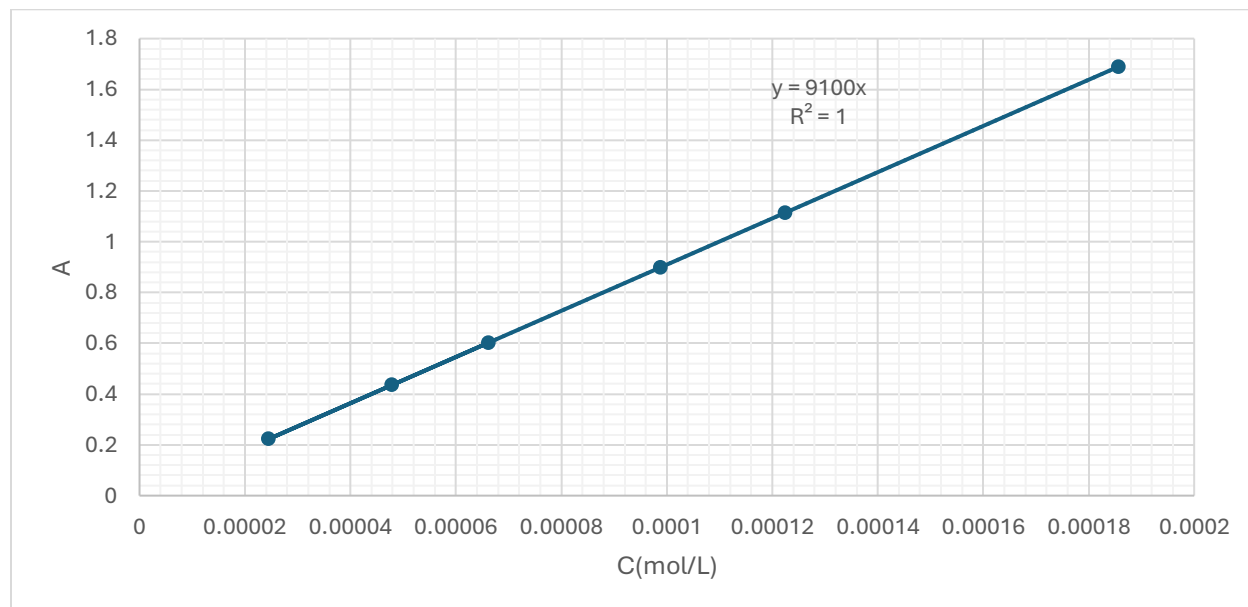
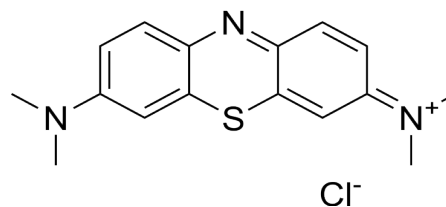


Figure 0.1 Calibration curve of Malachite green ($\lambda_{\max} = 619\text{nm}$)

The calibration curve for malachite green shows a clear linear relationship between absorbance and concentration at 619 nm. This indicates that the spectrophotometric method is reliable for measuring dye concentrations, and the system behaves consistently according to the Beer-Lambert law. This curve provides the foundation for later concentration measurements during retention experiments.

Methylene blue:

- Maximum Absorbance ($\lambda_{\max}$): 665 nm
- Molecular Formula: $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$
- Molecular Weight (M): 319.85 g/mol
- Melting Point: $\sim 100^\circ\text{C}$ (decomposes)
- Solubility: 43 g/L in water at 25°C

**Description:**

Methylene Blue is a cationic thiazine dye, highly soluble in water, and widely used in biology and chemistry. It is used for staining acidic cellular components (like nucleic acids),

bacterial identification, and redox indicators in analytical chemistry. It is also applied in medical diagnostics and wastewater treatment as a model pollutant.

b. Calibration curve of Methylene blue:

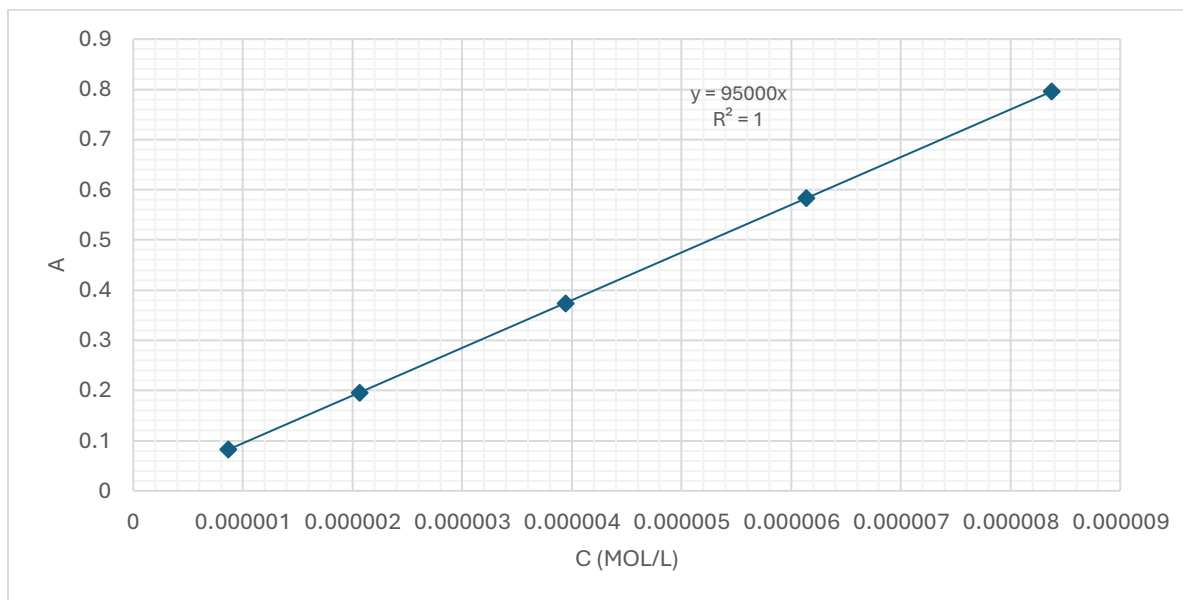


Figure 0.2 Calibration curve of Methylene blue (λ max= 665nm)

The calibration curve of methylene blue displays a strong linear correlation between absorbance and concentration at 665 nm. This confirms that the system follows the Beer–Lambert law, indicating reliable spectrophotometric behavior for quantitative analysis. The linearity suggests that the chosen wavelength is appropriate, and no saturation or deviation occurs within the concentration range tested. This curve provides the baseline to calculate unknown methylene blue concentrations during retention and filtration tests.

1.1. Dyes Retention Rate

The retention rate (or rejection rate) of a dye represents the membrane's ability to retain dye molecules and prevent them from passing into the permeate. It is a key parameter to evaluate membrane performance in filtration processes. The retention rate $R\%$ is typically calculated using the following equation:

$$R(\%) = \left(1 - \frac{C_p}{C_i}\right) \times 100$$

C_p : the concentration of the dye in the permeate

C_i : the concentration of the dye in the initial solution

1- Malachite green

Retention rate measures the percentage of dye molecules blocked by the membrane during filtration. It reflects how efficiently the membrane captures malachite green from solution. The figures below (IV.3 & IV.4) show how the retention rate evolves over time at two concentrations (10^{-4} M and 10^{-5} M).

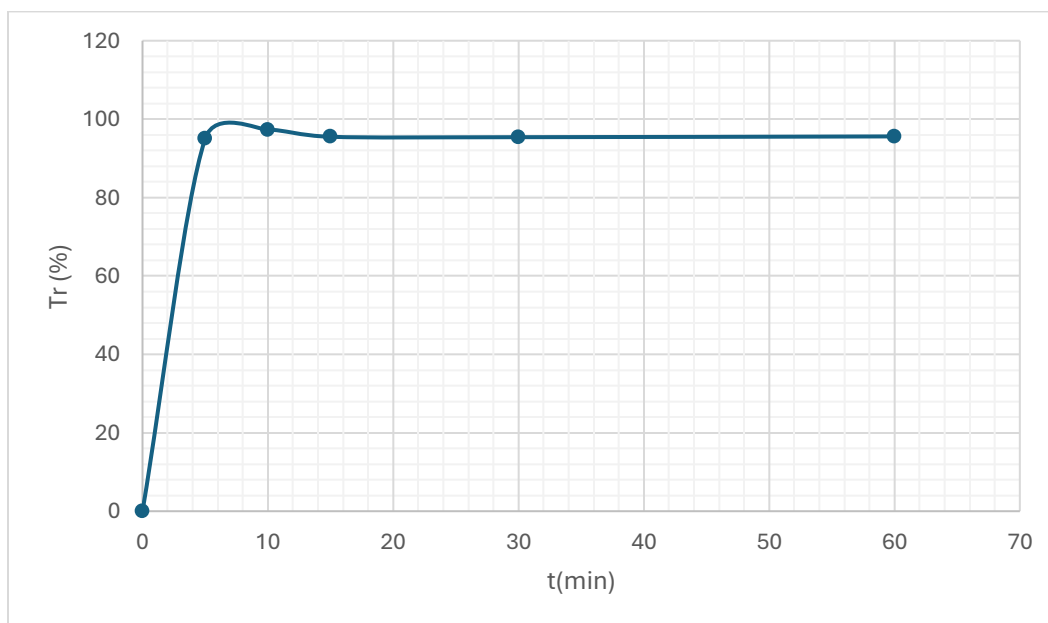


Figure 0.3 Variation of Malachite Green 10^{-4} Retention Rate as a Function of Time.

At 10^{-4} M concentration, the retention rate of malachite green increases steadily with time. This trend suggests that the membrane is progressively capturing more dye, likely due to adsorption and size exclusion mechanisms. Over time, the curve appears to stabilize, which could mean the membrane is approaching its saturation limit.

