Evaluation of Crosslinking Degree on the Integral Membrane by using Rice Husk Ash (RHA)

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ABSTRACT

Rice husk ash (RHA) is a waste product from the harvesting and processing of rice that contains a high quantity of silica, approximately 95% after combustion. Membrane technology is being developed to remove impurities like heavy metals and dyes as the natural environment deteriorates and water supplies become scarce. Many researchers and developers are now adopting this type of technology. However, it has various drawbacks such as being costly and easily being fouled during the separation process. Therefore, matrix modification of the membrane should be carried out to mitigate these problems. The incorporation of fillers from biomass materials is one of the ways. This research aims to demonstrate the significant effect of incorporating the extracted silica from rice husk ash (RHA) in a polymer-based membrane from a blend of Polyvinyl alcohol/chitosan/polysulfone on the membrane characteristics and antifouling properties. The membranes were prepared by using a phase inversion technique by the incorporation of silica from rice husk ash (RHA) at various concentrations such as 5 wt.%, 7.5 wt.%, and 10 wt.% with a fixed amount of the polymer blends. The results showed that the integral membrane M4 with 10wt.% silica has the best hydrophilicity properties and possesses excellent antifouling properties, which were portrayed through the greater value of pure water flux (PWF) of 20.38 L/m^2.h and the highest flux recovery ratio of 76.32%. This study has proven the potential utilization of rice

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husk ash to enhance the membrane properties for industrial wastewater treatment.

Keywords: *Rice Husk Ash (RHA); Polymer-based Membrane; Phase Inversion Technique; Antifouling; Silica*

Introduction

Water pollution is a critical issue that needs to be tackled because there is a large generation of wastewater effluent which can lead to environmental pollution. According to the United Nations World Water Development Report published in 2018, about 6 billion people throughout the world will encounter clean water scarcity by 2050, where the demand for clean water is growing at a rate of 1% annually [1]. Heavy metals, hydrocarbons, dyes, nitrogenous chemicals, pharmaceutical residues, detergents, and phosphorus are the most prevalent chemical contaminants in wastewater. These contaminants result in difficulty in treating wastewater since the contaminants are easily adsorbed to the suspended particles in water, then settled in the riverbed, and pose harm to the ecology. As a result, modern treatment for wastewater such as membrane separation technology (MST) consisting of polymeric membranes and ceramic membranes is being used to solve this problem.

The membrane is a selective barrier that is employed in the separation process to separate the presence of various phases by allowing some components to flow through while others are retained. When a membrane contains its driving force, which is a gradient of pressure, chemical, or electrical potential across the membrane, the separation process will occur. Membrane technology has been revived as a promising technology for removing impurities from effluent discharged from wastewater industries because it does not require phase change or chemical addition, making it a viable alternative to traditional wastewater treatment techniques such as distillation, precipitation, coagulation, and flocculation [2]. In wastewater treatment, the membrane is used to remove the pollutants such as suspended solids or dissolved solids to clean the water before being discharged into groundwater [3]. Membrane technology is also utilized in many types of industries such as gas separation, food processing, and protein purification.

As a biomass material, rice husk ash (RHA) is widely used as a filler in a polymeric membrane, where the formulated membranes are subsequently used in wastewater treatment [4]. Based on the findings from Alias et al. [5], the extracted silica from rice husk was used for fouling mitigation in the membranes since the amorphous structure from RHA creates strong hydrophilicity and improved the separation performance in terms of pure water flux, rejection of humic acid, and fouling mitigation for applications in liquid separation and water treatment respectively. Rice husk (RH) is a waste from

agriculture that is abundantly generated year by year, especially in the countries that produce rice as their staple food. As reported in World Atlast, rice is a staple food all over the world and Asia countries are the largest consumers of rice [6]. Rice husk contains approximately 20 wt.% of ash generated when it is burnt at a certain temperature. The silica dioxide in the ash offers a function to improve the wettability of the polymeric membranes to enhance the antifouling properties [4]. This study aims to extract the silica from rice husk ash by using a modified acid leaching process, and it was subsequently incorporated into the membrane formulation through a sol-gel reaction. The properties of the formulated membrane were evaluated in terms of functional group, thermal stability, and surface morphology, and the performance of the membrane was tested through pure water flux (PWF) and antifouling analysis. The effort to utilize rice husk ash (RHA) in the membrane fabrication will be able to reduce the amount of rice husk ash (RHA) generated as waste from the incinerator because the higher airborne concentration of rice husk ash (RHA) can cause a problem to the human respiratory system.

