Original Article

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Fabrication and characterization of SiO_2-embedded castor oil-based membrane (*Ricinus communis* L) for aqueous Fe adsorption

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Abstract

Castor seed oil (*Ricinus communis* L) can be utilized for the manufacturing of membranes reacted with toluene diisocyanate (TDI). The aim of this study was to examine the effect and membrane characteristics of castor seed oil with a combination of silica used as an adsorbent, with a combination of silica (SiO2) 0.5 mg, 1 mg, 1.5 mg, and 2 mg. The initial analysis of the membrane was carried out with a swelling test of 1.5 mg of silica variation to obtain the most ideal result of 200%. The best chemical resistance characterization occurred at a variation of 1.5 mg of silica. Further characterization, particularly the FTIR test, thermal test, mechanical test, and SEM test, showed that there were Si-O groups and amine groups (NH2), and on the membrane there were groups OH, C=O, and NH. The thermal characterization of the silica membrane (1.5 mg) gave the greatest residue concentration, measuring 3.2%. In mechanical terms, the silica membrane has a higher elongation value than the membrane. In SEM characterization, the silica membrane has holes and is solid. This study indicated that the highest drop in Fe metal occurred at an immersion period of 6 hours with a combination of silica with a flux value of 9.25 L/m2 hour and a rejection value of 0.098%.

Keywords: Castor oil, *Ricinus communis*, SiO2, Fe metal, adsorption

Introduction

Membrane technology has made significant progress in developing high-performance membranes for various separation processes. Membranes offer several advantages over other separation methods, as they enable a simpler process without the need for additional chemical materials. Further, membrane filtration can be combined with other processes, does not involve phase changes, requires low energy, can operate continuously, and does not need a large installation space. As a result, membranes have become widely utilized across multiple industries, including textiles, food, beverages, and more [1].

Membranes can be fabricated from both organic polymers and inorganic materials, although organic polymers are the most commonly used due to their straightforward manufacturing process, natural abundance, and ease of procurement [2]. One example of a membrane produced from an organic polymer is a polyurethane membrane synthesized from castor oil. Castor oil has hydroxyl moieties (O-H) in a single molecule. Thus, it can be used as the polyol source and reacted

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with isocyanate (R-N=C=O). TDI, on the other hand, is the most commonly used isocyanate in polyurethane synthesis due to its chemical property of being reactive. In previous studies, the TDI has been utilized to fabricate polyurethane polymers via the one-shoot method without requiring the addition of catalysts. However, polyurethane membrane fabricated using the aforementioned materials has several drawbacks such as having non-uniform surface and low adsorptive ability.

To improve the castor oil-based polyurethane membrane, silica oxide can be introduced to the membrane matrix. A previous study revealed that the addition of silica to a polymeric membrane could enhance its chemical and physical properties [3]. The addition of silica could improve the membrane porosity, thus increasing its ability to separate macromolecules from an aqueous solution [3]. A study fabricating polyether ether sulfone ketones membrane suggested that the addition of SiO_2 (3% w/w total membrane) increased the membrane conductivity while lowering its permeability [4]. A rice husk-derived SiO_2 incorporated to a chitosan polymer has been reported to increase the adsorption of biodiesel impurities [5]. Therefore, our research group hypothesized that the addition of SiO_2 into castor oil-based polyurethane membrane could increase its ability to remove pollutants such as heavy metals. Fe is particularly of our interest due to its frequent pollution occurrences and complex speciation in the water [6]. Among reported adsorbents designated to separate aqueous Fe, none has reported the castor-oil based polyurethane membrane with the addition of SiO_2 [7-9]. The aim of this study was to observe the effect of the SiO_2 to the characteristics and adsorptive ability of castor oil-based polyurethane membrane.

Methods

Material

Materials used in the research were toluene diisocyanate (TDI), distilled water, castor oil (*Ricinus communis* L), acetone (C_3H_6), silica powder (SiO_2), sulfuric acid (H_2SO_4), potassium hydroxide (KOH), sodium chloride (NaCl), and ethanol. Otherwise stated, all chemicals were analytical grade quality and procured from Merck (Selangor, Malaysia). Castor oil was procured from a local provider in Medan, Indonesia, and distilled water was produced in the laboratory.

Membrane preparation

We used the inversion phase method to fabricate the castor oil-based polyurethane membrane, where the raw materials were prepared in the liquid phase. In general, the membrane preparation followed the protocol reported previously [10]. Initially, the SiO_2 powder was dispersed homogeneously in castor oil (castor oil) with a magnetic stirrer (250 rpm; room temperature). The SiO_2 was varied from 0 g, 0.5 g, 1 g, 1.5 g, and 2 g, in each batch of the membrane preparation. Thereafter, the TDI (0.85 g) was added and the mixture was stirred for 15 min (250 rpm; 45 °C). The casting solution was subsequently sonicated with the addition of acetone (2 g) to remove the formed air bubbles. The solution was then casted on a glass plate and cured using an oven (40°C; 24 h). The produced membrane was cut into a small circular shape (diameter: 2.3 cm) before used for characterization.