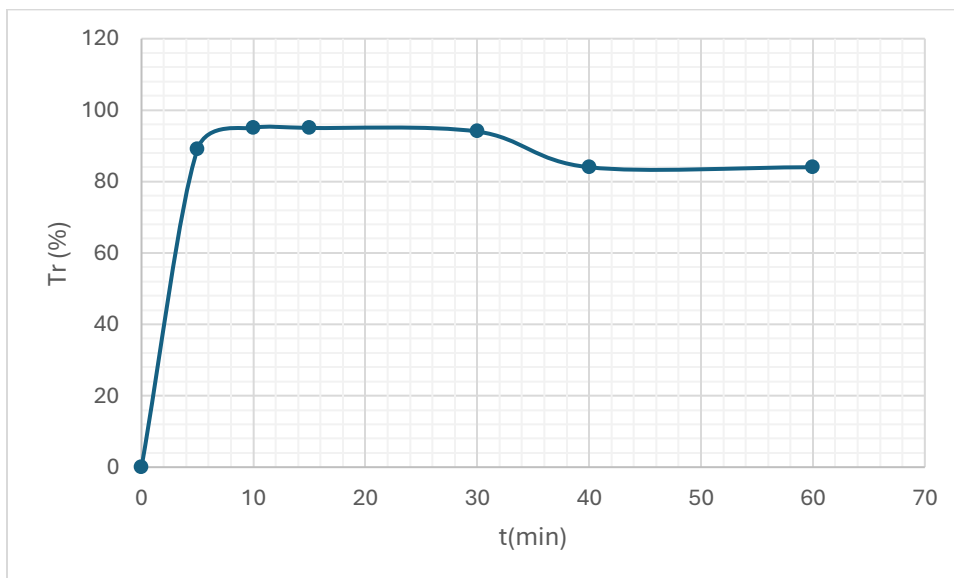


Figure 0.4 Variation of Malachite Green 10^{-5} Retention Rate as a Function of Time.

At the lower concentration of 10^{-5} M, the membrane still shows efficient retention, with the rate increasing before leveling off. The relatively quicker stabilization may be due to the lower amount of dye available, allowing the membrane to reach equilibrium faster. This confirms the membrane's ability to handle both high and low dye concentrations effectively.

2- Methylene blue:

The retention rate helps evaluate how effectively the membrane blocks methylene blue molecules. A rising retention value suggests good membrane performance. The following figures (IV.5 & IV.6) show this evolution for two concentrations over time.

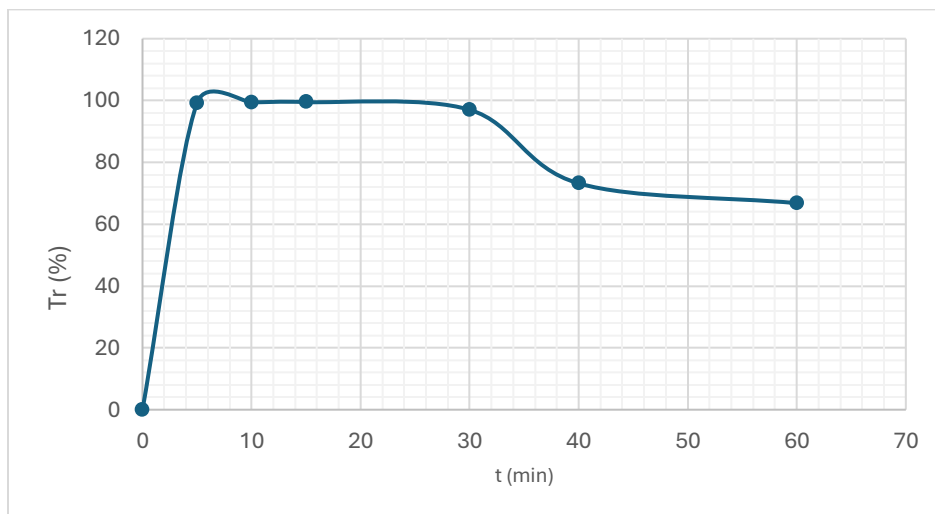


Figure 0.5 Variation of Methylene blue 10^{-4} Retention Rate as a Function of Time.

For methylene blue at 10^{-4} M, the retention rate follows a similar increasing pattern. This suggests that the membrane effectively filters this dye as well, probably through similar mechanisms as with malachite green. The consistent rise implies the membrane remains active and efficient over the test period.

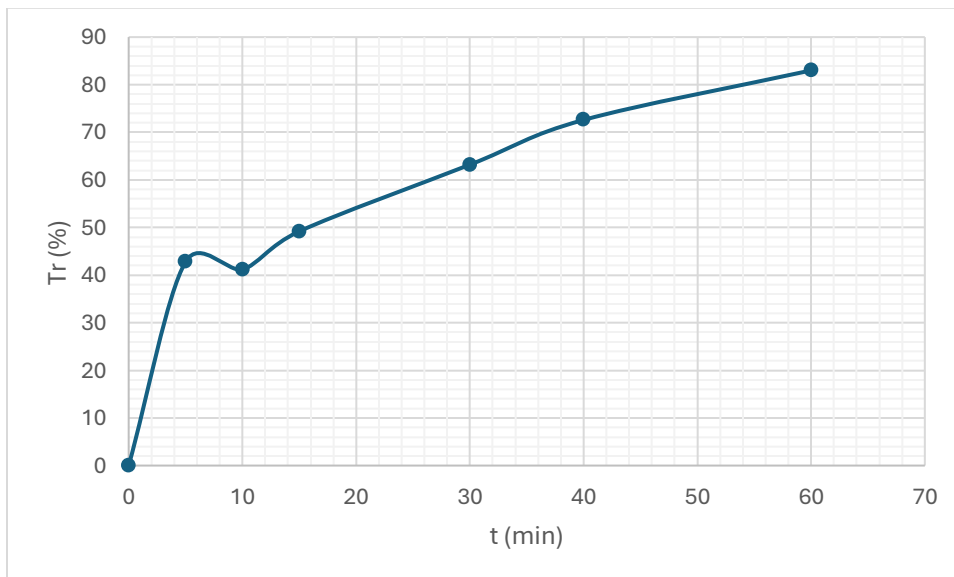


Figure 0.6 Variation of Methylene blue (10^{-5}) Retention Rate as a Function of Time.

At 10^{-5} M, methylene blue is also retained effectively by the membrane. The retention rate rises rapidly and then stabilizes, indicating fast initial adsorption. This performance at low concentrations further supports the membrane's versatility in treating diluted effluents.

1.2. pH of the Dyes

The pH of dye solutions plays a significant role in membrane filtration, as it can affect both the charge of the dye molecules and the ceramic membrane surface. In this study, the initial pH of the dye solutions was slightly acidic to neutral. Malachite green showed a pH around 6.5, while methylene blue were close to pH 6.8–7. These values remained relatively stable during the filtration process, indicating that no major chemical changes occurred. This stability supports the reliability of the retention results obtained.

1- Malachite green :

Monitoring pH helps determine whether chemical interactions occur between the membrane and malachite green during treatment. The following graphs (IV.7 & IV.8) show how the pH of malachite green solutions changes during filtration at two concentrations.

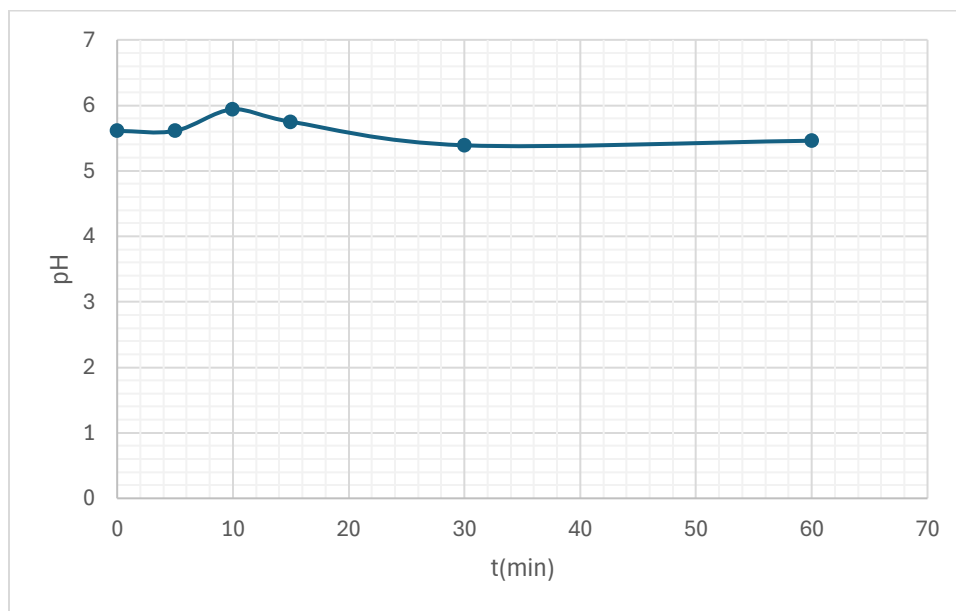


Figure 0.7 pH of Malachite green 10^{-4} as a Function of Time.

The pH of the malachite green solution at 10^{-4} M shows slight variation during filtration. These changes might result from interactions between the dye and membrane, or minor chemical transformations. However, the overall stability suggests that no strong acidic or basic compounds are being formed.

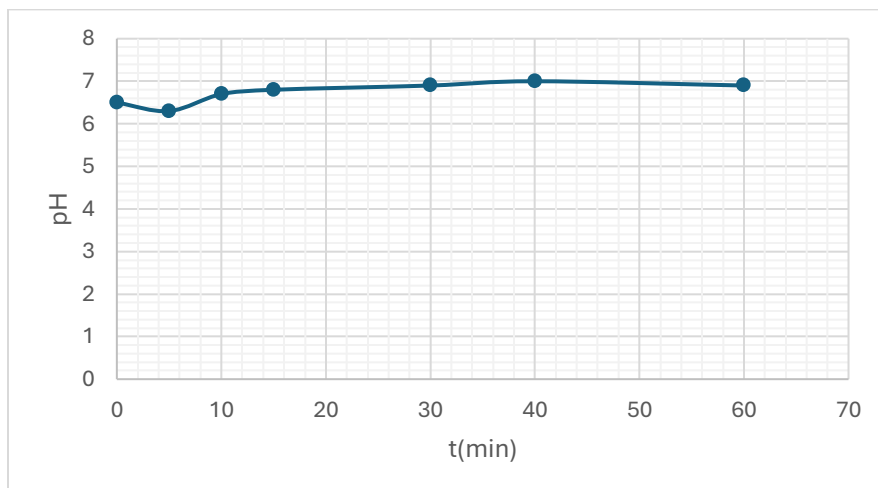


Figure 0.8 pH of Malachite green 10^{-5} as a Function of Time.

