Faculty of Engineering

HEAVY METAL REMOVAL USING MIXED MATRIX MEMBRANE INCORPORATED WITH MONTMORILLONITE

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HEAVY METAL REMOVAL USING MIXED MATRIX MEMBRANE INCORPORATED WITH MONTMORILLONITE

JUSTINA LUISA ANAK JANIS

A dissertation submitted in partial fulfilment

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ABSTRACT

The removal of heavy metal by using mixed matrix membrane (MMM) incorporated with montmorillonite (MMT) was studied. In general, MMM referred to membrane that consists of polymers filled with inorganic material in order to enhance the membrane's chemical and physical properties particularly in water separation application. In this study, MMM was prepared using polysulfone (PSf) polymer incorporated with MMT and casted using phase inversion technique. The prepared membrane were characterized using Fourier Transform Infrared (FTIR) to analyze the functional groups, Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) to observe the membrane cross section morphology and conduct elemental analysis, and water uptake analysis to know the membrane capability to absorb water. Then, the performance of prepared membranes were evaluated in terms pure water flux and capability of the membrane to remove heavy metal in wastewater. Based on the membrane performance, 1.0MMM was the most preferred as it has significant rejection of cadmium and lead as well as high pure water flux, surface porosity and permeate flux.

Keywords: *mixed matrix membrane, montmorillonite (MMT), heavy metal removal.*

ABSTRAK

Penyingkiran logam berat dengan menggunakan membran matriks campuran (MMM) yang digabungkan dengan montmorilonit (MMT) telah dikaji. Secara umum, MMM merujuk kepada membran yang terdiri daripada polimer yang diisi dengan bahan anorganik untuk meningkatkan sifat kimia dan fizikal membran terutama dalam aplikasi pemisahan air. Dalam kajian ini, MMM akan disiapkan menggunakan polimer polisulfon (PSf) yang digabungkan dengan MMT dan dilemparkan menggunakan teknik penyongsangan fasa. Membran yang disediakan dicirikan menggunakan Inframerah Fourier Transformasi (FTIR) untuk menganalisis kumpulan berfungsi, Mengimbas Mikroskopi Elektron-Sinar Penyebaran Tenaga (SEM-EDX) untuk memerhatikan morfologi keratan rentas membran dan melakukan analisis unsur, dan analisis pengambilan air untuk mengetahui kemampuan membran untuk menyerap air. Akhir sekali, prestasi membran yang disiapkan dinilai dari segi aliran air tulen dan keupayaan membran untuk membuang logam berat dalam air sisa. Berdasarkan prestasi membran, 1.0MMM adalah yang paling dikehendaki kerana mempunyai penolakan kadmium dan plumbum yang ketara serta mempunyai fluks air tulen, keliangan permukaan dan fluks meresap yang tinggi.

Kata kunci: *membrane matriks campuran, montmorilonit, penyingkiran logam berat.*

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Nowadays, water was regarded as one of the basic necessities required for urbanization and industrialization. Water was necessary for the development of cities since it is required for residents' drinking and sanitation needs in order to build and maintain a healthy environment. On the other hand, water was used in the industrialization process for a variety of purposes, including heating, cooling, dilution, and many more. Nonetheless, a growing problem arises when wastewater was not properly treated, resulting in water pollution. There were several water pollutants that present in water bodies such as nutrients, halogen, heavy metals, organic pollutants, and microbial pollutants (Madhav *et al*., 2020). The focus of this research project was on water pollutants, specifically heavy metals. Heavy metals were one of the toxic and hazardous substances that, when present in high concentrations, pose a concern to human health. According to Qasem *et al*., heavy metals were considered as non-biodegradable material. Non-biodegradable materials remain in water bodies for a long period of time because they were resistant to microbial and chemical degradation (Qasem *et al*., 2021). Moreover, heavy metals were highly soluble in water. Hence, heavy metals accumulated in food chains, providing major health concerns to living organisms that consume water with high heavy metal concentrations. Based on the study by Muharrem & Ince, heavy metals were primarily found in wastewater from modern industrial sources such as mining, metal processing factories, protective coatings, chemical manufacturing, electrolysis, tannery, metalworking, fuel source, pulp, and the manufacture of various polymers (Muharrem & Ince, 2017). As a result, heavy metal processing industries play an important role in treating heavy metals before discharging them into water bodies.

