Journal of Advanced Research in
Micro and Nano Engineering

Journal homepage:
https://semarakilmu.com.my/journals/index.php/micro_nano_engineering/index
ISSN: 2756-8210

Removal of Lead (Pb) Ions by using the Integral Membrane Incorporated with Silica from Rice Husk Ash

Hasminah Abbas[1], Norin Zamiah Kassim Shaari[1,*]

[1] School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

ARTICLE INFO

Article history:
Received 3 November 2024
Received in revised form 15 December 2024
Accepted 21 January 2025
Available online 28 February 2025

Keywords:
Lead ions; integral membrane; rice husk ash; silica; antifouling

ABSTRACT

Lead (Pb^{2+}) is one of the most toxic heavy metals, causing significant physical, biochemical, and behavioural health issues in humans. Sustainable water management practices and technologies have become crucial as clean water sources diminish. Membrane separation, a chemical-free and cost-effective method, has emerged as the preferred solution, offering economic and environmental benefits. This research focused on utilizing silica from rice husk ash as a cross-linker in the membrane formulation made from a blend of chitosan/polyvinyl alcohol/polysulfone. Chitosan is widely used as an adsorbent for removing heavy metals due to the hydroxyl and amino groups in its structures. This study examines the effect of silica incorporation in the membrane formulation on membrane properties and the removal of lead ions, focusing on various loadings of 1, 4, and 7 wt.%/wt. polymer. The properties were investigated using thermal gravitational analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), and membrane filtration experiments. TGA analysis and FTIR spectrum results showed that introducing silica from rice husk ash (RHA) particles enhanced the membranes' thermal stability and crosslinking structure. The performance results showed that the presence of silica substantially improved permeate flux, percentage removal of lead ions, and antifouling properties. This is because the silica in the RHA particles enhanced the membrane's hydrophilicity, which improves water absorption and permeation rates. 7 wt.%/wt. polymer of silica is the best formulation that removed 94% of the lead ion with the highest water flux and displayed the average antifouling performance with the relative flux recovery (RFR) of 40.26%. The formulated membrane has the potential to be used in wastewater treatment for the removal of lead ions.

1. Introduction

Despite numerous technological developments and triumphs, wastewater treatment remains a critical global issue. The wastewater, which contains heavy metal ions, poses a serious hazard to human health if not adequately treated, making its disposal critical [1]. According to Lin *et al.,* [2] and Pakade *et al.,* [3] as mentioned in Chowdhury *et al.,* [4] they are prevalent in water from diverse

---

[*] *Corresponding author.*
E-mail address: norinzamiah@uitm.edu.my

https://doi.org/10.37934/armne.30.1.101114

sectors such as electroplating, tanner mills, steel production, wood processing, plastic manufacture, metallurgical and mining activities, nuclear power plants, dyes and pigments, ceramic, paints, and fertilizer industries. Furthermore, heavy metals do not biodegrade easily, but they can be enriched hundreds of times through biological amplification, in which dangerous compounds migrate up the food chain and eventually enter the human body [5].

Heavy metals are among the most harmful contaminants on the planet's surface and lead (Pb) is one of the most hazardous heavy metals. Lead is a carcinogenic metal that rapidly enters the food chain. Lead appears in three forms: metallic lead, inorganic lead, lead compounds (or lead salts), and organic lead (including carbon). Lead in the environment is rarely found in its elemental state, but rather in its +2 oxidation state (Pb^{2+}) in diverse ores around the world [6]. The lead ions are generated from various industries such as manufacturing, chemical, battery, and paint and colouring industries [7]. In addition to severely impacting the standards of water required for human consumption, it can also result in the death of innumerable species. High amounts of lead ions affect living organisms by inhibiting or obstructing biological processes, resulting in severe and irreversible harm [8].

Various procedures for removing heavy metals from wastewater have been developed, including adsorption, chemical oxidation-reduction, precipitation, ion exchange, electrochemical processes, membrane filtration, and reverse osmosis. These procedures are often expensive and impracticable in remote places where heavy metal contamination is induced by geogenic sources [9]. On the other hand, De Gisi *et al.,* [10] stated that various treatment systems are available with varying degrees of efficiency to prevent and minimize water pollution. However, most of these systems have drawbacks such as high operational and maintenance costs, toxic sludge formation, and difficult treatment procedures. Thus, adsorption is considered a preferable choice in water and using activated carbon because of its convenience, ease of operation, and simplicity of design. Nevertheless, when a broader range of applications is required, the high cost of activated carbon is a disadvantage, and as mentioned in Ma *et al.,* [11], in a high-temperature setting, a considerable volume of saturated activated carbon can easily release hazardous chemicals.

According to Elorm *et al.,* [12] as indicated in Issaoui *et al.,* [13], membrane technology is still the most effective technique with the best prospects in wastewater treatment. Apart from that, according to Shi *et al.,* [14] as described in Issaoui *et al.,* [13] the membrane process has been widely employed in various fields as a new separation technique, and it is one of the most cost-effective water treatments accessible and saves water resources. Furthermore, membrane technology is used in the water sector to improve water quality for use, reuse, or release to the environment, as well as for wastewater [15]. As a result, membrane separation has emerged as the favoured alternative due to its mild, environmentally benign approach and lack of sludge generation. It has been reported that chitosan is a good adsorbent for heavy metal ions and the crosslinking with silica resulted in a denser membrane structure for higher entrapment of heavy metal ions [16]. Furthermore, the organics-inorganic hybrid polymer combinations of chitosan/polyvinyl alcohol blend with silica from tetraethylorthosilicate have good thermal stability and cause strong binding affinities toward metal ions such as copper and ferum [16]. In the previously reported process [16], the hybrid membrane solution was layered on the polysulfone membrane. In this current research, an integral membrane was formulated where a blend of chitosan/polyvinyl alcohol/silica from rice husk ash was mixed with a polysulfone solution. To utilize the biomass from rice, the silica from rice husk ash was extracted using a modified method and incorporated into the hybrid membrane solution [17]. The utilization of the abundant biomass from rice milling could reduce the impact to the environment due to the burning activities of the rice husk [18]. This research aims to investigate the potential utilization of silica from rice husk ash as a crosslinker in the membrane's structure made of polyvinyl alcohol/chitosan/polysulfone to remove lead ions. The effect of silica loadings on the thermal stability, water flux, and antifouling performance was further evaluated.