At the lower concentration, the pH remains fairly constant over time. This indicates that the filtration process does not significantly alter the chemical balance of the solution, reinforcing the idea that the membrane is primarily acting through physical rather than chemical mechanisms.

2- Methylene blue :

The pH monitoring of methylene blue solutions during filtration allows us to observe whether any acid-base imbalance occurs due to membrane interaction. The graphs below (IV.9 & IV.10) show pH variation throughout the process.

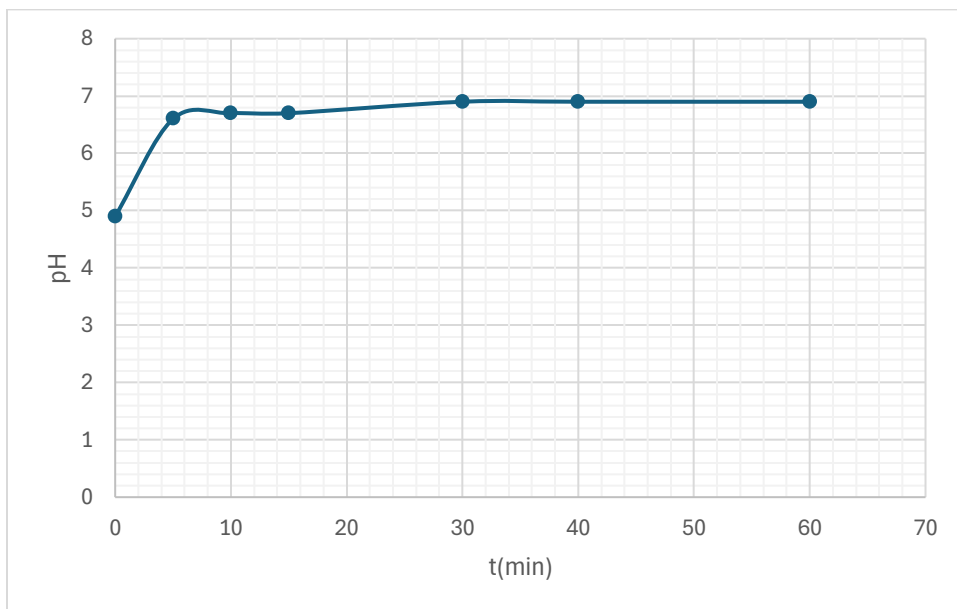


Figure 0.9 pH of Methylene blue 10^{-4} as a Function of Time.

In this case, a minor drop in pH is observed, which could suggest slight acidification due to dye-membrane interaction or gradual breakdown of the dye. Nonetheless, the variation is limited, and the solution remains chemically stable overall.

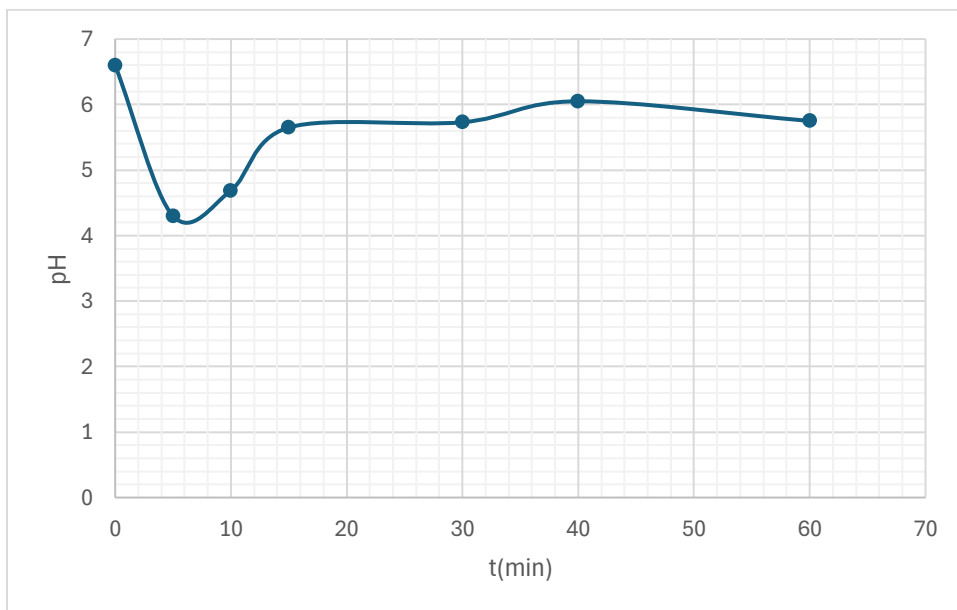


Figure 0.10 pH of Methylene blue 10^{-5} as a Function of Time.

As with malachite green, the 10^{-5} M methylene blue solution maintains a relatively stable pH. This supports the idea that filtration at low concentrations does not significantly disrupt the solution's acidity, pointing to gentle and selective retention by the membrane.

1.3. Conductivity of the dyes :

The conductivity of dye solutions reflects the presence of ions and dissolved salts, which can influence membrane performance during filtration. In this study, the dye solutions such as malachite green and methylene blue exhibited moderate conductivity levels due to their ionic nature. These values varied slightly depending on the dye concentration and purity but generally remained within a range that did not cause significant membrane fouling. Monitoring conductivity before and after filtration helped confirm the retention of charged dye molecules and the reduction of ionic content in the permeate.

1- Malachite green :

Conductivity reflects the total ionic content in the solution. A decreasing trend during filtration indicates the membrane's ability to retain charged species like dye ions. The graphs below (IV.11& IV12) show conductivity evolution for malachite green over time.

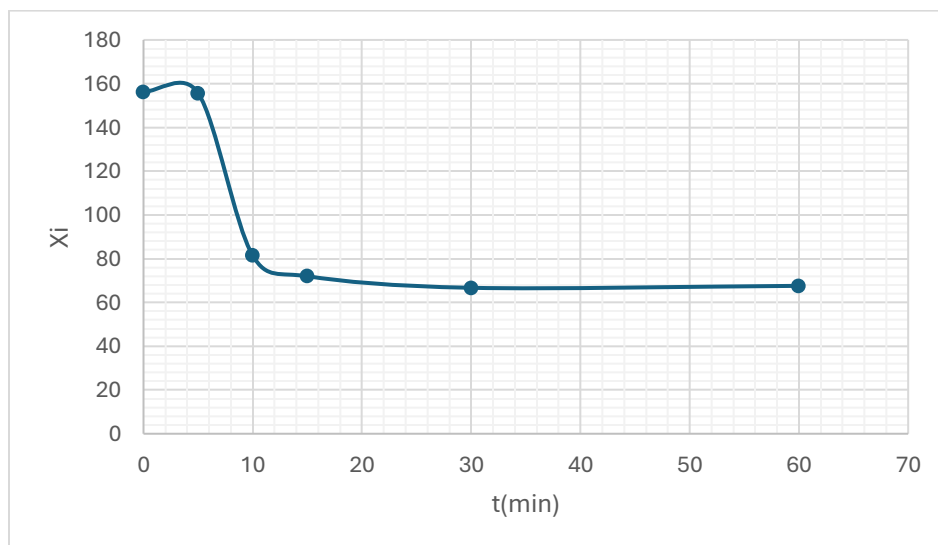


Figure 0.11 Conductivity of Malachite green 10^{-4} as a function of time

The gradual drop in conductivity suggests that ions associated with the dye are being retained by the membrane. This is a good indicator of the membrane's effectiveness in

removing charged species from the solution, likely through a combination of physical filtration and electrostatic interactions.

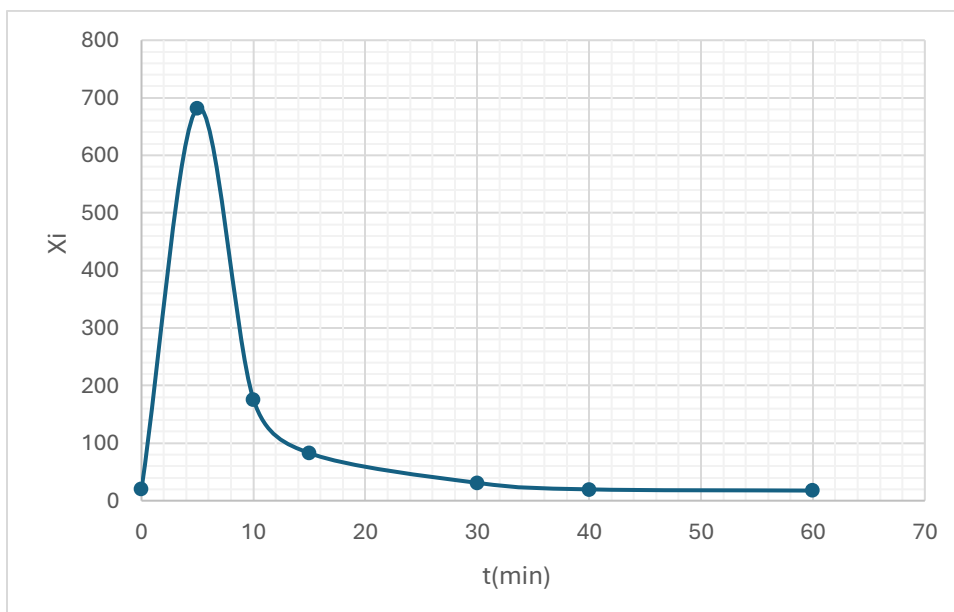


Figure 0.12 Conductivity of Malachite green 10^{-5} as a function of time

Although the change in conductivity is less pronounced than at higher concentration, it still decreases over time. This shows that even in more diluted conditions, the membrane continues to remove ionic compounds efficiently.