Most manufacturing operations generate wastewater as an inevitable by-product. It was critical to have effective wastewater treatment so that freshwater resources and water supply can be augmented and made available to living organisms. This also helps to alleviate water scarcity. As stated by Crini & Lichtfouse, flotation, precipitation, oxidation, solvent extraction, evaporation, carbon adsorption, ion exchange, membrane filtration, electrochemistry, biodegradation, and phytoremediation are some of the technologies used in wastewater treatment (Crini & Lichtfouse, 2019). Membrane materials can be classed as organic, inorganic, or a combination of both. Organic membranes were created from synthetic organic polymers meanwhile inorganic membranes are usually from ceramics, metals, clay minerals, zeolites or silica (Obotey Ezugbe & Rathilal, 2020). Membrane technology in wastewater treatment had various advantages, including low capital cost, reduced equipment size, low energy requirements, minimal chemical usage, easy accessibility, and environmental friendliness. Hence, membrane technology has lately been demonstrated as a more viable option in wastewater treatment.

Organic membranes, commonly known as polymeric membranes were extensively used in the water separation field because they are cost-effective and versatile. These membranes can be customised to meet the unique requirements of the process in which they were utilised. Thus, selective separation was possible when utilizing the polymeric membranes. Based on the literature of Sonawane *et al*., the most significant property required in polymeric membranes was affinity for a certain component (Sonawane *et al*., 2021). A suitable selection of polymeric membrane for a certain function is critical. This was because the polymer must have the right affinity and be able to tolerate the separation environment (Dickhout *et al.*, 2017). Some examples of polymeric membranes were polysulfone (PSf), polyethersulfone (PES), cellulose acetate (CA), polyvinylidene fluoride (PVDF) and many more. A various characteristics including dense and porous polymeric membranes can be executed depending on the requirement. It was also trivial to regulate the pore size of polymeric membranes during their synthesis (Sonawane *et al*., 2021). This indicates that polymeric membranes were highly flexible, and that the polymeric membranes may be synthesised in a small amount of space. According to Ladewig & Al-Shaeli, the key advantages of polymeric membranes were their ease of preparation, low cost, lower energy requirements, flexibility in membrane layout, and relatively low working temperature, which is also connected with less rigorous material requirements in module assembly (Ladewig & Al-Shaeli, 2017). Therefore, the application of polymeric membranes can be employed for the removal of heavy metal in water separation mechanism.

Montmorillonite (MMT) belongs to smectite group that was one of the most commonly used clay minerals. It derives from the 2:1 clay layer structural family, which consists of two fused siloxane tetrahedral sheets that share edges with an octahedral sheet in the middle (Bee *et al*., 2018). The octahedral sheet was made from either aluminium or magnesium hydroxide. According to Liu *et al*., MMT was obtained through mineralization of ores in which warm and humid environments are the main factors of the process (Liu *et al*., 2019). MMT can be employed as a pollutants natural scavenger through isomorphic substitution. Processes such as ion exchange, adsorption, dispersion, expansibility, and suspension were used in isomorphic substitution to uptake cations and anions (Liu *et al*., 2021). In general, negative layer charges of magnesium ions and aluminium ions were obtained in the alumina octahedral and silicon tetrahedral respectively, results from the isomorphic substitution. The negative layer charges of magnesium ions are for aluminium ions meanwhile the negative layer charges of aluminium ions are for silicon ions. Due to the presence of numerous active sites including Bronsted and Lewis acid sites on MMT's surfaces, MMT was known to be a good adsorbents. To maintain the equilibrium of metal ions between the layers, certain substitutable cations can be exchanged by other cations. Aside from that, the Van der Waals force and electrostatic force connect the MMT interlayer to the nanosheets that hold the MMT particles together. Lastly, because of its low cost, non-hazardous, and vast deposits, MMT was particularly appealing as an inorganic membrane.

1.2 Problem statement

Membrane fouling was one of the most significant issues associated to the usage of polymeric membranes in wastewater treatment. This was owing to the hydrophobic nature of the polymeric membrane, which allows foulants to adhere to the membrane's surface. Based on the literature of AlSawaftah *et al*., the membrane foulants include particulate, organic, inorganic, and biological microorganisms (AlSawaftah *et al*., 2021). Consequently, membrane fouling cause a reduction of membrane water flux (Hebbar *et al*., 2018). Membrane water flux refers to the total water flow across the membrane. As the membrane's surface was blocked by the deposition of foulants, only a limited amount of water may pass through the membrane. This results in increasing of operational cost and the lifespan of the polymeric membrane is reduced. To address this problem, the fabrication of a polymeric membrane with the hydrophilic nature of an inorganic membrane is required to improve the polymeric membrane's performance.