Methodology

Material

In the silica extraction method, the rice husk ash (RHA), 37% purity of hydrochloric acid (HCl), and 1N sodium hydroxide (NaOH) were used. The RHA was obtained from BT Science Sdn. Bhd., Selangor Malaysia. HCl and NaOH were purchased from R&M Chemicals, Subang, Malaysia. Polyvinyl alcohol (PVA) 87-89% hydrolyzed with a molecular weight average of 85000-124000, dimethyl sulfoxide (DMSO), and chitosan were purchased from Sigma-Aldrich (M) Sdn. Bhd., Merck Sdn. Bhd. and Aman Semesta Enterprise Sdn. Bhd., respectively. Polysulfone beads with 22000 molecular weights were purchased from Sigma-Aldrich (M) Sdn. Bhd. N-Methyl-Pyrrolidone (NMP) and polyethylene glycol (PEG 400) were purchased from Merck Sdn. Bhd.

Extraction of silica from rice husk ash (RHA)

The 50 g of rice husk ash (RHA) was mixed with 250 g of deionized water and 8 g of 37% purity of hydrochloric acid (HCl). This mixture was heated at 90 ºC and stirred at the speed of 650 rpm for 1 hour. Then, this mixture was left at room temperature before being filtered using a Smith filter paper to obtain a cleaned RHA. This cleaned RHA residue then was treated with 250 g of 1 N NaOH for the acid-leaching process by heating the solution to 80 ºC while stirring at 600 rpm for 1 hour. After that, the solution was filtered by using a Smith filter paper to extract sodium silicate.

Preparation of 2wt% of acetic acid

To prepare 2 wt.% of the acetic acid aqueous solution, 2 g of glacial acetic acid was added into 98 g of distilled water and the mixture was stirred at 150 rpm for 1 hour [7].

Preparation of polymer blend solution

To prepare the polymer blend PVA/chitosan, both solutions were prepared separately. 10 g of polyvinyl alcohol (PVA) was added into 90 g of dimethyl sulfoxide (DMSO) followed by heating at 90 ℃ while stirring at 400 rpm for 4 hours until the solution become homogeneous. Then, 0.02 g of chitosan was dissolved into 99.98 g of 2 wt% aqueous acetic acid solution followed by stirring at 400 rpm and heating at a temperature of 90 ℃ for 4 hours [8].

Preparation of polysulfone solution

To prepare 13 wt.% of polysulfone solution, 13 g of polysulfone beads pellets were dissolved in the 82 g of N-Methyl-Pyrrolidone (NMP). Next, 5 g of polyethylene glycol (PEG) was added to the mixture before it was stirred at a speed of 400 rpm with heating at 60 ℃ for 6 hours. Then, this solution was left at room temperature.

Preparation of hybrid solution with rice husk ash (RHA)

The hybrid solution was prepared by mixing 50 g of the prepared polyvinyl alcohol (PVA) with 50 g of chitosan solution, followed by the addition of 5 wt.% of the extracted sodium silicate and 2 g of hydrochloric acid. The mixture was heated at 60 ℃ for 7 hours with continuous stirring at 400 rpm. The solution was cooled at room temperature before the membrane making process. The above procedure was repeated with the 7.5 wt.%, 10 wt.% sodium silicate, and without sodium silicate respectively.

Preparation of membrane

In the last stage, the membrane was prepared by adding 1 g of hybrid solution to 50 g of polysulfone solution. Then, this mixture was stirred at 80 ℃ at 750 rpm until the solution turned into a homogeneous mixture. After that, the solution was left at room temperature for 1 hour before proceeding to the casting process. In the casting process, the thickness of Baker's film applicator was adjusted to 100 µm. The produced film was left at room temperature for 30-40 seconds before it was immersed in a large amount of water for a coagulation process. This membrane was left in the water for 24 hours. Then, it was dried at room temperature for 2 days before the characterization process was conducted. Table 1 depicts the formulations used in this study.

Table 1: Formulation of integral membrane

Composition (g)				
Membrane	PVA	CS	RHA	HCl
M1	50	50	-	2
M2	50	50	5	2
M3	50	50	7.5	2
M4	50	50	10	2

Membrane characterization

The formulated membranes were characterized in terms of the functional group by using FTIR, and surface morphology by using FESEM.