2- Methylene blue:

Conductivity changes during methylene blue filtration indicate ionic removal. A consistent decline suggests that both dye and related ions are being effectively retained. The figures below (IV.13 & IV.14) represent this variation.

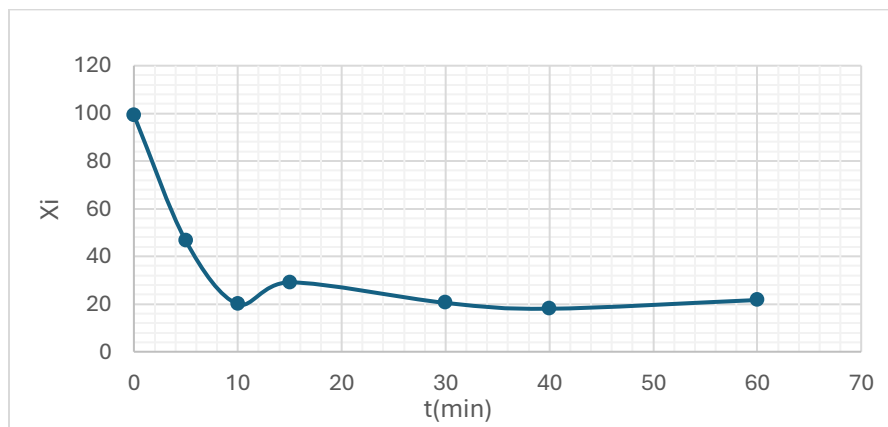


Figure 0.13 Conductivity of Methylene blue 10^{-4} as a function of time

Conductivity decreases consistently, which confirms the removal of methylene blue and its associated ions. This supports the results seen in the retention rate graph and reinforces the membrane's capacity to purify dye-contaminated solutions.

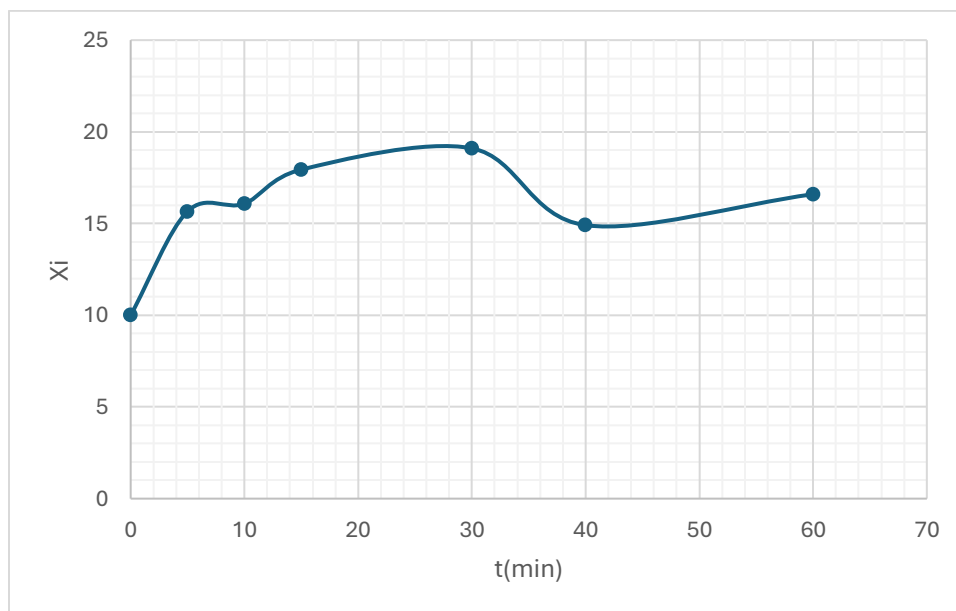


Figure 0.14 Conductivity of Methylene blue 10^{-5} as a function of time

As with the other tests, conductivity drops gradually, even at low dye concentration. This implies consistent membrane performance across a range of concentrations, making it suitable for treating both concentrated and diluted effluents.

1.4. Total Dissolved Solids of the dyes :

Total Dissolved Solids (TDS) represent the combined mass of all dissolved substances—including dye ions, counter-ions, and trace salts—present in the solution. In our experiments, initial TDS values for both malachite-green and methylene-blue feeds were moderate, reflecting their ionic character and the laboratory-grade salts used in preparation. During cross-flow filtration, TDS readings in the permeate dropped markedly, confirming that the ceramic membrane effectively retained not only the dye molecules but also associated dissolved species. This decline in TDS underscores the membrane's ability to improve overall water quality by simultaneously reducing colour and salinity.

1- Malachite green :

TDS indicates the amount of dissolved substances, including dye molecules and salts. Its reduction during filtration reveals how well the membrane removes solutes from malachite green solutions. The figures below (IV.15 & IV.16) present this variation at both tested concentrations.

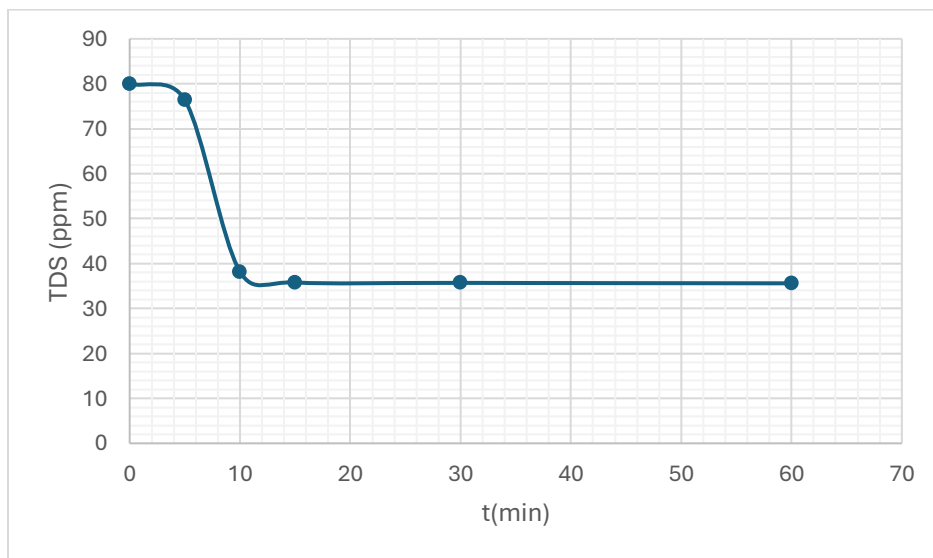


Figure 0.15 Total Dissolved Solids of Malachite green 10^{-4} as a function of time

The TDS decreases gradually over time, indicating that the membrane is effectively removing dissolved components associated with malachite green. This confirms its capability not only in dye retention but also in reducing overall salinity.

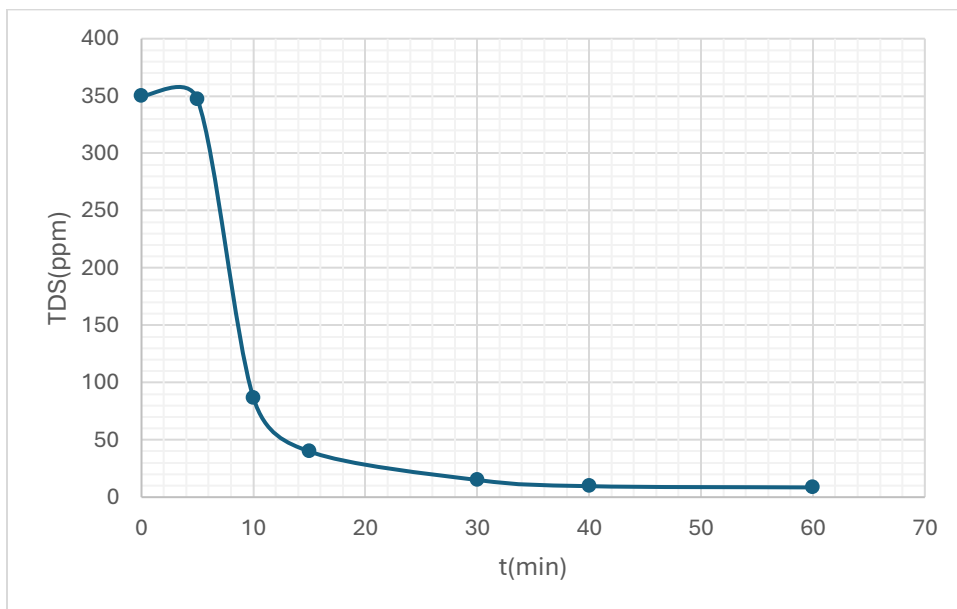


Figure 0.16 Total Dissolved Solids of Malachite green 10^{-5} as a function of time

At the lower concentration, a similar but less pronounced decrease is observed. This shows that the membrane remains effective in treating even diluted dye solutions, although the absolute amount of dissolved solids is smaller

2- Methylene blue :

TDS reduction demonstrates how well the membrane filters out dissolved compounds associated with methylene blue. The following graphs (IV.17 & IV.18) present TDS variation over time for both tested concentrations.