2. Methodology

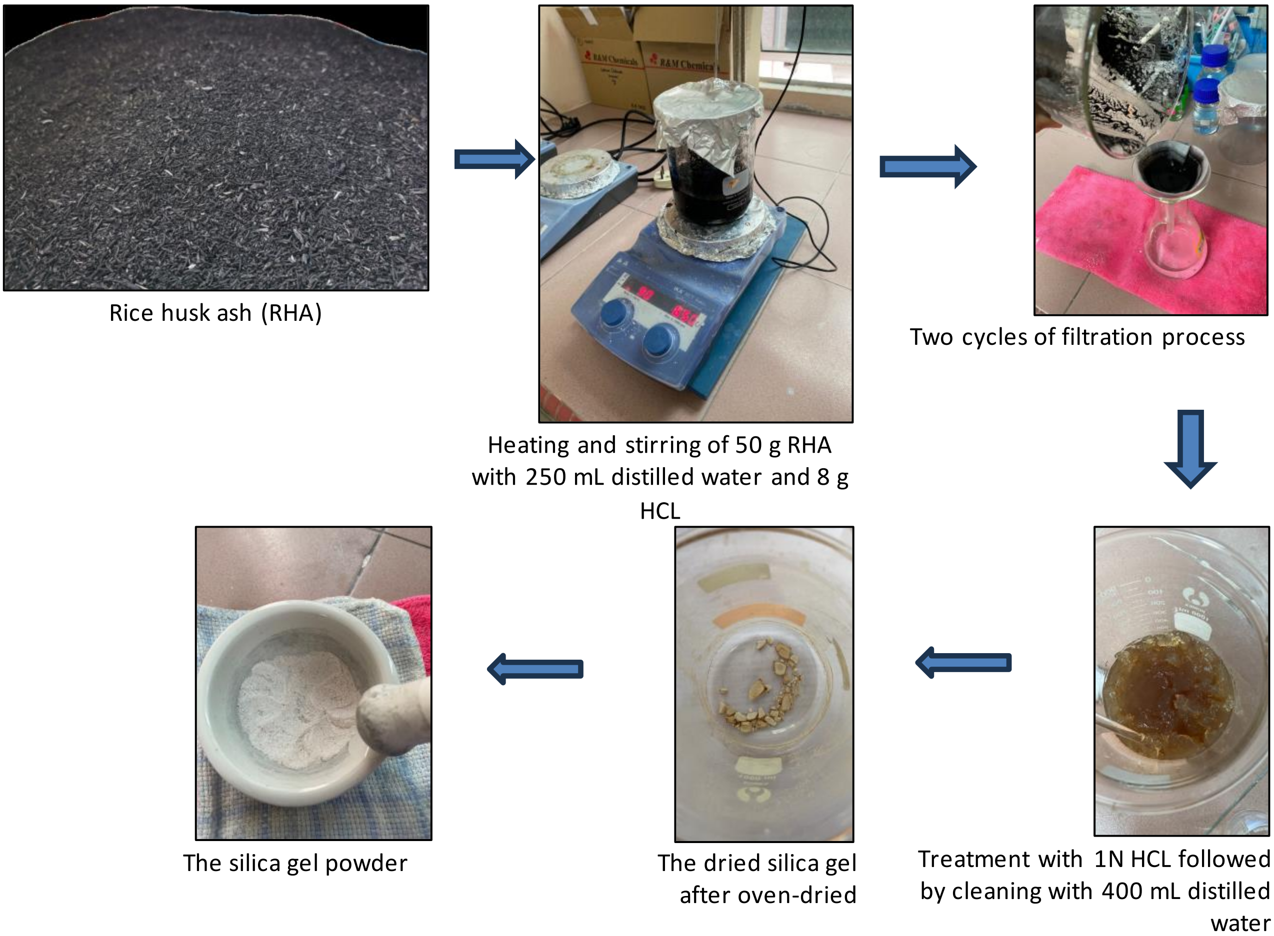
2.1 Material

Polyvinyl alcohol powder with a molecular weight of 85000-124000 and 87-89% hydrolyzed, and polysulfone pellets with 35000 molecular weights were purchased from Sigma-Aldrich (M) Sdn. Bhd. The chitosan powder was purchased from Aman Semesta Enterprise. 1-methyl-2-pyrrolidone (NMP), and polyethylene glycol 400 (PEG 400) were purchased from Merck Sdn. Bhd. The dimethyl sulfoxide, humic acid, and hydrochloric acid (HCl, 35-37% purity) were purchased from R&M Chemicals. The rice husk ash was supplied by BT Science Sdn. Bhd. Other materials such as sodium hydroxide, acetic acid, lead nitrate, and distilled water were obtained from the chemical laboratory of the School of Chemical Engineering, UiTM Shah Alam.

2.2 Methods

2.2.1 Silica extraction method for rice husk ash (RHA)

The extraction of silica from rice husk ash (RHA) was prepared according to the procedure outlined by Chik *et al.,* [17] and Mohd Zainuddin *et al*., [19]. The first step was cleaning the raw material where 50.0 g of raw RHA was mixed with 250 mL of distilled water and 8.0 g of HCl. After that, the mixture was heated to 90°C and stirred for one hour at 650 rpm. The mixture was filtered overnight with Smith 102 filter paper after being cooled overnight. Next, 250 mL of 1.0 M NaOH was added to the RHA, then heated to 90°C and stirred for one hour at 650 rpm. Before the mixture was filtered using filter paper Smith 102, the mixture was once again cooled to ambient temperature overnight. The filtrate was treated with 1N HCl to bring the pH to 7, initially at pH 14. The gel was then thoroughly cleaned and rapidly agitated with 400 mL of distilled water at 1,500 rpm. The washing procedure was repeated until clear gel was obtained to remove sodium chloride (NaCl) and obtain water and high-purity silicon dioxide (SiO_2). The gel was dried in a 60°C oven for 24 hours. The powdered dried silica gel was then obtained. The extraction of silica is represented in Figure 1.


Rice husk ash (RHA)

Heating and stirring of 50 g RHA with 250 mL distilled water and 8 g HCL

Two cycles of filtration process

Treatment with 1N HCL followed by cleaning with 400 mL distilled water

The dried silica gel after oven-dried

The silica gel powder

Fig. 1. The silica extraction process

2.2.2 Blend membrane solution preparation

As mentioned in Sulaiman *et al.,* [20], a homogenous solution of polyvinyl alcohol was created by heating 90.0 g of dimethyl sulphoxide and 10.0 g of polyvinyl alcohol (PVA) powder at 90°C for 4 hours while being stirred at 400 rpm. The solution was then chilled until it reached room temperature. An amount of 0.02 g chitosan was dissolved in 99.98 g aqueous acetic acid, which was then heated at 90°C for 4 hours while being stirred at 400 rpm. This resulted in a 2 wt.% chitosan solution. After that, it was kept at room temperature.

2.2.3 Preparation of hybrid membranes

PVA and chitosan were each made in separate solutions, which were then combined at a weight ratio of 1:1 and heated at 60°C for 6 hours while stirred at 400 rpm. A 1 wt.% concentration of the extracted silica powder from RHA powder was added. Table 1 shows the silica powder's composition. A volume of 1.0 mL HCl was used as a catalyst for the sol-gel method. After that, the mixture was allowed to cool to ambient temperature. Another membrane was created without the inclusion of silica by repeating the previous stages with 4 wt.% and 7 wt.% of silica powder. Table 2 displays the composition of each formulation [20]. The schematic diagram of the preparation of the hybrid membrane solution is displayed in Figure 2.