Aside from that, polymeric membrane had a trade-off relationship between permeability and selectivity. In general, permeability relates to the rate at which water passes through a membrane, whereas selectivity refers to the degree to which heavy metals were rejected from water. The capital cost of the polymeric membrane was decreased when higher permeability was obtained. This was because the amount of membrane area required to treat heavy metals in water is reduced. Conversely, higher selectivity results in higher purity of water as the membrane was impermeable to heavy metals and separates them from the water. High permeability and selectivity polymeric membranes were desirable. Nevertheless, more permeable polymeric membranes were usually found to be less selective, and vice versa (Cheng *et al*., 2018). This was attributable to the porous membranes' wide distribution of pores and the nonspecific interactions of heavy metals with polymeric membranes. According to Kheirieh *et al*., porosity and hydrophilicity were the most essential parameters that determine the permeability capabilities of polymeric membrane (Kheirieh *et al*., 2018). The permeability of the polymeric membrane increased as the broad distribution of pores in the porous membrane grows, but the selectivity of heavy metals decreased. Therefore, MMT was a viable option to increase the number of small surface pores in the membrane.

As for inorganic membranes, these membranes plays an essential role in the membrane technology evolution. Unfortunately, inorganic membranes had a number of disadvantages, including brittleness, high production costs, a complicated fabrication technique, and difficulty scaling up, as reported by Vinoba *et al.* (Vinoba *et al.*, 2017). Moreover, Qadir *et al*. stated that despite the fact that a large and new number of inorganic membranes had been researched in the literature, they had yet to achieve popularity due to a variety of factors such as expensive or the high cost of the synthesis process (Qadir *et al*., 2017). Therefore, critical studies and understanding of the inorganic membrane's chemical and physical properties, stability, as well as the compatibility factor, were required before they can be successfully implemented in membrane technology, particularly for the removal of heavy metals. Successful implementation of inorganic membrane in water separation mechanism aids in obtaining high selectivity of heavy metals in water. Finally, suitable inorganic membranes must be considered so that heavy metals can be properly removed in wastewater treatment.

1.3 Research Questions

This research project will focus on the study of heavy metal removal by embedding MMT into polymeric membrane via phase inversion approach. Therefore, the research questions of this project are:

a) What is the significant effect of the membrane surface modification with MMT via phase inversion approach?
b) How will the chemical properties and morphology influenced the prepared membrane?
c) What is the efficiency of the prepared membrane on heavy metal removal capability?

1.4 Objectives

The objectives of this study are as follows:

a) To fabricate polymeric membrane with MMT at different loading via phase inversion technique.
b) To characterize the MMT and the prepared membrane.
c) To analyze performance of the prepared membrane on pure water permeability and analyze the capability on heavy metal removal in wastewater.

1.5 Scopes of study

The following are the scopes of study that have been identified:

For Objective 1:

a) Prepare a flat sheet polysulfone (PSf) polymeric membrane via phase inversion method.
b) Modify the flat sheet polymeric membrane surface with additional of MMT using three different concentrations by using phase inversion technique.

For Objective 2:

a) Characterize the MMT using PSA to analyze the particle size distribution, FTIR to evaluate the functional groups, SEM to determine the morphology and BET to investigate porosity and surface area.
b) Study the characteristics of the prepared membrane using FTIR, SEM-Energy Dispersive X-ray (EDX) to evaluate morphology along with elemental composition, BET and water uptake analysis.

For Objective 3:

a) Evaluate the effects of MMT loading parameters on water permeability.
b) Assess the heavy metal removal capability at different concentration of MMT.

1.6 Summary

In this chapter, the background of study for heavy metals, wastewater treatment via membrane technology, polymeric membrane, and MMT are presented. Besides, the issues regarding the utilisation of polymeric membrane as well as inorganic membrane are also discussed. Finally, the research questions, objectives, scopes of study and expected results are considered.

CHAPTER 2

LITERATURE REVIEW

2.1 Heavy metals

Heavy metals are generated from human sources in a variety of sectors, including agriculture, industry, home sewage, and others, and have thus become a hazard to human health and the environment. Following that, they had the most direct impact on the environment, with marine ecosystem degradation, soil deterioration, and heavy metals entering the food chain through plants, producing severe consequences on humans and animals. This is owing to its properties, which include being extremely soluble, stable, and non-biodegradable, as well as the ability to travel across aqueous medium without being digested by the body, resulting in buildup in soft tissues (Chai *et al*., 2021). Furthermore, Zuo *et al*. reported that heavy metal contamination is becoming more widely recognised as a severe global environmental problem, owing to its high toxicity, non-biodegradability, and bioaccumulation, all of which pose serious threats to human health and ecosystem stability (Zuo *et al*., 2021). This research study focuses on cadmium and lead as the heavy metals since they were considered as one of the most toxic and widespread elements in water bodies.