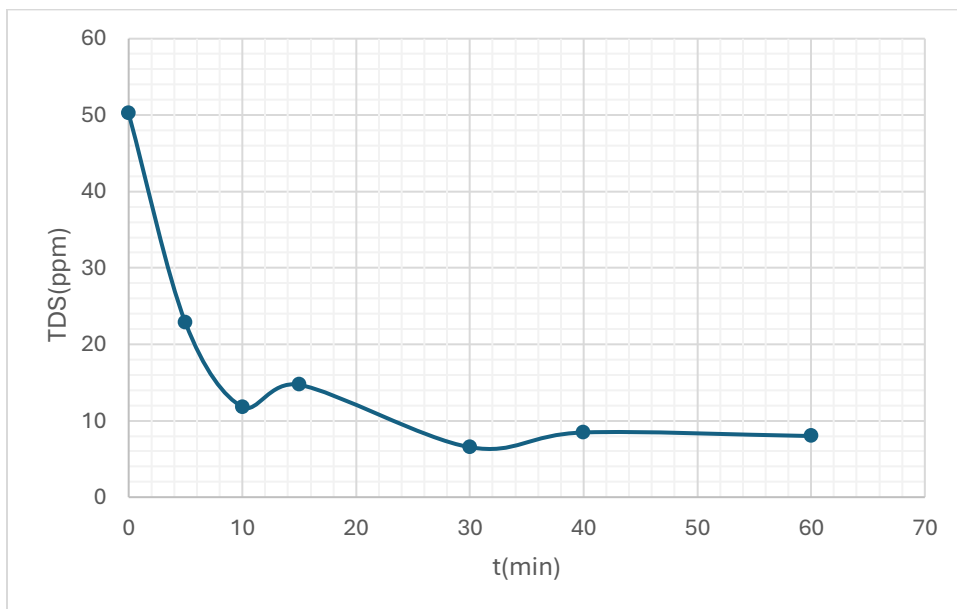


Figure 0.17 Total Dissolved Solids of Methylene blue 10^{-4} as a function of time

A steady reduction in TDS over time suggests that both the dye and its associated ions are successfully retained. This aligns with the conductivity results and highlights the membrane's filtration efficiency.

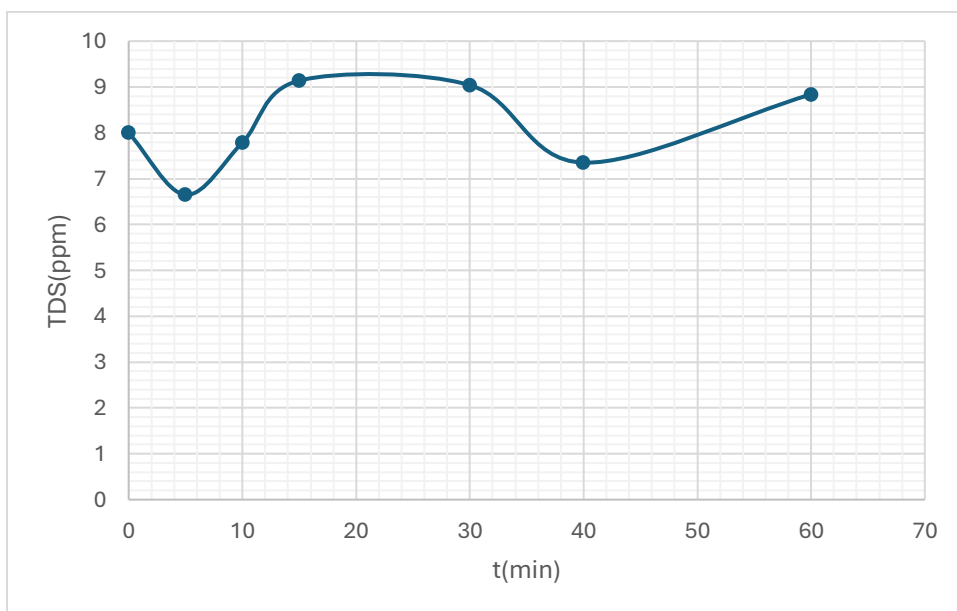


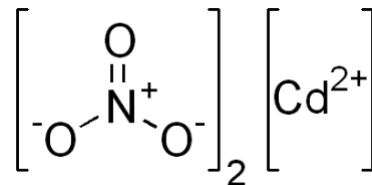
Figure 0.18 Total Dissolved Solids of Methylene blue 10^{-5} as a function of time

Even at this low concentration, the TDS shows a downward trend, supporting the membrane's strong capacity for treating dilute organic pollutant solutions.

2. Solid metals:

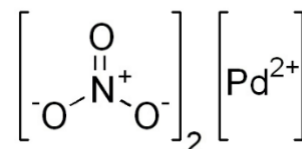
1- Cadmium Nitrate ($\text{Cd}(\text{NO}_3)_2$)

- **Structure type:** Ionic, inorganic salt
- **Molecular formula:** $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$
- **Molar mass:** 308.48 g/mol
- **Melting point:** 100 °C (decomposes on further heating)
- **Solubility:** Very soluble in water (~1330 g/L at 20 °C)
- **Properties:**
 - Toxic heavy metal salt
 - Used in pigment manufacturing, cadmium plating, and as a laboratory reagent
 - Forms Cd^{2+} ions in solution
- **Hazards:** Toxic, carcinogenic, and environmentally hazardous — requires careful handling and disposal.



2- Lead Nitrate ($\text{Pb}(\text{NO}_3)_2$)

- **Structure type:** Ionic, inorganic salt
- **Molecular formula:** $\text{Pb}(\text{NO}_3)_2$
- **Molar mass:** 331.2 g/mol
- **Melting point:** 470 °C (decomposes with emission of toxic fumes)
- **Solubility:** Very soluble in water (~524 g/L at 20 °C)
- **Properties:**
 - Source of Pb^{2+} ions
 - Used in the production of matches, explosives, and heat-stabilizers
 - Strong oxidizing agent
- **Hazards:**
 - Highly toxic and neurotoxic
 - Can cause environmental damage to water and soil



- Must be handled with proper protective equipment and under controlled conditions

2.1. pH of the solid metals

The pH of heavy metal solutions is an important parameter influencing their solubility, ionic form, and interaction with the membrane surface. In this study, solutions containing metals such as cadmium, lead, copper, and iron were prepared in slightly acidic conditions, with pH values ranging between 5.0 and 6.5. These values help maintain the metals in a soluble ionic state, preventing precipitation during filtration. The pH remained relatively stable throughout the process, ensuring accurate evaluation of membrane performance in retaining metal ions.

1- Cadmium Nitrate ($\text{Cd}(\text{NO}_3)_2$) :

Monitoring pH during filtration of cadmium nitrate helps assess whether hydrolysis or other reactions occur in the presence of the membrane. The figures below (IV.19 & IV.20) show pH changes for 10^{-4} M and 10^{-5} M cadmium solutions.

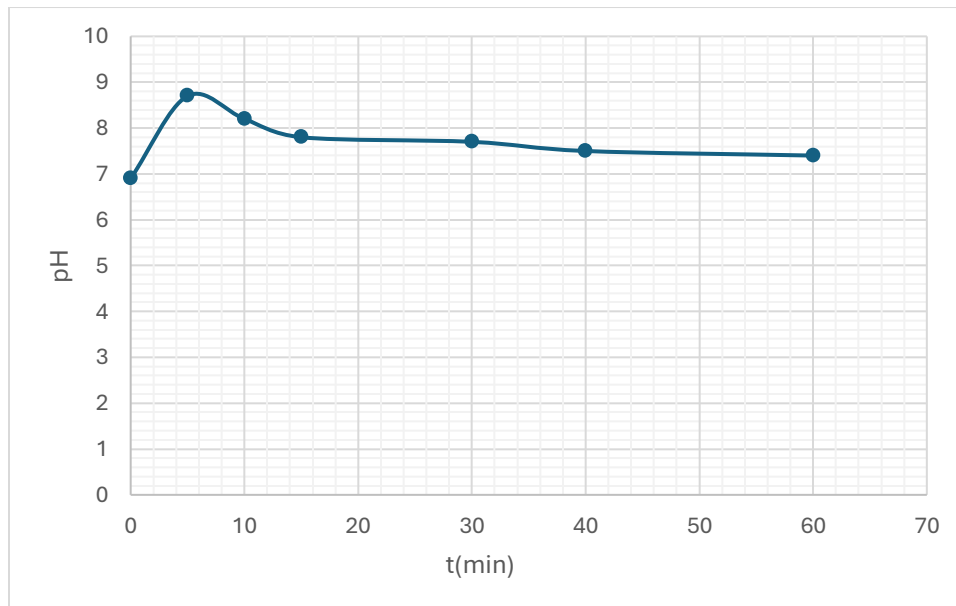


Figure 0.19 pH of Cadmium Nitrate 10^{-4} as a function of time

A slight drop in pH is observed, potentially due to hydrolysis of cadmium ions or interaction with the membrane surface. The overall pH remains acidic and stable.

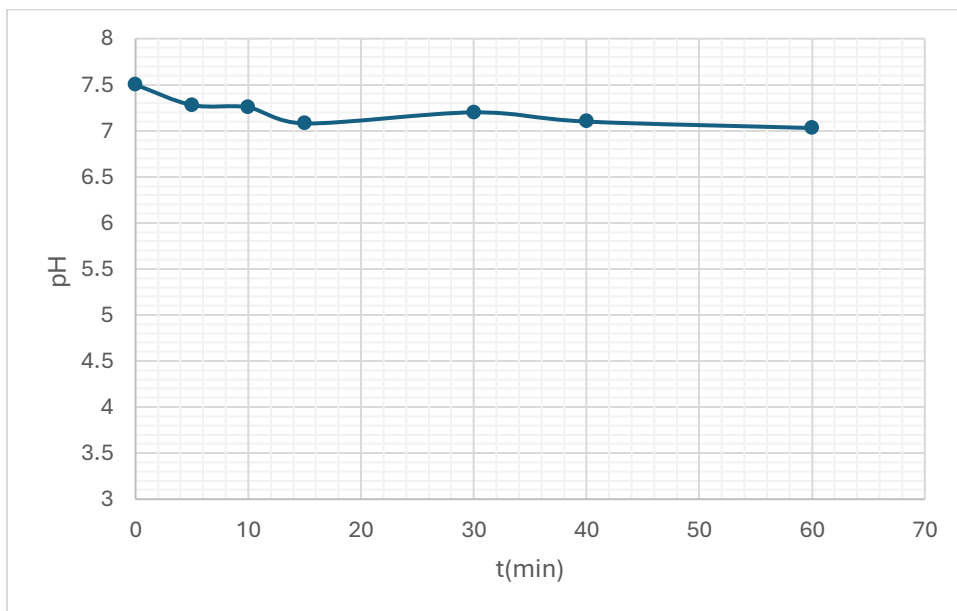


Figure 0.20 pH of Cadmium Nitrate 10^{-5} as a function of time

pH remains more stable, likely due to lower ionic strength. This reflects that less cadmium is present, reducing chemical changes in the solution.