Table 1
Composition of silica powder

Compound	Percentage (%)
Si	77.02
Na	0.49
Cl	0.53
Other impurities	21.97

Table 2
Composition of various membrane solutions

Membrane Code	PVA Solution (g)	CS Solution (g)	Silica Powder (wt./wt.% polymer)
0 RHA	20	20	0
1 RHA	20	20	1
4 RHA	20	20	4
7 RHA	20	20	7

2.2.4 Preparation of polysulfone (PSF) solution

Research by Sulaiman *et al.,* [20] as cited in Shaari *et al.,* [16] demonstrated the preparation of a polysulfone (PSF) solution. As stated, after dissolving 13.0 g of PSF beads in 82.0 g of 1-methyl-2-pyrrolidone (NMP), 5.0 g of polyethylene glycol was added. The solution was heated and constantly agitated at 60°C at 400 rpm for about 6 hours. The mixture was then allowed to cool to room temperature.

2.2.5 Preparation of integral membrane

An amount of 1.0 g of each hybrid membrane solution as shown in Table 2 was mixed with 40.0 g polysulfone solution. The mixture was heated to 80°C with continuous agitation at 800 rpm for 3 hours. After cooling, the mixture was put onto a glass plate and a film was formed using a Baker Film Applicator to a thickness of 100 µm. The plate was then submerged in a large amount of water for 24 hours. The membrane was then allowed to dry for 24 hours at room temperature [21,22]. The production process of the membrane is represented in Figure 3.

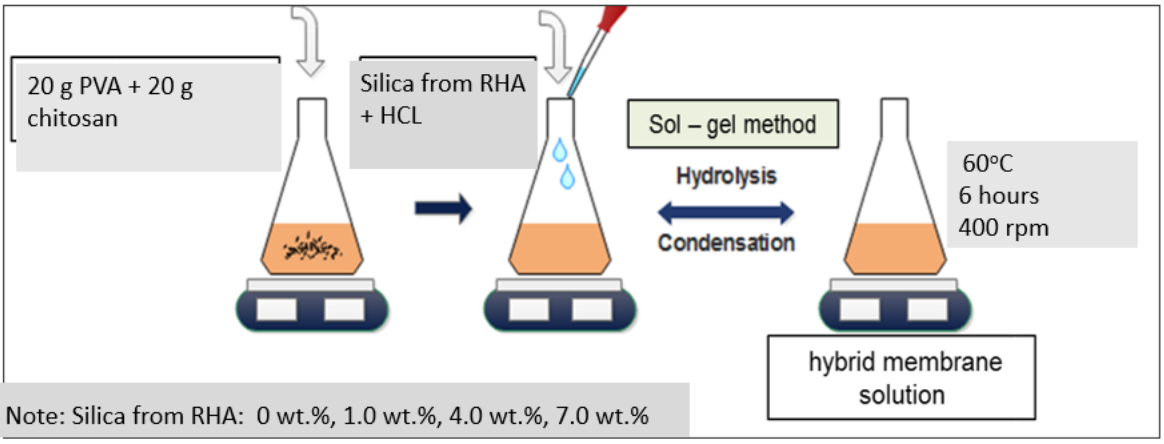


Fig. 2. The preparation process of the hybrid membrane solution

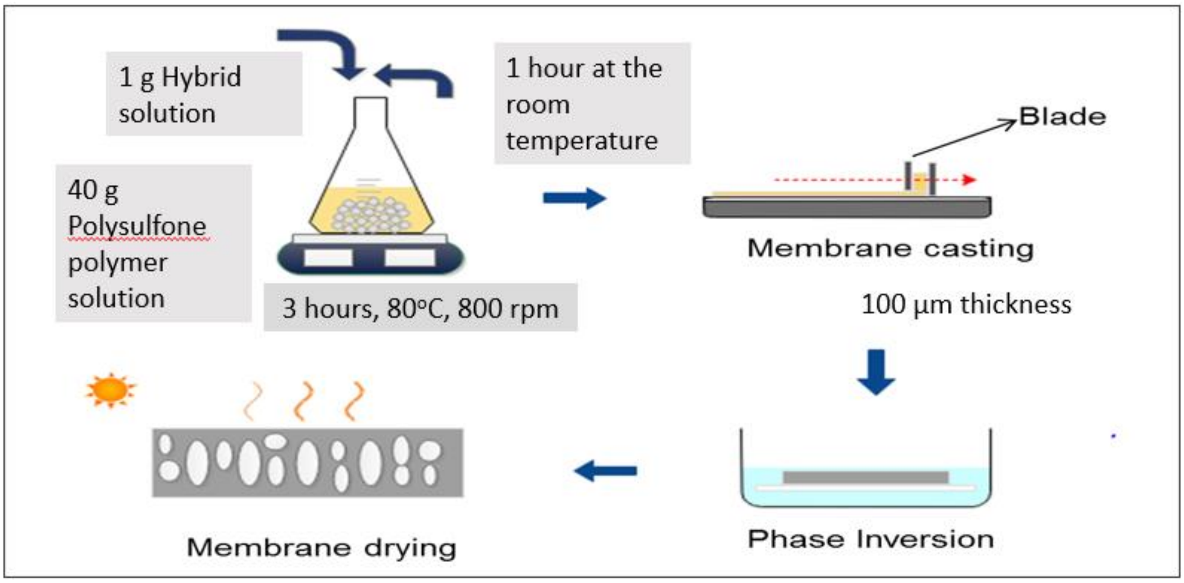


Fig. 3. The preparation process of the integral membrane

2.2.6 Characterizations of membrane

The membrane was cut into small pieces weighing 50.0 and 60.0 mg and placed into the crucible. The membranes were heated with nitrogen gas at a rate of 10°C/min from 0°C to 900°C [19]. The Mettler Toledo model Stare SW served as the basis for the TGA model. Research by Ghiggi *et al.,* [23] as cited in Shaari *et al.* [16], mentioned that through FTIR research, the integral membrane's functional groups were observed.

2.2.7 Performance of membranes

The performance of the membrane in terms of lead removal and antifouling analysis was carried out using the fabricated dead-end filtration apparatus as shown in Figure 4 [20]. Using deionized water and a dilution process from a 100-ppm lead solution, the lead solutions at 4, 8, and 12 ppm were formed. Nitrogen gas was used to produce the requisite pressure (2 bar) to run the dead-end configuration [24]. Each membrane piece was subjected to a one-hour filtration with 15-minute sampling intervals. Based on Eq. (1), flux from the withdrawn sample solution was measured every 15 minutes.

$$Flux = \left(\frac{\Delta V}{A\Delta t}\right) \tag{1}$$

where ΔV is the volume of the collected permeate solution (mL), A is the membrane's cross-sectional area (m^2), and Δt is the time interval (min). Atomic Absorption Spectroscopy (AAS) from Mettler Toledo was used to quantify lead concentrations in the feed (C_f) and permeate stream (C_p). Eq. (2) was used to obtain the percentage rejection of lead ions from the initial solution.