2.1.1 Cadmium

The regulatory limit of cadmium in drinking water was 0.005 ppm (Azimi *et al*., 2017). As for wastewater, the cadmium allowable limit was 0.003 ppm as mentioned by Kinuthia *et al*. (Kinuthia *et al*., 2020). As cadmium was exposed above the allowable limit, kidney damage was one of the most prominent potential health effects resulted from the exposure (Dutta *et al.*, 2021). Besides, cadmium had chronic toxicity towards children including body system damage and cancers of internal organ which may be due to the consumption of contaminated food or surrounded by the environment, workplace or industries that containing cadmium in the waterways (Kinuthia *et al*., 2020). Lastly, the particle size of cadmium was ranging from 0.05µm to 0.1µm based on the study of Vadgama *et al*. (Vadgama *et al*., 2017).

2.1.2 Lead

As for lead, the regulatory limit in drinking water was 0.015 ppm (Azimi *et al*., 2017). On the other hand, 0.01 ppm was the allowable limit for lead in wastewater as reported by Kinuthia *et al*. (Kinuthia *et al*., 2020). High concentration of lead can cause serious health issues such as gastrointestinal, neurological, hematological, cardiovascular, and renal problems when excessive amounts of lead were exposed to human being (Dutta *et al.*, 2021). Aside from that, Kinuthia *et al*. studied that high concentration of lead threats human health such as anaemia and deterioration in synthesizing haemoglobin, thus having headache, dullness, and memory loss as initial symptoms (Kinuthia *et al*., 2020). Moreover, lead toxicity in excess of the permitted limit causes inferior intelligence ability in children (Kinuthia *et al*., 2020). Finally, the particle size of lead was within 1µm as stated by Matthew & Krishnamurthy (Matthew & Krishnamurthy, 2018).

The particle size of contaminants were one of the most essential parameters in membrane filtration technology. This was because the contaminant's size contributes to the development of the membrane mechanism needed to remove heavy metals from water. For instance, if the size of cadmium and lead were smaller than the pore size of the membrane, the heavy metals will easily pass through the pore of the membrane, thereby causing an incomplete membrane separation process. As a result, in-depth research into heavy metal removal, including the consequences, allowable limit, and particle size, are required in order for the membrane separation mechanism to function entirely and properly.

2.2 Membrane for water separation

The membrane separation mechanism for water separation in this study was based on water filtration module. According to Judd, the technology of membrane had been developed since in the mid-19th century in the application of water treatment which includes the UF separation using bovine heart-based, synthetic UF, MF, RO membrane (Judd, 2017),. Each membrane mechanism had its own set of characteristics, such as pore size and the operating parameters that must be met when using them. It was discovered that the pore size of the membrane affects the rejection of pollutants using membrane technology, thereby a suitable type of membrane mechanism must be chosen to achieve maximum contaminant rejection. Subsequently, an in-depth evaluation of membrane filtration mechanisms including microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) membrane are presented.

First and foremost, MF membrane had pore sizes ranging from 0.1 μm to 10 μm and operating pressure within 1 bar to 6.2 bar, as reported by Maddah *et al*. (Maddah *et al*., 2018). It was able to remove suspended matter, zooplankton, algae, bacteria, and protein aggregates (Abetz *et al*., 2021). Despite its removal capabilities, the MF membrane had two main weaknesses which are it cannot entirely remove viruses and it cannot remove pollutants with a size of less than 1 mm. Since MF membrane was not an absolute virus barrier, it can be used in combination with disinfection to manage these microorganisms in water. In disinfection process, chlorination was one of the common steps in removing pathogens which leads to the addition of chemical substance. Aside from that, pollutants that have 1 mm in size or smaller than the pore size of the MF membrane will pass through the membrane easily. To conclude, MF membrane was employed to remove micrometer sized matter.