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier-Transform Infrared Spectroscopy (FTIR) is an analytical technique used to identify the organic, polymeric, and inorganic materials in a membrane film by determining the presence of the peak of wavenumber in the graph of transmittance versus wavenumber. In this study, the observation was done with the wave number that ranges from 4000-400 cm^{-1}. The equipment used was PerkinElmer/TGA/SDTA851 [9].

Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscopy (FESEM) is an analysis technique to identify the structure of the membrane. The samples were coated with the platinum coating to obtain the perceived images and to avoid the charging effect. Then, the images were taken by magnifying them at 10 k until 20 k, and the voltage was accelerated at 5 k. The configuration of samples was obtained by using electron dispersive spectroscopy (EDS) attached to the machine. The model used was Jsm-7600f Joel [10].

Performance test

The performances of the formulated membranes were evaluated in terms of pure water flux and antifouling analysis.

Pure Water Flux (PWF)

For pure water flux analysis, a membrane filtration rig with a dead-end mode was used. The membrane was cut into a circular shape with a surface area of 19 cm^2 before placing it onto the porous disk of the stainless-steel filtration cell. Then, 300 mL of deionized water was filled inside the cell as the feed solution before applying a nitrogen gas at 6 bars of pressure for the filtration process. The flux was recorded at 15-minute interval time for 1 hour of duration and the water flux value was measured by using Equation 1.

$$J_w = \frac{Q}{A\Delta T} \quad (1)$$

where, Jw is the pure water flux (L.m^2.h), Q is the volume of permeate (L), A is the area of the membrane (m^2), and Δt is the sampling time (h).

Antifouling analysis

The antifouling analysis was performed to determine the antifouling resistance of the membrane and it was carried out through 3 stages at 10 bars of pressure [11]. Humic acid was used as the foulant model. In the first stage, the deionized water was used as the feed solution and the flux was recorded after 30 minutes (J_{w1}). In the second stage, the humic acid solution that consists of 0.2 g of humic acid (HA) in 1 L of 1000 ppm sodium hydroxide (NaOH) was then used as the feed solution, where the permeation was conducted for 120 minutes. Every 20 minutes, the permeate volume was recorded, and the final flux was recorded as J_{HA}. In the third stage, the membrane was backwashed to remove the adsorbed foulant for 30 minutes by immersing it in the deionized water with a stirring speed of 150 rpm. Then, the first step was repeated and the final flux was recorded as J_{W2}. Lastly, the flux recovery ratio (FRR), reversible fouling ratio (RFR), irreversible fouling ratio (IFR), and relative flux decay (RFD) were calculated using the equations below [12]:

$$FRR = \frac{J_{W2}}{J_{W1}} \times 100 \quad (2)$$

$$RFR = \frac{J_{W2} - J_{HA}}{J_{W1}} \times 100 \quad (3)$$

$$IFR = \frac{J_{W1} - J_{W2}}{J_{W1}} \times 100 \quad (4)$$

$$RFD = \frac{J_{W1} - J_{HA}}{J_{W1}} x100 \quad (5)$$

Result and Discussion

Membrane characterization

Fourier Transform Infrared Spectroscopy (FTIR)

Figure 1 demonstrates the Fourier Transform Infrared Spectroscopy (FTIR) for all formulated membranes. Based on the figure, the similar peaks existing in

the spectra are illustrating the fingerprint region that composes of polysulfone, polyvinyl alcohol, and chitosan for membrane M1 with an additional peak that corresponds to rice husk ash components for M2, M3, and M4 ranging from 600 cm^{-1} to 1500 cm^{-1}. For the membranes incorporated with sodium silicate from RHA, the crosslinking process resulted in two peaks emergence at 1015 cm^{-1} and 1105 cm^{-1} that was corresponding to the absorption bands from organic siloxane (Si- O- Si) and (Si- O- C) respectively, where these peaks are very intense for the M4 membrane due to the highest crosslinking process involved [5], [13]. For M1, the peak at 1105 cm^{-1} is attributed to the presence of C-O-C from the blended polymer [6]. Membrane M1 also shows the strongest bonds of C=O at 2700 cm^{-1} to 2800 cm^{-1} reflecting that the acetaldehyde group was not completely transformed and it indicates no crosslinking process occurs for pure membrane [14].