2- Lead Nitrate ($\text{Pb}(\text{NO}_3)_2$) :

Tracking pH for lead nitrate solutions during filtration provides insight into the chemical stability of the solution. Any shifts may be due to partial hydrolysis of Pb^{2+} ions or membrane interaction. The figures below (IV.21-IV.22) display these variations.

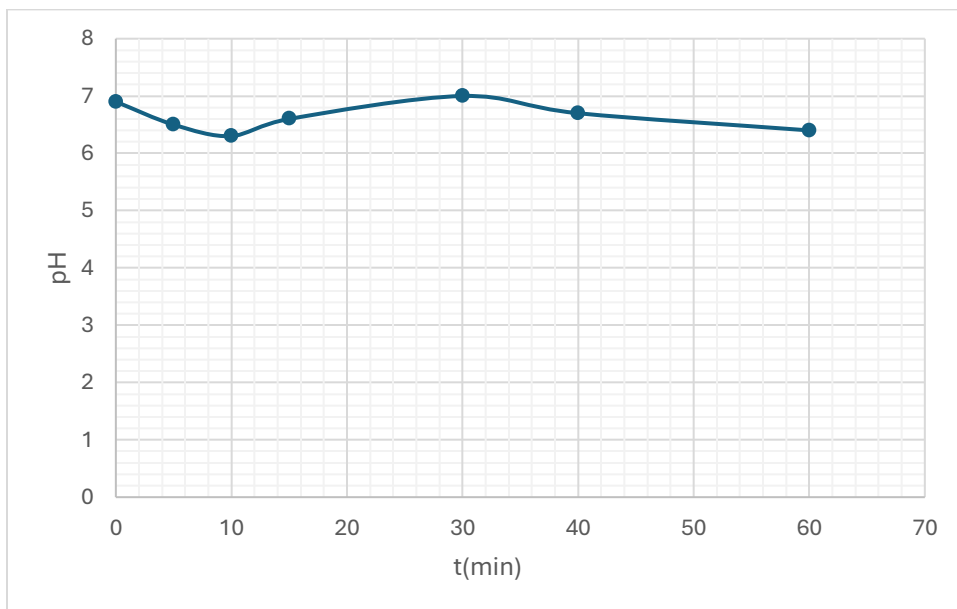


Figure 0.21 pH of Lead Nitrate 10^{-4} as a function of time

A gradual pH decline may result from partial hydrolysis of Pb^{2+} ions. This could indicate membrane interaction or mild precipitation.

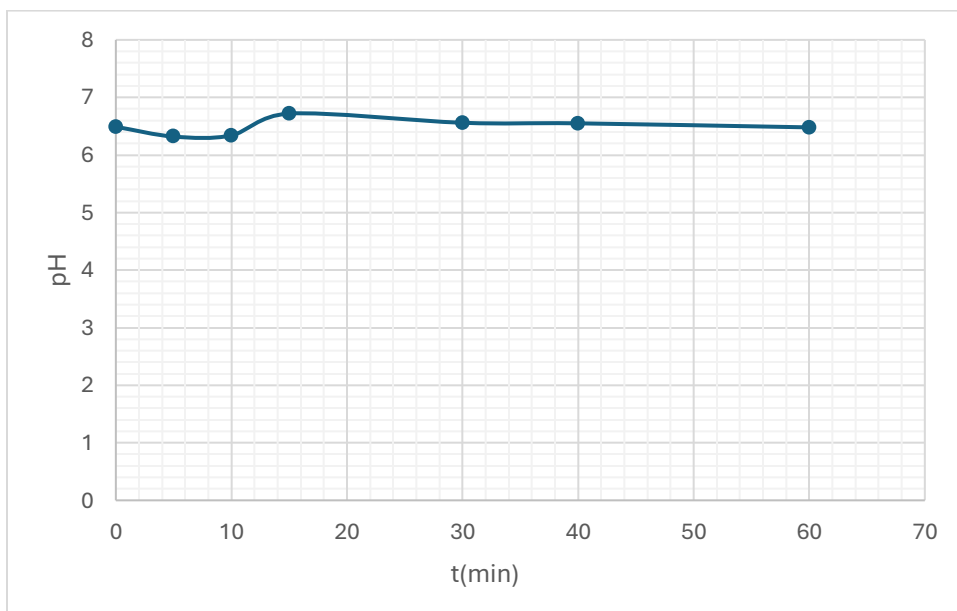


Figure 0.22 pH of Lead Nitrate 10^{-5} as a function of time

The pH remains relatively stable, with only minor fluctuations, suggesting chemical equilibrium in dilute solutions.

2.2. Conductivity of the solid metals

The conductivity of heavy metal solutions reflects the concentration of dissolved metal ions and their associated anions. In this work, solutions containing metals such as cadmium, lead, copper, and iron showed relatively high conductivity values due to the presence of free ions in solution. This parameter is crucial, as it helps assess the ionic strength of the feed before and after filtration. A noticeable decrease in conductivity after membrane treatment confirmed the effective removal of metal ions by the ceramic membrane, indicating its strong separation capability.

1. Cadmium Nitrate ($\text{Cd}(\text{NO}_3)_2$) :

Conductivity indicates the presence of cadmium and nitrate ions. A decrease during filtration reflects ion retention by the membrane. The following graphs (IV.23 & IV.24) present conductivity variation over time.

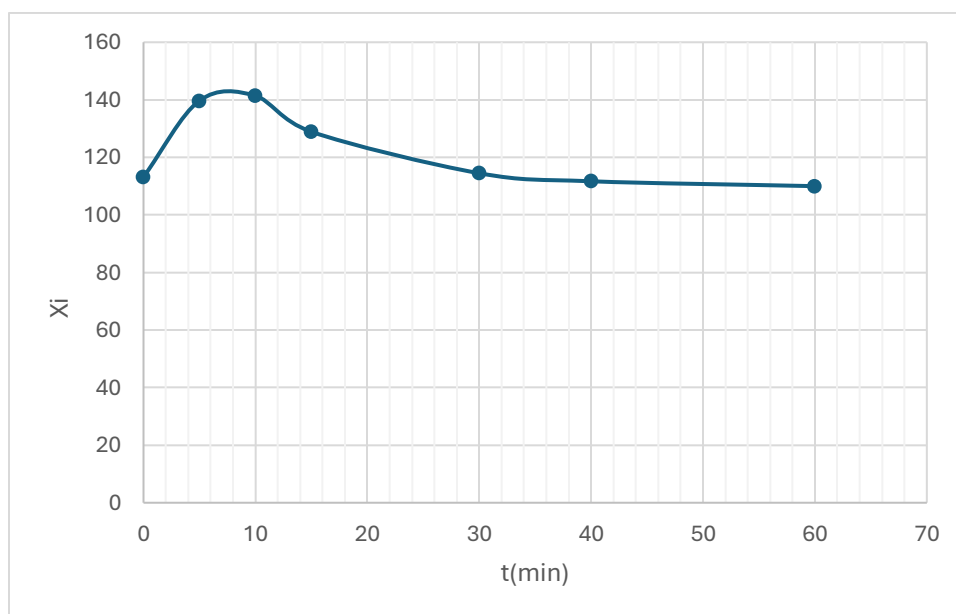


Figure 0.23 Conductivity of Cadmium Nitrate 10^{-4} as a function of time

Conductivity drops consistently, indicating ion removal by the membrane. The retention of Cd^{2+} and NO_3^- contributes to this reduction.

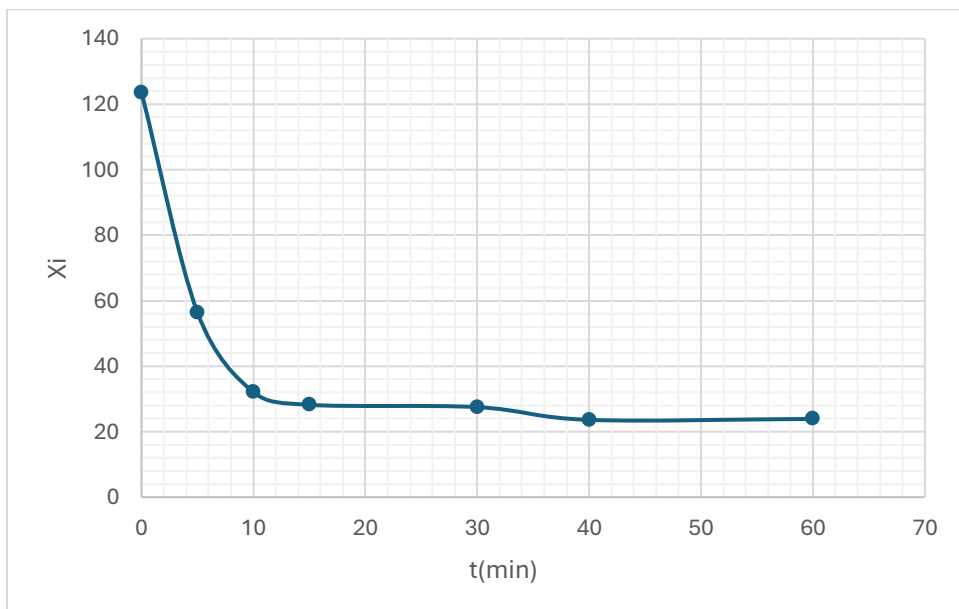


Figure 0.24 Conductivity of Cadmium Nitrate 10^{-5} as a function of time

The pattern is similar but less steep, suggesting that even at low concentrations, cadmium ions are effectively retained.