On the other hand, UF membrane was denser than MF membrane and widely used to remove some viruses, colloidal matter, macromolecules, proteins, and vitamins (Abetz *et al*., 2021). Based on the study of Maddah *et al*., it operated with pressure of 1 bar to 10 bar and has pore sizes within 1 nm to 100 nm (Maddah *et al*., 2018). It was discovered that there was no usage of chemicals required in UF membranes. In contrast, it possessed a simple automation in terms of size-exclusion filtration, consistent quality of treated water in terms of particle and microbial removal, process and plant compactness. However, Nqombolo *et al*. stated that UF membrane had certain disadvantages including inability to remove any dissolved inorganic pollutants from water and the need for routine cleaning to ensure high pressure water flow due to the membrane fouling phenomenon (Nqombolo *et al*., 2018). In summary, UF membrane was one of the pressure-driven membrane that removes pollutants with ultrafine porosity feature.

Apart from that, the other membrane technology namely NF membrane had denser and higher hydrodynamic resistance as compared to UF membrane, necessitating a larger driving force for filtration and used to separate small organic molecules and divalent salts (Asad *et al.*, 2020). In addition, Nqombolo *et al.* stated that the NF membrane can remove ions that contribute considerably to osmotic pressure, allowing for higher operation pressures (Nqombolo *et al.*, 2018). The loose selective thin film structure and small pore diameters of NF membranes were adequate and efficient for isolating metal salts (Abdullah *et al.*, 2019). NF membrane had pore sizes of 1 nm to 10 nm with operating pressure ranging from 20 bar to 40 bar (Maddah *et al.*, 2018). In addition, the NF membrane can eliminate alkalinity and hardness from water. Since the NF membrane was capable of removing alkalinity from water, the resulting product water may be corrosive. To reduce water corrosivity, control techniques such as blending raw and product water or adding alkaline chemicals were required. Moreover, pre-treatment of hard water was necessary to avoid hardness ions from accumulating on the NF membrane that may reduce the performance of the membrane. In short, the NF membrane was a good membrane for removing contaminants from water that demands high operating parameters.

Last but not least, RO membrane was one of the pressure-driven membranes that were solely permeable to water molecules and were used to remove dissolved solids and smaller particles. It was categorized as nonporous membrane as it does not have definite pores and the membrane was denser than NF membrane that capable of isolating monovalent ions. The pore sizes for RO membrane was less than 1 nm and the range of operating pressure was from 30 bar to 100 bar (Maddah *et al.*, 2018). The pressure delivered to the RO membrane must be sufficient to allow water to overcome the osmotic pressure. The net movement of water, according to the theoretical principle of osmosis, was from an area of low solute concentration to a region of high solute concentration. RO membrane operates in the reverse direction, forcing water molecules to travel against the concentration gradient by applying pressure (Abdullah *et al.*, 2019). Moreover, RO membrane had a significantly tighter pore structure than UF membranes, therefore they require less maintenance as it able to convert hard water to soft water and were essentially capable of eliminating all particles, bacteria, and organics. As stated by Nqombolo *et al.*, the utilization of high pressure, the fact that RO membrane was more expensive than other membrane technologies, and the fact that they are prone to fouling are all negatives (Nqombolo *et al.*, 2018). To summarise, the RO membrane was a potential solution for effectively removing smaller particles like salts or ions.

2.3 Types of Membrane

Membranes can be classified as organic membrane, inorganic membrane and MMM. The in-depth understanding of most common used organic membranes such as cellulose acetate (CA), polyethersulfone (PES), polyvinylidene fluoride (PVDF) and polysulfone (PSf) were provided in this subtopic. As for inorganic membrane, the review of materials including ceramics, silica, zeolite and layer silicates clay mineral membranes were presented. Moreover, the evaluation of various types of layer silicates clay minerals such as kaolinite, smectite, and chlorite were discussed thoroughly. Lastly, the literature works regarding the fabrication of MMM was reviewed in terms of the type of organic and inorganic membrane, the membrane mechanisms, water flux, types of heavy metals with its concentration, removal capacity as well as advantages and disadvantages of the membrane.

2.3.1 Organic membrane

Organic membranes also referred as polymeric membranes were widely used for the application of lab and industry. Nowadays, polymeric membranes were the most frequently utilized membrane in water treatment and desalination technology. Organic membranes that were usually implemented in water separation processes such as cellulose acetate (CA), polyethersulfone (PES), polyvinylidene fluoride (PVDF) and polysulfone (PSf) were analysed critically.

Primarily, CA membrane had been found in most literature research as the commonly employed polymer in the fabrication of membrane. It can be used to prepare various types of membrane mechanism including MF, UF, NF, and RO membranes. Cellulose was the main component to derive CA and considered as biodegradable material that can be obtained from natural resources. Due to the insoluble nature of cellulose, other chemical compounds such as acetic anhydride and acetic acid were necessary in the manufacture of CA membrane. Apart