The existence of the stretching hydroxyl (O-H) symmetric group appears at 3369 cm^{-1} and 3560 cm^{-1} respectively for all membranes, where strong intensity represents great hydrophilicity of the membrane surface and the anticipated increase in the water permeation performance of the membrane [15]. These bands were attributed to the vibration of hydroxyl groups that bonded to the chitosan, polyvinyl alcohol, and carbon of silica, respectively. For membranes M2 to M4, the stretching vibrations of a secondary amine (N-H) from sodium silicate overlapped with the adsorption band of a hydroxyl group (O-H) at the wavenumber of 3422 cm^{-1}. As a result of crosslinking reaction, the intensity of the peak shows a decreasing trend due to the interaction of silica from sodium silicate with amide and hydroxyl groups of the blended organic polymers [16]. The enhanced integral stability of the membranes was expected to happen as a result of the rapid crosslinking reaction especially on membrane M4 [17].

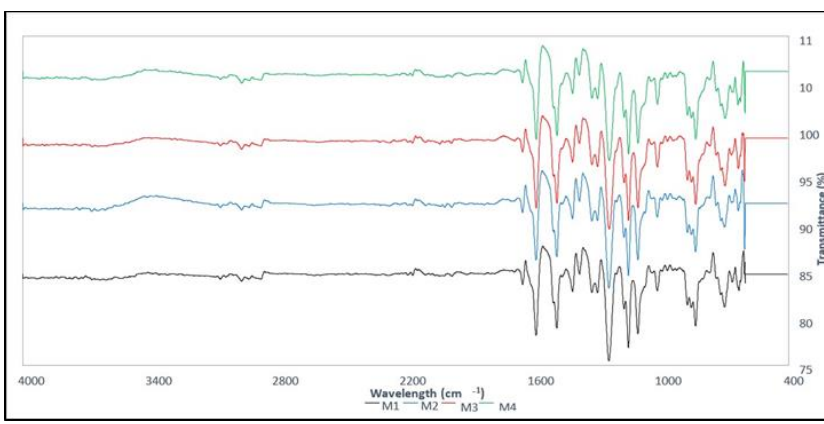
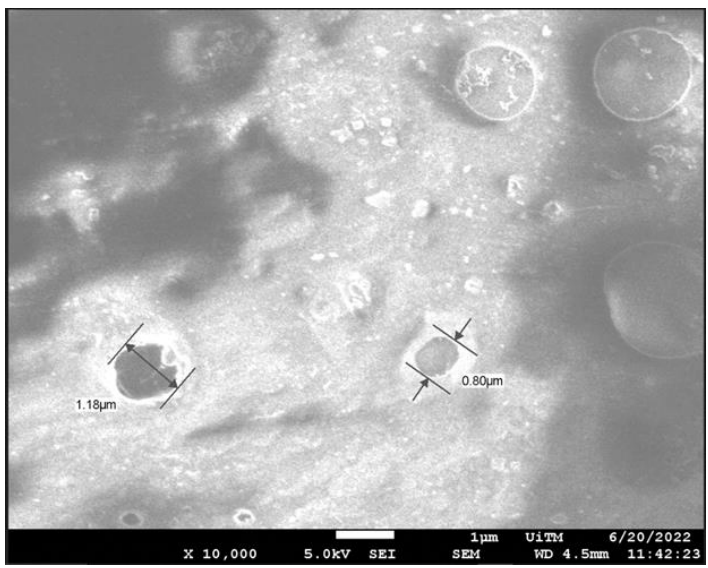


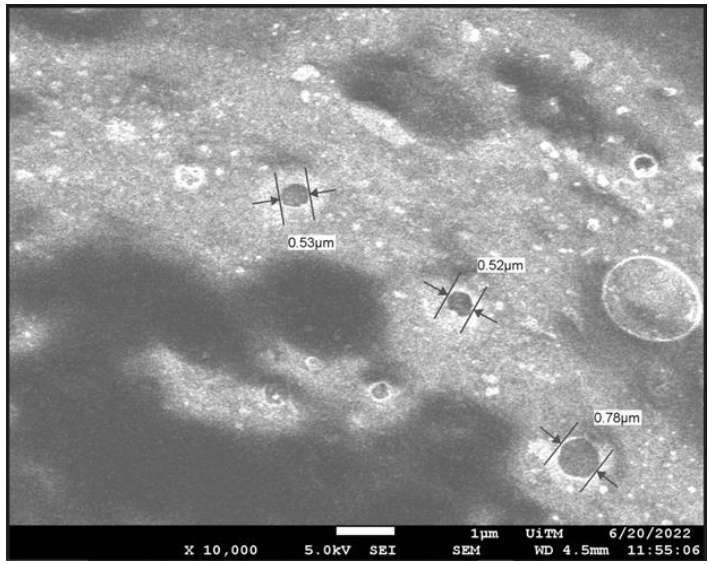
Figure 1: FTIR spectra for membranes

Field Emission Scanning Electron Microscopy (FESEM)