2. Lead Nitrate ($\text{Pb}(\text{NO}_3)_2$) :

The conductivity of lead nitrate solutions drops as ions are retained. This indicates successful filtration of Pb^{2+} and NO_3^- . The graphs below (IV.25 & IV.26) show this behavior over time at two concentrations.

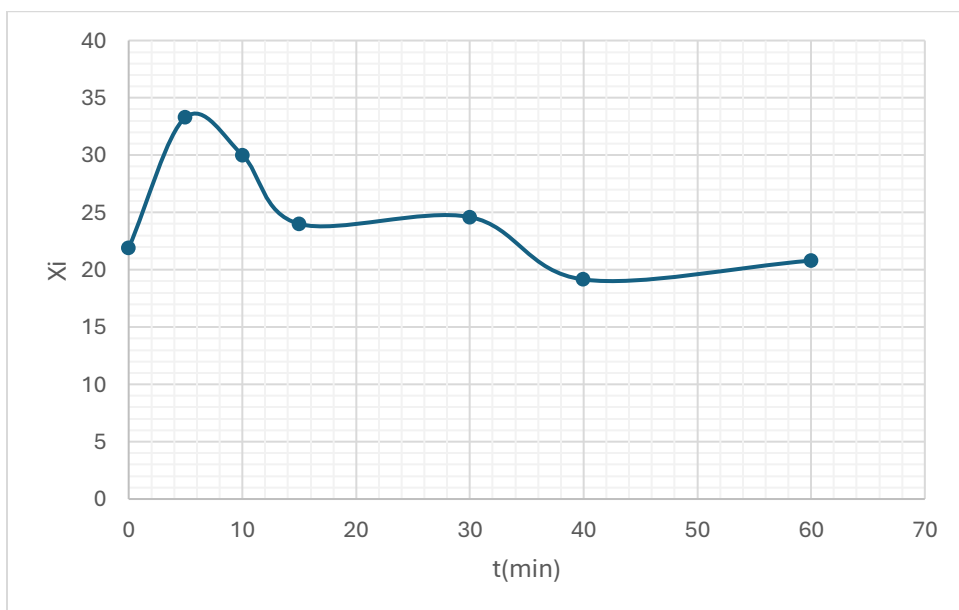


Figure 0.25 Conductivity of Lead Nitrate 10^{-4} as a function of time

There is a notable decline in conductivity, reflecting the removal of Pb^{2+} and associated ions. This shows strong filtration performance.

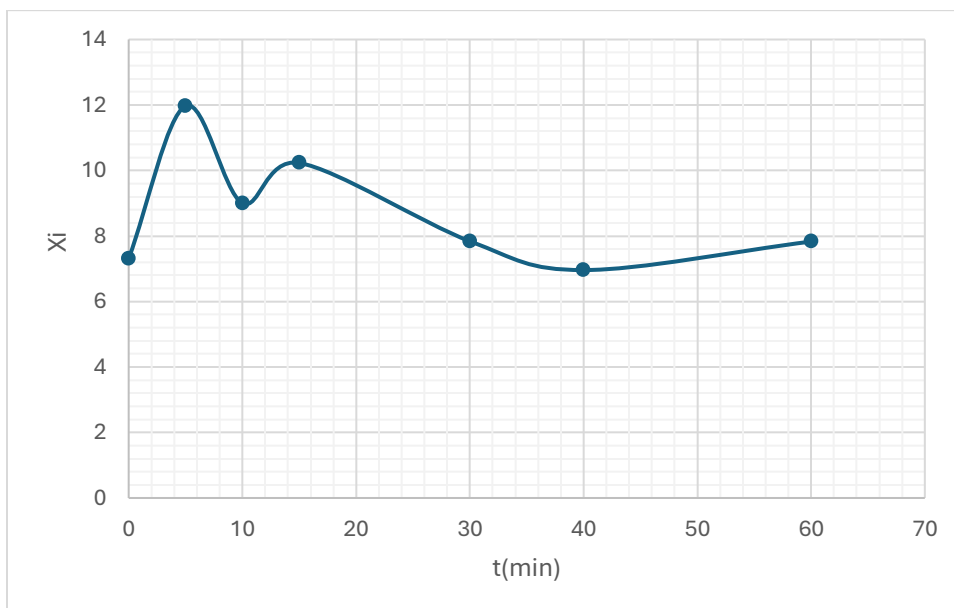


Figure 0.26 Conductivity of Lead Nitrate 10^{-5} as a function of time

The downward trend confirms consistent removal at low concentrations, highlighting the membrane's versatility.

2.3. Total Dissolved Solids of the solid metals

Total Dissolved Solids (TDS) in heavy metal solutions represent the combined concentration of all ionic and molecular species present, including metal cations and their counter-ions. In this study, metal salt solutions such as those of cadmium, lead, copper, and iron exhibited high TDS values, reflecting their strong ionic content. After filtration through the ceramic membrane, a significant reduction in TDS was observed, indicating efficient removal of dissolved metal species. This confirms the membrane's potential not only for metal ion retention but also for overall improvement of water quality.

1-Cadmium Nitrate ($\text{Cd}(\text{NO}_3)_2$) :

TDS reflects the total dissolved content, including cadmium ions and nitrates. Its decline over time demonstrates the membrane's effectiveness in filtering out these species. The figures below (IV.27 & IV.28) illustrate TDS evolution for cadmium nitrate solutions.

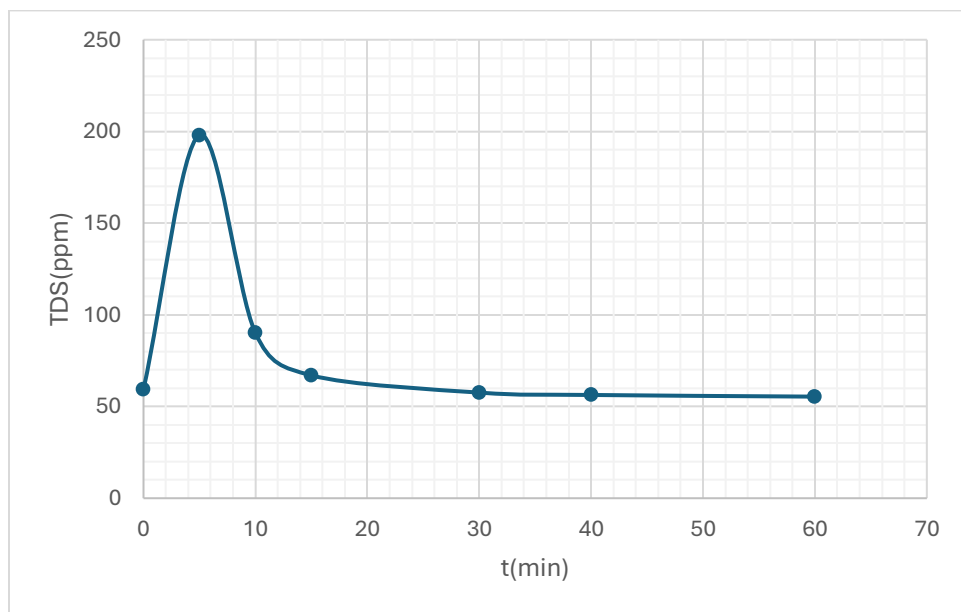


Figure 0.27 Total Dissolved Solids of Cadmium Nitrate 10^{-4} as a function of time

TDS steadily decreases, confirming the membrane's efficiency in removing dissolved cadmium nitrate species.

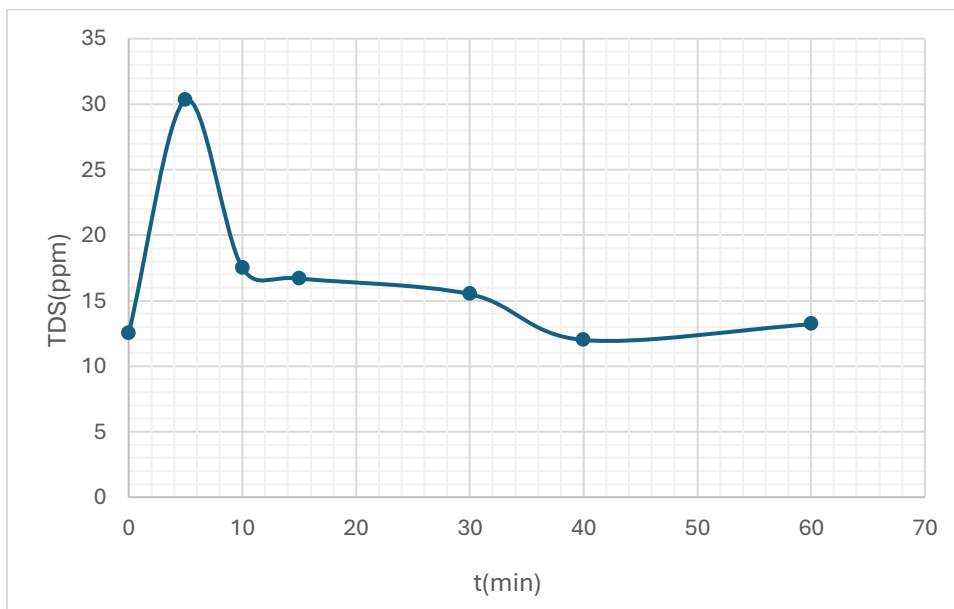


Figure 0.28 Total Dissolved Solids of Cadmium Nitrate 10^{-5} as a function of time

A smaller but noticeable decrease suggests good performance at low concentrations.

2- Lead Nitrate ($\text{Pb}(\text{NO}_3)_2$) :

A decrease in TDS confirms the removal of lead nitrate components from the solution. The following figures (IV.29 & IV.30) present how TDS varies with time during the filtration of both concentrated and diluted solutions.