$$Rejection\ (\%) = \left(\frac{C_f - C_p}{C_f}\right) \times 100 \tag{2}$$

In the anti-fouling experiment, the nitrogen gas pressure was 2 bar [25]. The foulant model was created by dissolving 2.0 g of humic acid in 1.0 L of 200 ppm sodium hydroxide. The procedure was completed in three steps. The first step was completed within 30 minutes by the filtration of deionized water. The flux was measured and labelled as J_0. The deionized water as the feed solution was replaced with humic acid (HA) solution and the filtration was performed for 2 hours. The final flux at this stage was measured and labelled as J_p. The membrane was removed from the stirred cell and cleaned by soaking it in 50 mL water and stirred at 150 rpm in a shaker for 30 minutes. The final stage was carried out by repeating the first step, and the flux was designated as J_1 [26]. The fouling resistance of the membrane was determined using the flux recovery decay (RFD) and relative flux recovery (RFR) methods described by Zhu *et al.,* [26] according to Eq. (3) and Eq. (4) respectively.

$$RFD\ (\%) = \left(\frac{J_0 - J_p}{J_0}\right) \times 100 \tag{3}$$

$$RFR\ (\%) = \left(\frac{J_1}{J_0}\right) \times 100 \tag{4}$$

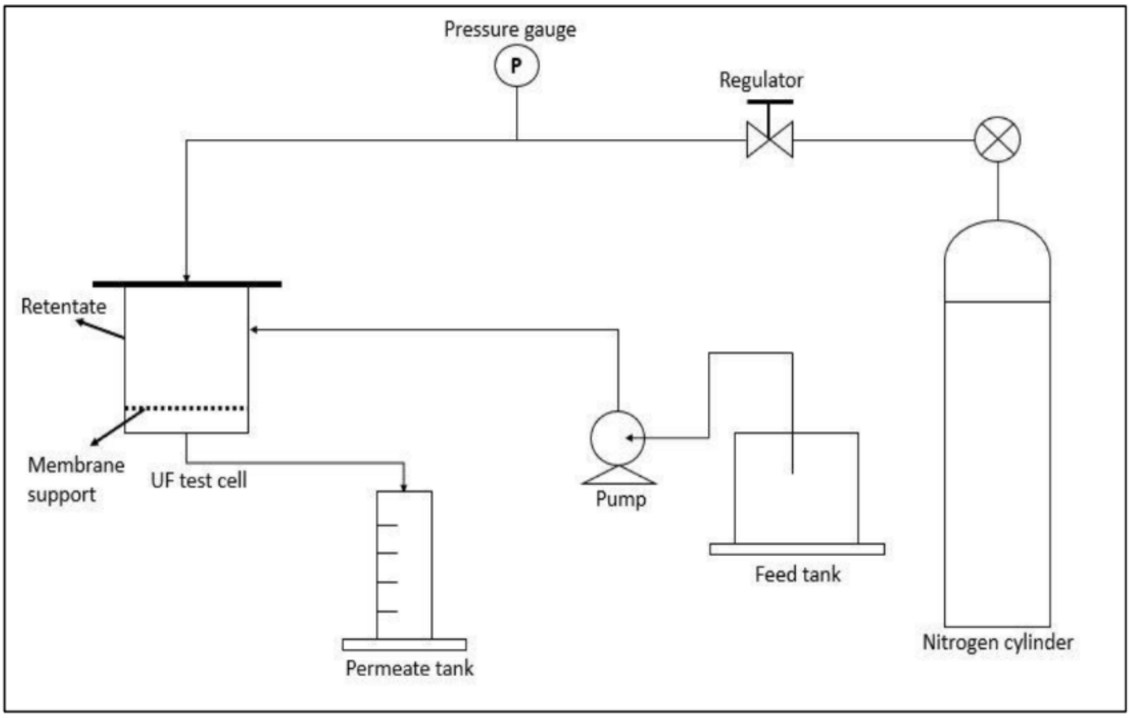


Fig. 4. The schematic diagram of the dead-end membrane filtration rig [19]

3. Results

3.1 Thermal Stability

Thermogravimetric analysis (TGA) is a potent method for determining a material's thermal stability [27]. The results from the TGA analysis are displayed in Figure 5. All the membranes showed a small weight loss which ranges from 30°C to 400°C. According to Huang *et al.,* [28] and Jamalludin *et al.,* [25], this is due to the evaporation of the absorbent water and the residual NMP, an organic solvent with a boiling point of 202 °C.

The weight loss occurs between 428–598 °C corresponding to the combustion of the polymer. It was observed that the change in the TG curves indicates that adding silica from RHA raises the membrane's degradation temperature [25]. Furthermore, Jamalluddin *et al.,* [25] have mentioned that this is possibly due to the interaction between the PSF and SiO_2 groups. Adding silica from RHA enhances the membranes' thermal stability because they are inorganic polymer nanocomposite membranes [29]. This phenomenon describes that adding fillers enhances the mass transport barrier effects against the volatile compounds produced during degradation and the oxidizing atmosphere [29].

The residual masses after TG analysis are 33%, 32%, 36%, and 36% for 0 RHA, 1 RHA, 4 RHA, and 7 RHA, respectively. The increase in the residual mass corresponds to the amount of RHA added except for 0 RHA. This confirms the successful incorporation of the silica from RHA in the membranes. 7 RHA exhibits a greater residual mass compared to the other three membranes. Due to its higher silica content, the polymer undergoes thermal degradation at elevated temperatures. According to Sulaiman *et al.,* [20], an increased RHA content enhances the integration of silica into the chitosan/PVA polymer matrix, demanding more reaction activation energy and leading to a higher order. Consequently, the polymer gains higher strength and is thermally stable with higher silica content [16].

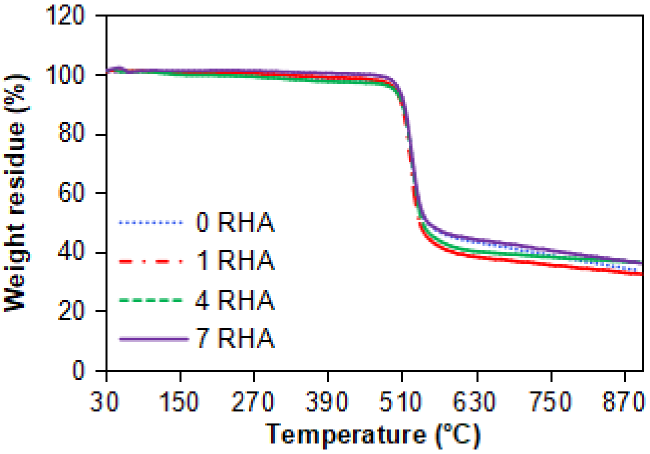


Fig. 5. Percentage weight residue of the membranes at different temperatures

3.2 FTIR Analysis

The surface functional group's main polymers of the membrane structure as demonstrated by the FTIR spectrum and derived from the Fourier Transform spectroscopy analysis, are the primary determinants of its adsorption properties. Figure 6 displays the FTIR analysis findings. The fingerprint of the membrane is represented by an array of peaks, as each figure makes evident. These peaks are similar and are mainly composed of polysulfone, polyvinyl alcohol, chitosan, and rice husk ash, with most of the peaks falling within the 1600–500 cm^{-1} range [30].

The raw hybrid membrane (chitosan/PVA) is represented by spectrum (a). The carboxyl stretching (C=O) of the secondary amide band (amide I) of pure chitosan is represented by the two peaks at spectra (a) at 1503.79 and 1585.12 cm^{-1}. A very strong absorption peak is also visible at 1240.08 cm^{-1} in the spectrum (a), and it is linked to the –C-O stretching of carboxylic acid [20].