Figure 2 shows the images of the surface morphology of each membrane from FESEM analysis. Based on the images, the pore size of each membrane was extracted and presented in Table 2. As can be seen from the table, the mean pore size of the membrane decreases with the increasing amount of silica incorporated in the membrane. The cross-linking process between the -OH group of the blended polymers with the silica resulted in a dense structure of the membrane with the increment of small finger-like pores on the membrane surfaces. The dense structure provides a significant adsorption area for greater rejection of ions particularly for heavy metal ion rejection as a potential application of the formulated membrane [18]. Furthermore, it can be observed that many microvoids are present in Figures 2b until 2d due to the increased silica content incorporated in the membrane, where it is highly preferable for rejection properties [14]. Besides tightening the surface of the membrane, the addition of silica from RHA has been reported to create high hydrophilicity properties of the membrane [18].



(a)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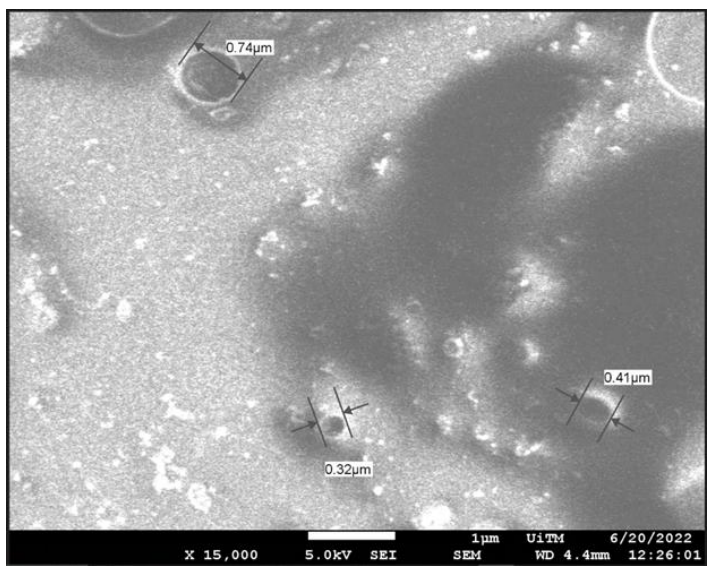
(b)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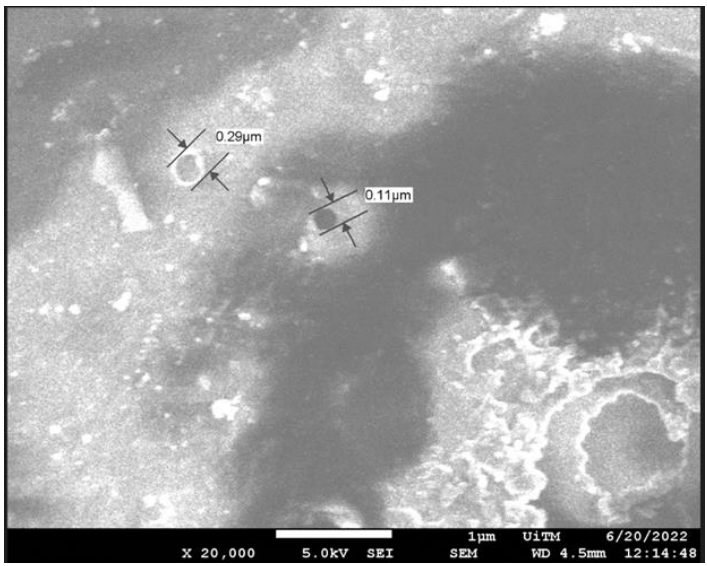
(c)



(d)

Figure 2: FESEM images for (a) M1, (b) M2, (c) M3, and (d) M4

Table 2: Formulation of integral membrane

Membrane	Mean pore size
M1	0.99
M2l	0.61
M3l	0.49
M4l	0.20

Performance test

Pure water flux

Figure 3 illustrates the performances of each membrane in pure water flux (PWF) analysis. Based on the figure, the value of permeation flux for M1, M2, and M3 shows a higher flux for the first 30 minutes as compared to M4. However, the fluxes for these three membranes (M1, M2, and M3) declined significantly after 30 minutes while M4 shows a constant flux value throughout the one-hour filtration period. Membrane M4 portrays good surface hydrophilicity that contributes to the antifouling characteristic as less concentration polarization was observed from the plot.