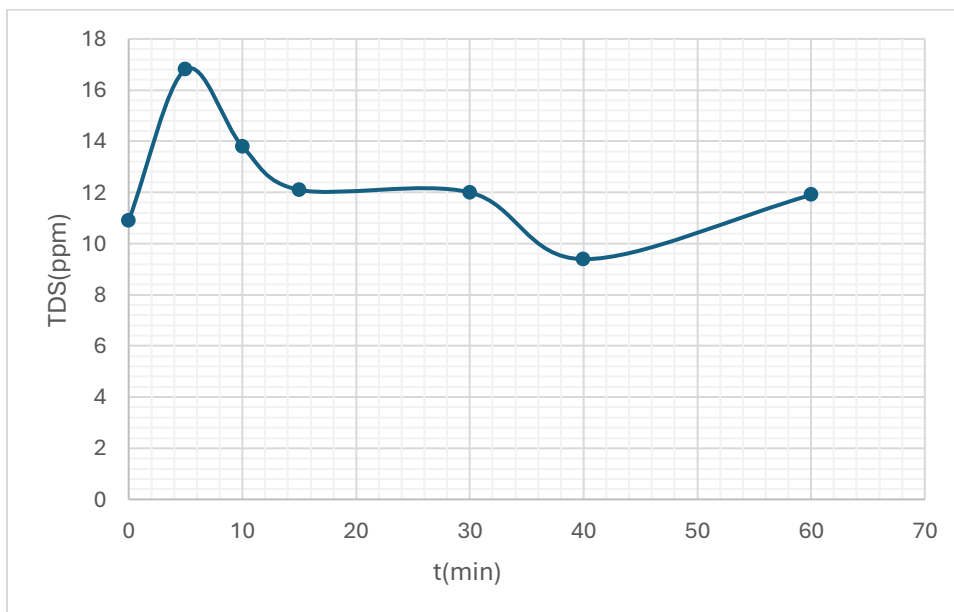


Figure 0.29 Total Dissolved Solids of Lead Nitrate 10^{-4} as a function of time

A clear reduction in TDS shows the membrane's ability to retain lead and associated anions effectively.

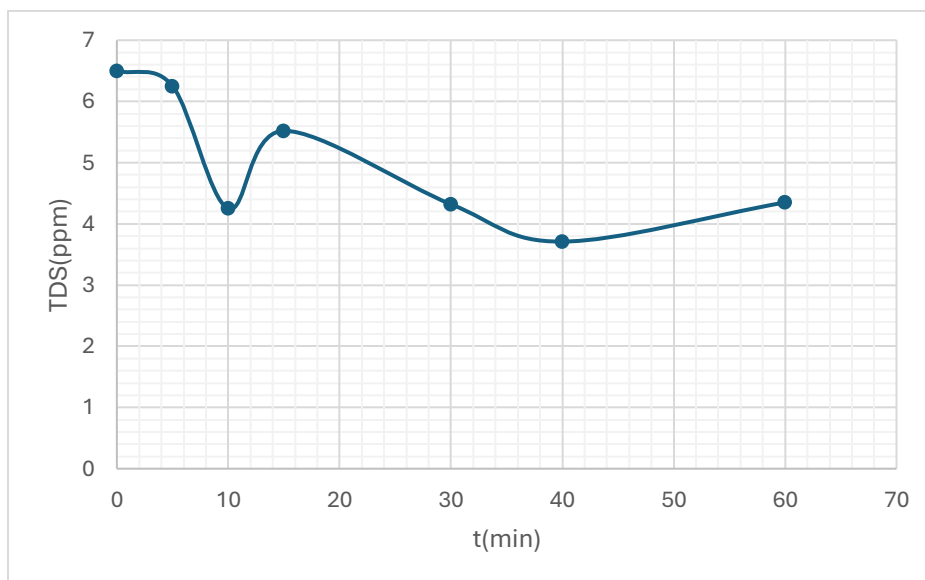


Figure 0.30 Total Dissolved Solids of Lead Nitrate 10^{-5} as a function of time

A gradual decrease further demonstrates reliable performance for diluted metal solutions.

3. Wastewater from the Tayal factory

3.1. pH of the TAYAL wastewater:

The pH of the Tayal textile factory wastewater was monitored to observe any changes caused by the membrane during real effluent treatment. The figure below shows pH stability over time, reflecting the membrane's impact on complex industrial water.

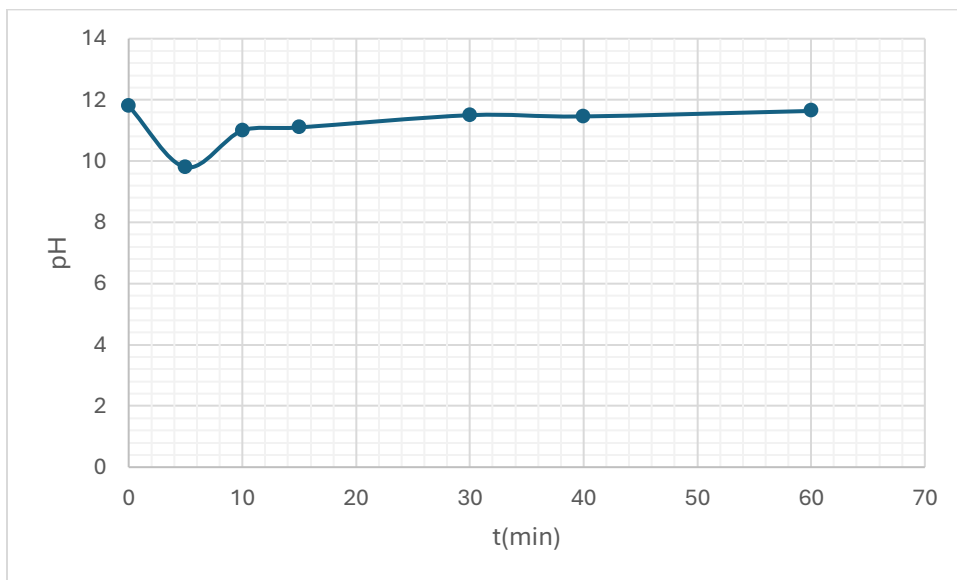


Figure 0.31 pH of the TAYAL wastewater as a function of time

The pH profile of the Tayal wastewater shows relatively stable behavior over time, with only minor fluctuations during the filtration process. This stability implies that the ceramic membrane does not significantly alter the acid-base balance of the effluent. It also suggests that the wastewater lacks strong acidic or basic species that would otherwise shift pH drastically. The membrane likely acts through physical separation and adsorption, rather than any strong chemical interaction that could affect pH. This result supports the membrane's potential for pre-treatment or polishing steps in textile or industrial wastewater applications.

Conclusion

This study explored the potential of using ceramic membranes fabricated from local materials as an effective and sustainable solution for treating various types of liquid effluents. Through a comprehensive investigation encompassing synthetic dyes (Malachite Green and Methylene Blue), heavy metal solutions (Cadmium and Lead), and real wastewater from the Tayal textile factory, the results clearly demonstrated the capability of the developed ceramic membranes to significantly reduce pollutants.

The membranes exhibited excellent performance in terms of dye and heavy metal retention, with notable improvements in water quality parameters such as pH stability, reduced conductivity, and lower total dissolved solids (TDS). The results also highlighted the advantages of crossflow filtration, which minimized membrane fouling and maintained consistent filtration efficiency over time.

Furthermore, the use of natural raw materials for membrane fabrication represents a cost-effective and environmentally friendly alternative to conventional commercial membranes. These findings support the potential integration of ceramic membrane technology into industrial wastewater treatment systems, particularly in regions with access to suitable clays and limited treatment infrastructure.

Overall, this work contributes to the development of innovative and accessible water treatment technologies and underscores the importance of combining material science with environmental engineering to address global water pollution challenges.

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الجمهورية الجزائرية الديمقراطية الشعبية
وزارة التعليم العالي والبحث العلمي

جامعة عبد الحميد بن باديس - مستغانم -

كلية العلوم الدقيقة والإعلام الآلي

تصريح شرفي خاص بالالتزام بقواعد النزاهة العلمية
لإنجاز البحث

أنا الممضي أدناه،

الطالب(ة): بلوان صروان رقم التسجيل الجامعي: 202037029820

الحامل لبطاقة التعريف الوطنية رقم: 422375066 والصادرة بتاريخ:

عن

المسجل بكلية العلوم الدقيقة والإعلام الآلي / قسم Master 2 chimie appl.

شعبة / التخصص chimie appliquée

والمكلف بإنجاز مذكرة ماستر بعنوان:

Application d'un nouveau liant dans le traitement
des effluents liquides

أصرح بشرفي أنني ألتزم بمراعاة المعايير العلمية والمنهجية ومعايير الأخلاقيات العلمية والنزاهة الأكاديمية
المطلوبة في إنجاز البحث ، وأتحمل المسؤولية الشخصية عن كل المحتوى المتضمن في البحث المذكور أعلاه .

التاريخ: 21/06/2025

إمضاء المعني



* ملحق القرار الوزاري رقم 933 المؤرخ في 28 جويلية 2016 الذي يحدد القواعد المتعلقة بالوقاية من السرقة العلمية ومكافحتها.

الجمهورية الجزائرية الديمقراطية الشعبية
وزارة التعليم العالي والبحث العلمي

جامعة عبد الحميد بن باديس - مستغانم -

كلية العلوم الدقيقة والإعلام الآلي

تصريح شرفي خاص بالالتزام بقواعد النزاهة العلمية
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عن مستغانم

المسجل بكلية العلوم الدقيقة والإعلام الآلي / قسم الكيمياء

شعبة الكيمياء / التخصص كيمياء تحليلية

والمكلف بإنجاز مذكرة ماستر بعنوان:

The application of a new binder in
the treatment of liquid effluents

أصرح بشرفي أنني ألتزم بمراعاة المعايير العلمية والمنهجية ومعايير الأخلاقيات العلمية والنزاهة الأكاديمية
المطلوبة في إنجاز البحث ، وأتحمل المسؤولية الشخصية عن كل المحتوى المتضمن في البحث المذكور أعلاه

التاريخ: 18-06-2025

إمضاء المعني

Abdenour

* ملحق القرار الوزاري رقم 933 المؤرخ في 28 جويلية 2016 الذي يحدد القواعد المتعلقة بالوقاية من السرقة العلمية ومكافحتها.