Spectrum (b), (c), and (d) represent the spectrum of chitosan/PVA hybrid membrane with 1% 4%, and 7% of RHA content respectively. In spectrums (b), (c), and (d), the presence of silica is identified with the existence of a crosslinked bond of the hybrid membrane. The FTIR spectrum of the silica showed the characteristic peaks assigned to the asymmetric and symmetric stretching of Si-O-Si groups at 1014 cm^{-1} and 833 cm^{-1}, respectively [31]. The peak at 2973–2962 cm^{-1} corresponded to C-H stretching that overlapped with amine bands [20]. As seen in Figure 6, an absorption band ranging from 3000 to 3794 cm^{-1} was found to represent the hydroxyl groups from all membranes where the intensity decreases with the increase in loading of silica powder in the membrane's formulation [30].

Peaks that exist at 1322.76 cm^{-1} are attributed to the blended chitosan/PVA's C-N stretching. The Si-O-C groups' distinctive band, located between 1013.97 cm^{-1} and 1014.11 cm^{-1}, demonstrated the condensation reaction between the alcohol group in PVA and the silanol groups in RHA, resulting in the formation of a covalent bond between PVA and silica. According to Reino Olegário da Silva *et al.,* [30] the condensation reaction enhanced the compatibility and crosslinking network between the inorganic and organic components in the integral membrane.

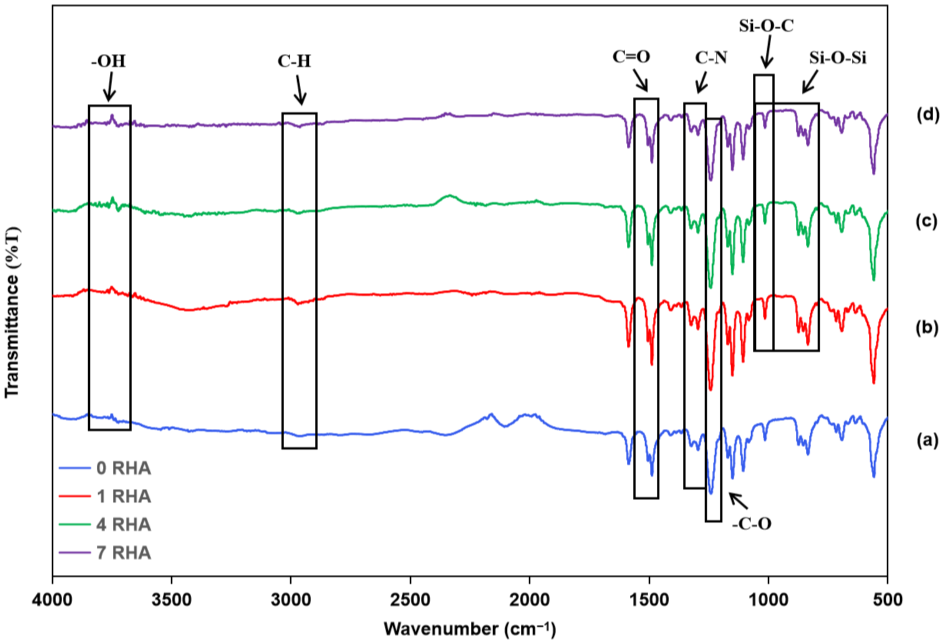
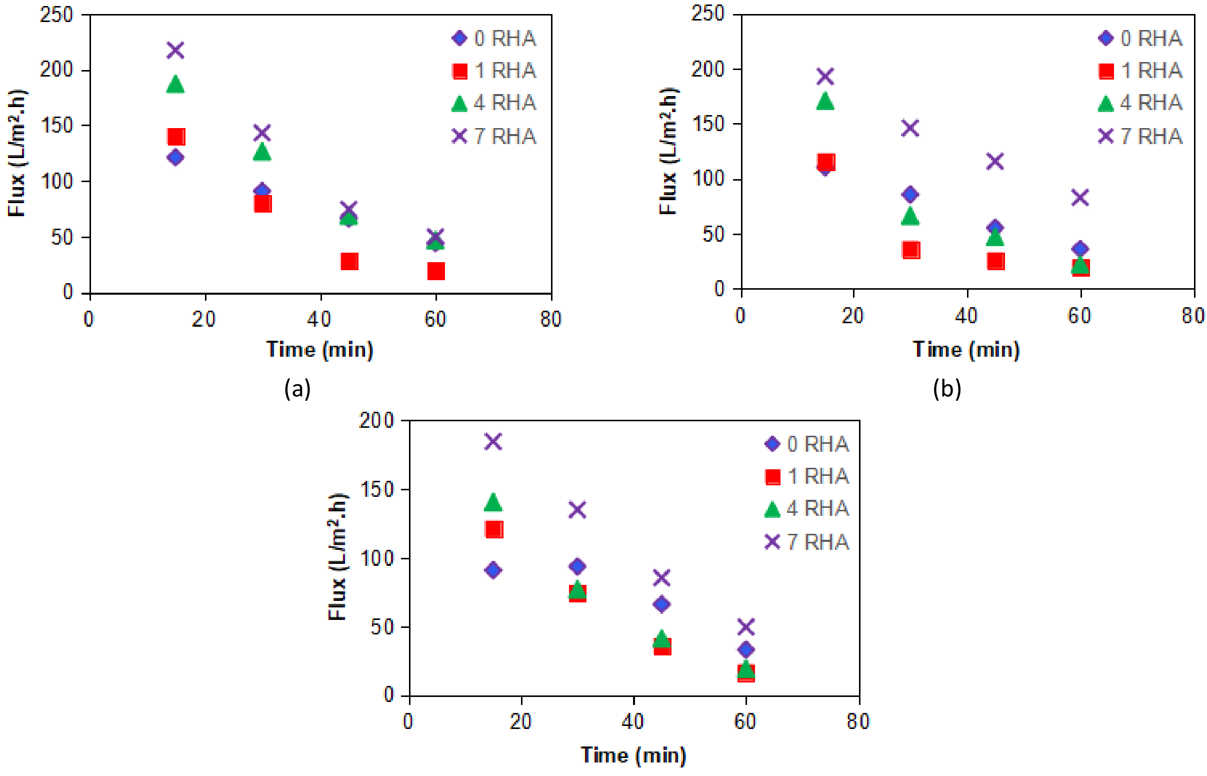


Fig. 6. FTIR spectra of membranes (a) FTIR analysis for 0 RHA (b) FTIR analysis for 1 RHA (c) FTIR analysis for 4 RHA (d) FTIR analysis for 7 RHA

3.3 Permeate Flux and Membrane Rejection

The permeation fluxes from the lead removal process at different feed concentrations of 4 ppm, 8 ppm, and 12 ppm are displayed in Figure 7(a)-(c). These three figures show that all membranes observed a consistent drop in fluxes after a 15-minute interval of filtration for 1 hour. Based on Figure 7(a), the control membrane, 0 RHA followed by a low silica loading of 1 wt.% of RHA exhibited the lowest permeate flux. The permeate flux increased sharply as the RHA particles were increased to 4 wt.% and 7 wt.%. Toosi *et al.,* [24] state that the presence of silica from RHA increases the hydrophilicity of the membrane, which explains the increase in permeate flux from the addition of RHA particles.