Furthermore, M4 incorporated with the highest concentration of sodium silicate has greater water permeation compared to membrane M1 because the silica itself possesses strong hydrophilicity properties [14]. This hydrophilicity

also corresponds to the amounts of microvoids exhibited on the membrane surface as observed by FESEM because more microvoids will allow greater amounts of water molecules to pass through the membrane.

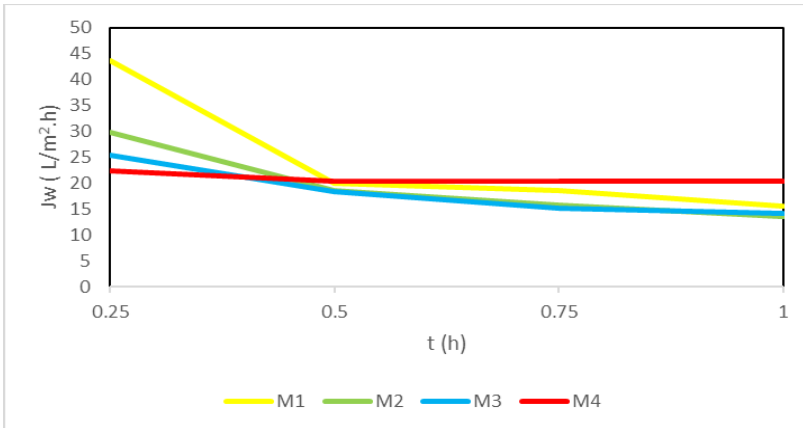


Figure 3: Permeation of pure water for all membrane

Antifouling analysis

Based on Table 3, although all the recorded fluxes for M1 were the highest, it has the lowest FRR and RFR, and the highest RFD and IFR, which show poor antifouling behaviour of the membrane. On the other hand, the flux recovery ratio (FRR) for membranes incorporated with sodium silicate showed higher values with M4 marked as the highest. The flux recovery rate is a vital parameter in the evaluation of antifouling properties because a higher flux recovery rate suggests a better antifouling performance and better hydrophilicity of the membrane, where the preferable value is ranging between 40% to 90 % [19]. It was proven from the obtained result that the membrane with the highest amount of silica shows stronger hydrophilicity properties in the membrane resulting in the highest antifouling ability. The relative flux decay (RFD) shows a declining trend with the increasing amount of incorporated silica inside the membrane. The membrane M4 has the lowest value of RFD which is 66.84%, which further indicates the presence of a hydration layer on the membrane's surface. As for IFR (irreversible flux ratio), it measures the degree of the particles attached to the membrane that causes clogging, which cannot be subsequently removed by the physical cleaning of the membrane unlike the RFR (reversible fouling ratio). As can be observed from the table, the value of RFR decreased with the incorporation of sodium silicate from 5 wt.% to 10 wt.%, indicating a better resistance of the membranes against the fouling exerted by the humic acid. There was a trend shown from the result that when the FRR increases with the incorporation of

sodium silicate, IFR showed a declining trend which indicates the clogging of foulant inside the porous structure of the membrane can be removed easily, particularly for M4. Furthermore, the coarse structure of membranes due to the incorporation of silica also facilitates the removal of the foulant from the membrane by enhancing the internal hydraulic flow across the membrane [20]. Based on the smaller pore size displayed by M4 as shown through FESEM analysis, it will provide a larger area for water permeation through the membrane [21], and most importantly membrane M4 has the best antifouling properties.

Table 3: Antifouling properties of membranes from various formulations

Membrane code	J_{W1}	J_{HA}	J_{W2}	FRR (%)	RFD (%)	RFR (%)	IFR (%)
M1	49	11.4	27	55.10	76.73	31.84	44.90
M2	35	7.5	25	71.43	78.57	50	28.57
M3	23	7.4	17.1	74.35	67.83	42.17	25.65
M4	19	6.3	14.5	76.32	66.84	43.16	23.68

Conclusion

It is apparent from this research that an extraction process of sodium silicate from rice husk ash was successfully conducted. The incorporation of sodium silicate has resulted in the enhanced hydrophilicity and antifouling behaviour of the membranes where membrane M4 with 10 wt.% silica displayed the best performance. These findings have demonstrated the value addition to rice husk as the biomass material and the formulated membranes have the potential to be used for wastewater treatment.

Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

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Conflict of Interests

All authors declare that they have no conflicts of interest

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