Figure 7(a)-(c), show that, at the first 15 minutes of the time interval for all lead solution concentrations, membrane 7 RHA had the highest flux with 217.60 L/m^2.h at 4 ppm followed by 192.81 L/m^2.h at 8 ppm, and 184.55 L/m^2.h at 12 ppm, accordingly. Furthermore, in general, it is evident from Harun *et al.,* [32] also mentioned that a higher hydrophilicity value corresponds well with a higher permeation value when there is an increase in the silica loading. Additionally, according to Huang *et al.,* [28] the addition of silica from RHA increases the water flux due to the enlargement of the membrane's finger-like pore size and improved interconnectivity across the membrane thickness, which decreases membrane hydraulic resistance. Therefore, these findings demonstrate that as the quantity of RHA particles increases, the permeate flux also increases, as evidenced by the results indicating that 7 RHA exhibits the highest flux.



(a)

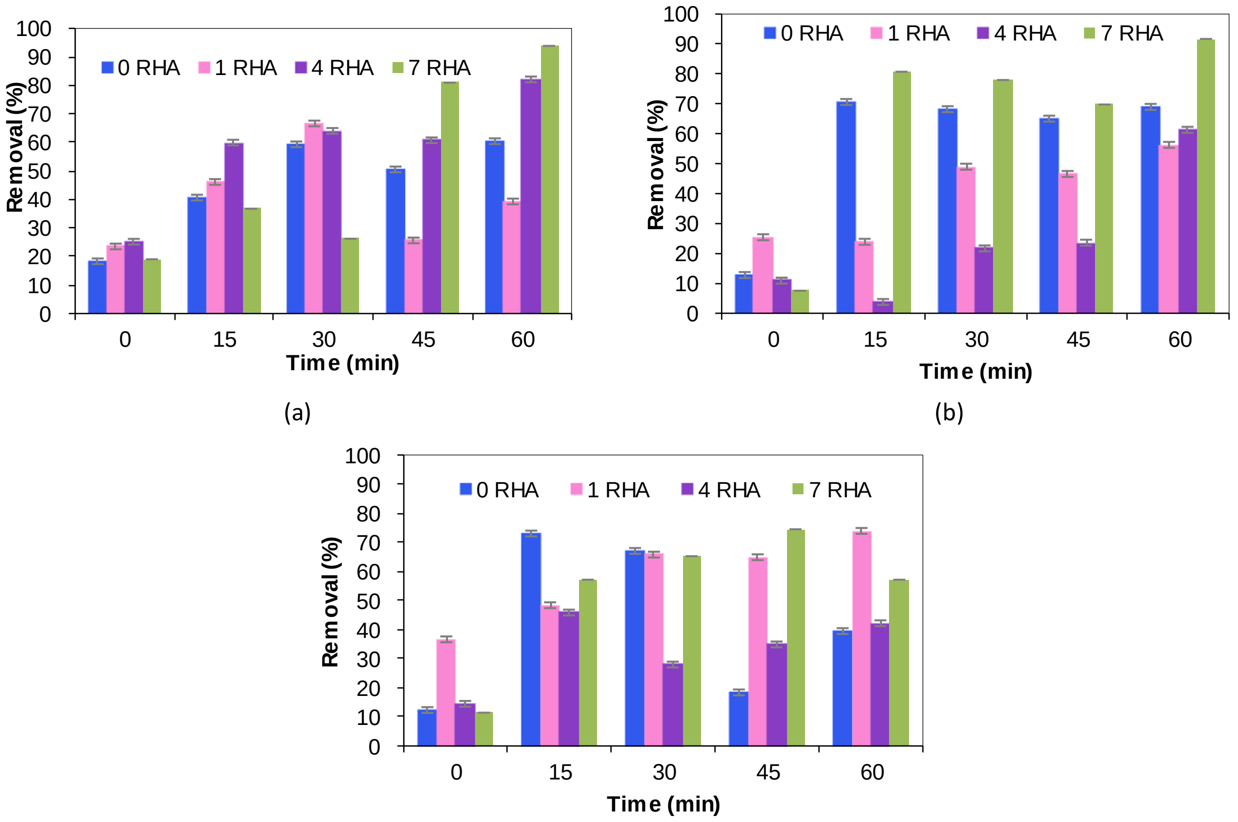
(b)

(c)

Fig. 7. (a) Flux for 4 ppm of lead solution (b) Flux for 8 ppm of lead solution (c) Flux for 12 ppm of lead solution

The membrane's ability to remove the lead ions was assessed by examining the percentage removal of lead ions from various feed solution concentrations, as depicted in Figure 8. The results suggest that the percentage of lead removal rises with prolonged exposure time. The elevated adsorption at the initial concentration is likely attributed to increased open sites available on the membrane surface in the early stages. Since the amount of chitosan as an adsorbent for lead ions is fixed in the membrane formulation, the only variable affecting the rejection of lead ions is RHA loading as the crosslinker.

As illustrated in Figure 8(a)-(c) for each lead ion concentration, there was a fluctuation trend in the percentage removal of lead ions for all membranes, where the percentage reached a maximum at 30 minutes before it declines until 60 minutes, especially at 12 ppm lead solution. As shown in the figures, membrane 7 RHA displayed the highest percentage removal of lead ions with a value of 94% at 4 ppm followed by 92% at 8 ppm after a 1-hour filtration. However, lower removal was demonstrated at 12 ppm lead solution due to the accumulation of lead ions on the membrane's surface [33]. Membrane 7 RHA removes the highest percentage of lead ions (75%) from 12 ppm solution after 45 minutes of filtration time. This result was anticipated because the increase in RHA resulted in enhancing the crosslinking of the polymer membrane and tightening of the membrane surface to provide greater surface area for metal ion adsorption [32]. It can facilitate more interaction, more diffusion of the feed solution into the membrane, and more adsorption of Pb^{2+} ions. Based on the significant amount of flux discharged by Membrane 7 RHA as shown in Figure 7 (a)-(c) along with higher removal of lead ions, the incorporation of 7 wt.% silica from RHA is the optimum loading in the membrane formulation.



(a) (b)

(c)

Fig. 8. Removal efficiency of lead ions at various loadings of silica from RHA with different lead feed concentrations (a) 4 ppm (b) 8 ppm (c) 12 ppm

3.4 Antifouling Performance

Humic acid (HA) was used as the foulant model to evaluate the membrane's antifouling performance. According to Fan *et al.,* [34] as cited in Zhu *et al.,* [26] humic substances, including humic acids, frequently represent a significant portion of natural organic matter in surface water or groundwater. The relative flux decay (RFD) and recovery flux ratio (RFR) results were calculated and displayed in Table 3.

According to Zhu *et al.,* [26] incorporating additives such as silica from rice husk ash polymer into the membrane enhances antifouling performance compared to the control membrane, resulting in a lower flux decline rate and higher flux recovery rate. The formulated membrane must withstand organic fouling brought on by natural organic matter. Based on Table 3, high RFD was portrayed by all membranes where the control membrane (0 RHA) showed a flux decline of 86.61% which was the lowest value, and the membrane with silica from RHA showed the highest value with 94.25%, 93.80%, and 95.16% for 1 RHA, 4 RHA, and 7 RHA, respectively. The RFD value is very high for all membranes due to employing high concentrations of humic acid.

As shown in Table 3, the experimental results for the RFR stand at 43.75% for 0 RHA after 2 hours of filtration, indicating significant fouling on the base membrane surface. The RFR increased to 51.82% for 1 RHA followed by the highest value of 63.86% for 4 RHA as the silica loading increased to 4% and then decreased to 40.26% for 7 RHA at a 7% silica loading. Despite the RFR being lower for all membranes, higher water fluxes (J_0 and J_1) were displayed by membranes with silica from RHA. This situation was attributed to the enhancement of the hydrophilicity from the incorporation of SiO_2 nanoparticles arising from hydrogen bonding between the hydroxyl group of water and SiO_2, [35].

This situation also led to effective antifouling capabilities and increased flux recovery in membranes with silica loading as portrayed particularly by 1 RHA and 4 RHA. Membrane 7 RHA has comparable RFR with 0 RHA and a decrease in RFR due to pore blockage, as explained by Huang *et al.,* [28] Furthermore, the 7 RHA membrane exhibits lower flux recovery due to the difficulty of washing out trapped humic substances in its pores. Despite this, most of the RFR values of membranes with silica loading are higher than that of the control membrane, meaning that modifying the membranes with silica from RHA particles improves the fouling resistance.

Table 3
Antifouling properties of membrane from various formulations

Membrane Code	J_o (L/m^2.h)	J_p (L/m^2.h)	J_1 (L/m^2.h)	RFD (%)	RFR (%)
0 RHA	66.11	8.85	28.92	86.61	43.75
1 RHA	151.49	8.71	78.50	94.25	51.82
4 RHA	114.31	7.09	72.99	93.80	63.86
7 RHA	106.05	5.13	42.69	95.16	40.26

4. Conclusions

The introduction of silica particles from rice husk ash has increased the thermal stability of the membranes because of the crosslinking process as displayed through the FTIR spectra. It was also demonstrated that the incorporation of silica enhances the hydrophilicity of membranes through water flux increment. A higher percentage removal of lead ions with good antifouling properties was also portrayed. The results indicated that membrane 7 RHA, which has 7 wt.% silica from RHA was the best membrane which exhibits the highest percentage removal of lead ions irrespective of the concentration of the lead solution without jeopardizing the flux and it has good thermal stability. These findings underscore the potential of RHA in membrane fabrication for wastewater treatment, particularly in removing lead ions.

Acknowledgment

This research was not funded by any grant and the authors would like to thank the College of Engineering, UiTM Shah Alam for providing the facilities to conduct the research and perform the analysis.

References

[1] Samavati, Zahra, Alireza Samavati, Pei Sean Goh, Ahmad Fauzi Ismail, and Mohd Sohaimi Abdullah. "A Comprehensive Review of Recent Advances in Nanofiltration Membranes for Heavy Metal Removal from Wastewater." *Chemical Engineering Research and Design* 189 (2023): 530–71. https://doi.org/10.1016/j.cherd.2022.11.042

[2] Lin, Chunxiang, Sha Qiao, Wei Luo, Yifan Liu, Danhui Liu, Xiaojuan Li, and Minghua Liu. "Thermodynamics, kinetics, and regeneration studies for adsorption of Cr (VI) from aqueous solutions using modified cellulose as adsorbent." *BioResources* 9, no. 4 (2014): 6998-7017. https://doi.org/10.15376/BIORES.9.4.6998-7017

[3] Pakade, Vusumzi E., Nikita T. Tavengwa, and Lawrence M. Madikizela. "Recent advances in hexavalent chromium removal from aqueous solutions by adsorptive methods." *RSC advances* 9, no. 45 (2019): 26142-26164. https://doi.org/10.1039/C9RA05188K

[4] Chowdhury, Noman, Solaiman, Chanchal Kumar Roy, Shakhawat H. Firoz, Tahmina Foyez, and Abu Bin Imran. "Role of ionic moieties in hydrogel networks to remove heavy metal ions from water." *ACS omega* 6, no. 1 (2020): 836-844. https://doi.org/10.1021/acsomega.0c05411.

[5] Gong, Zhaoyuan, Hiu Ting Chan, Qilei Chen, and Hubiao Chen. "Application of nanotechnology in analysis and removal of heavy metals in food and water resources." *Nanomaterials* 11, no. 7 (2021): 1792. https://doi.org/10.3390/nano11071792

[6] Sanders, Talia, Yiming Liu, Virginia Buchner, and Paul B. Tchounwou. "Neurotoxic effects and biomarkers of lead exposure: a review." *Reviews on environmental health* 24, no. 1 (2009): 15-46. https://doi.org/10.1515/reveh.2009.24.1.15

[7] Awad, Aymen. "Removal efficiency, metal uptake, and breakthrough curve of aqueous lead ions removal using olive stone waste." *Results in Engineering* 22 (2024): 102311. https://doi.org/10.1016/j.rineng.2024.102311

[8] Hawal, Laith Hamdan, Ali Omran Al-Sulttani, and Nagam Obaid Kariem. "Adsorption of lead ions from aqueous solutions onto rice husks, continuous system." *Journal of Ecological Engineering* 22, no. 10 (2021): 269-274. https://doi.org/10.12911/22998993/141936

[9] Raikar, Rajkumar V., Sefra Correa, and Praveen Ghorpade. "Removal of lead (II) from aqueous solution using natural and activated rice husk." *International Research Journal of Engineering and Technology* 2, no. 03 (2015): 1677-1685.

[10] De Gisi, Sabino, Giusy Lofrano, Mariangela Grassi, and Michele Notarnicola. "Characteristics and adsorption capacities of low-cost sorbents for wastewater treatment: A review." *Sustainable Materials and Technologies* 9 (2016): 10-40. https://doi.org/10.1016/j.susmat.2016.06.002

[11] Ma, Yuanze, Xiuxia Zhang, and Jingyang Wen. "Study on the harm of waste activated carbon and novel regeneration technology of it." In *IOP Conference Series: Earth and Environmental Science*, vol. 769, no. 2, p. 022047. IOP Publishing, 2021. https://doi.org/10.1088/1755-1315/769/2/022047

[12] Obotey Ezugbe, Elorm, and Sudesh Rathilal. "Membrane technologies in wastewater treatment: a review." *Membranes* 10, no. 5 (2020): 89. https://doi.org/10.3390/membranes10050089

[13] Issaoui, Mansour, Salah Jellali, Antonis A. Zorpas, and Patrick Dutournie. "Membrane technology for sustainable water resources management: Challenges and future projections." *Sustainable Chemistry and Pharmacy* 25 (2022): 100590. https://doi.org/10.1016/j.scp.2021.100590

[14] Shi, Yan, Songtao Zhong, and Zhaohui Li. "Pilot tests on the treatment of bath wastewater by a membrane bioreactor." *Membranes* 11, no. 2 (2021): 85. https://doi.org/10.3390/membranes11020085.

[15] Zorpas, Antonis A. "Chemical oxidation and Membrane Bioreactor for the treatment of Household heating wastewater." *Desalination and Water Treatment* 51, no. 37-39 (2013): 6952-6960. https://doi.org/10.1080/19443994.2013.794575

[16] Shaari, Norin Zamiah Kassim, Nurul Aida Sulaiman, and Norazah Abd Rahman. "Thin film composite membranes: preparation, characterization, and application towards copper ion removal." *Journal of Environmental Chemical Engineering* 7, no. 1 (2019): 102845. https://doi.org/10.1016/j.jece.2018.102845

[17] Chik, NS Izzati, NZ Kassim Shaari, N. A. Ramlee, and MR Abdul Manaf. "Extraction of silica from rice husk ash and its effect on the properties of the integral membrane." *ASM Sci. J* 17 (2022): 1-13. https://doi.org/10.32802/asmscj.2022.1035

[18] Hasan, Touhid, Ma Wadud, Mahmudul Hasan Niaz, Md. Kutub Uddin, and Md Shariful Islam. "Fabrication and Characterization of Rice Husk Ash Reinforced Aluminium Matrix Composite." *Malaysian Journal of Composite Science and Engineering* 14, no. 1 (2024): 34–43. https://doi.org/10.37934/mjcsm.14.1.3443

[19] Mohd Zainuddin, Nurul Atirah Fitriah, Norin Zamiah Kassim Shaari, Nur Syazwanie Izzati Chik, and Fazlena Hamzah. "Evaluation of crosslinking degree on the integral membrane by using Rice Husk Ash (RHA)." *Journal of Mechanical Engineering (JMechE)* 20, no. 2 (2023): 57-71. https://doi.org/10.24191/jmeche.v20i2.22053

[20] Sulaiman, Nurul Aida, Norin Zamiah Kassim Shaari, and Norazah Abdul Rahman. "Removal of Cu (II) and Fe (II) ions through thin film composite (TFC) with hybrid membrane." *J. Eng. Sci. Technol* 11 (2016): 36-49.

[21] Kassim Shaari, Norin Zamiah, Nabilah Huda Alias, and Nurul Ati. "Removal of manganese ion using integral membrane incorporated with rice husk ash." *Scientific Research Journal* 20, no. 1 (2023): 125-143. https://doi.org/10.24191/srj.v20i1.20332

[22] Francisco, Gil J., Amit Chakma, and Xianshe Feng. "Membranes comprising of alkanolamines incorporated into poly (vinyl alcohol) matrix for CO2/N2 separation." *Journal of Membrane Science* 303, no. 1-2 (2007): 54-63. https://doi.org/10.1016/j.memsci.2007.06.065

[23] Ghiggi, Fernanda F., Liliane D. Pollo, Nilo SM Cardozo, and Isabel C. Tessaro. "Preparation and characterization of polyethersulfone/N-phthaloyl-chitosan ultrafiltration membrane with antifouling property." *European Polymer Journal* 92 (2017): 61-70. https://doi.org/10.1016/j.eurpolymj.2017.04.030

[24] Toosi, Mohammad Reza, Mohammad Reza Sarmasti Emami, and Sudeh Hajian. "Dynamic filtration and static adsorption of lead ions in aqueous solution by use of blended polysulfone membranes with nano size MCM-41 particles coated by polyaniline." *Environmental Science and Pollution Research* 25 (2018): 20217-20230. https://doi.org/10.1007/s11356-018-2236-3

[25] Jamalludin, Mohd Riduan, Zawati Harun, Siti Khadijah Hubadillah, Hatijah Basri, Ahmad Fauzi Ismail, Mohd Hafiz Dzarfan Othman, Mohd Fikri Shohur, and Muhamad Zaini Yunos. "Antifouling polysulfone membranes blended with

green SiO_2 from rice husk ash (RHA) for humic acid separation." *Chemical Engineering Research and Design* 114 (2016): 268-279. https://doi.org/10.1016/j.cherd.2016.08.023

[26] Zhu, Xiaoying, Hong-En Loo, and Renbi Bai. "A novel membrane showing both hydrophilic and oleophobic surface properties and its non-fouling performances for potential water treatment applications." *Journal of Membrane Science* 436 (2013): 47-56. https://doi.org/10.1016/j.memsci.2013.02.019

[27] Ebnesajjad, Sina. "Surface and material characterization techniques." In *Handbook of adhesives and surface preparation*, pp. 31-48. William Andrew Publishing, 2011. https://doi.org/10.1016/b978-081551523-4.50006-7

[28] Huang, Jian, Kaisong Zhang, Kun Wang, Zongli Xie, Bradley Ladewig, and Huanting Wang. "Fabrication of polyethersulfone-mesoporous silica nanocomposite ultrafiltration membranes with antifouling properties." *Journal of membrane science* 423 (2012): 362-370. https://doi.org/10.1016/j.memsci.2012.08.029

[29] Nandiyanto, Asep Bayu Dan, Rosi Oktiani, and Risti Ragadhita. "How to read and interpret FTIR spectroscope of organic material." *Indonesian Journal of Science and Technology* 4, no. 1 (2019): 97-118. https://doi.org/10.17509/ijost.v4i1.15806

[30] da Silva, Danielli Alessandra Reino Olegário, Luana Carolina Bosmuler Zuge, and Agnes de Paula Scheer. "Preparation and characterization of a novel green silica/PVA membrane for water desalination by pervaporation." *Separation and Purification Technology* 247 (2020): 116852. https://doi.org/10.1016/j.seppur.2020.116852

[31] Elham, Asrari, Tavallali Hossein, and Hagshenas Mahnoosh. "Removal of Zn (II) and Pb (II) ions using rice husk in food industrial wastewater." *Journal of Applied Sciences and Environmental Management* 14, no. 4 (2010): 159-162. https://doi.org/10.4314/jasem.v14i4.63306

[32] Harun, Zawati, Muhamad Fikri Shohur, Mohd Riduan Jamalludin, Muhamad Zaini Yunos, and Hatijah Basri. "Hydrophilicity effect of rice husk silica on mixed matrix PSF membrane properties." *Jurnal Teknologi* 70, no. 2 (2014): 15-18. https://doi.org/10.11113/jt.v70.3428

[33] Dakhil, Ihsan Habib. "Adsorption of lead from industrial effluents using rice husk." *International Journal of Engineering and Management Research (IJEMR)* 5, no. 1 (2015): 109-116.

[34] Fan, Linhua, John L. Harris, Felicity A. Roddick, and Nic A. Booker. "Influence of the characteristics of natural organic matter on the fouling of microfiltration membranes." *Water Research* 35, no. 18 (2001): 4455-4463. https://doi.org/10.1016/s0043-1354(01)00183-x

[35] Jhaveri, Jainesh H., and Z. V. P. Murthy. "A comprehensive review on anti-fouling nanocomposite membranes for pressure driven membrane separation processes." *Desalination* 379 (2016): 137-154. https://doi.org/10.1016/j.desal.2015.11.00