Characterization membrane

The swelling degree of the membrane was determined by comparing its weight before and after 24 hours of soaking in distilled water. The percentage change in weight was used to express the swelling degree. The chemical resistance was tested by soaking polyurethane samples in various chemical solutions. The membrane was initially oven-dried at 115°C for 15 minutes, cooled, and weighed. The samples were then immersed in solutions containing NaCl (10%), KOH (3%), H2SO4 (3%), and ethanol (25%), sealed, and stored for 7 days. After this period, the samples were retrieved, washed with distilled water, dried again at 115°C for 15 minutes, cooled, and reweighed. The weight change indicated the stability of the sample in each solution. The functional group of the membrane was characterized by the Fourier transform infrared (FT-IR) spectrometer—Shimadzu Prestige (Kyoto, Japan). The thermal behaviors of the membrane were observed through thermal gravimetry analysis (TGA) on Shimadzu DTG-60 Thermal Gravimetric Analyzer run from 50°C to 600°C at 40°C/min under dynamic nitrogen atmosphere (flow rate: 20

mL/min). The surface morphology of the membrane was studied under a scanning electron microscope (SEM) Jeol Jsm 6360 LA (Tokyo, Japan). Mechanical properties were determined based on the analysis using Universal Testing Machine HT8503 (Hung Ta Instrument Co., Ltd., Taichung, Taiwan).

Batch adsorption

For batch adsorption studies, the resultant membranes were cut into square shapes sized 3×3 cm^2. The artificial Fe-containing wastewater was prepared by diluting $FeCl_3$ in deionized water. The membrane was immersed in an Erlenmeyer containing 50 mL of the artificial Hg solution. The contact time was varied from 0 h, 2 h, 4 h, to 4 h. Thereafter, the membrane was removed from the Erlenmeyer and the filtrate was analyzed using atomic absorption spectroscopy (AAS; Shimadzu AA-7000, Kyoto, Japan) for the remaining Hg. The study was conducted in triplicate, and the data are presented as the average value.

Results

Characteristics of the membrane

Swelling degree

The swelling degrees of the resultant membranes with a variation of the SiO_2 contents are presented in **Table 1**. The swelling degree of the polyurethane membrane increased with the addition of SiO_2. Without SiO_2, the membrane exhibited a swelling degree of 20%. With 0.5 g and 100 g of SiO_2, the swelling degree increased to 50% and 100%, respectively. The swelling degree further increased to 200% and 220% with 1.5 g and 2 g of SiO_2 additions, respectively.

Table 1. Effect of the SiO_2 addition to the swelling degree of the castor oil-based polyurethane membrane

SiO_2 (g)	Initial weight (g)	Final weight (g)	Swelling degree (%)
0	0.05	0.06	20
0.5	0.02	0.03	50
1	0.02	0.04	100
1.5	0.05	0.15	200
2	0.03	0.096	220

Chemical resistance

The weight loss of the polyurethane membrane with varying SiO_2 contents in different chemical solutions is presented in **Figure 1**. The neat polyurethane membrane had a weight loss of 3.30% in NaOH, 0.77% in KOH, 1.45% in ethanol, and 0.31% in H_2SO_4. The addition of 0.5 g SiO_2 reduced 2.34%, 2.73%, 1.94%, and 0.37% of the membrane weight when soaked in NaOH, KOH, ethanol, and H_2SO_4, respectively. At 1 g of SiO_2, the weight loss was 1.45% in NaOH, 2.27% in KOH, 2.74% in ethanol, and 0.50% in H_2SO_4. Weight gains of 1.72% and 2.04% were observed in a membrane with 1.5 g SiO_2 addition when soaked in NaOH and KOH, respectively. At the same SiO_2 content, the weight loss in ethanol was found to be 0.95%, while no loss was observed in H_2SO_4. Further addition of the SiO_2 (2 g) yielded a weight loss of 0.83%, 31.3%, and 2.12% when soaked in NaOH, KOH, and ethanol, respectively. Soaking in H_2SO_4 for 7 days did not change the weight of the membrane with 2 g SiO_2.

Functional group characteristics

FTIR spectroscopy was used to analyze and identify the functional groups of the resultant membranes, where the spectra are presented in **Figure 2**. Based on the spectra of the membrane with and without SiO_2, a broad absorption band was observed at a range of 3991.86 to 3782.57 cm^{-1} assigned to the presence of O-H and N-H groups. Absorption bands from 2800–3000 cm^{-1} confirmed the presence of C-O-NH_2 groups. The emergence of these absorption bands corresponds to the formation of urethane linkage. Supposedly, the Si-O-Si vibrations can be observed in the range of 877.65–959.63 cm^{-1}. However, the absence of the absorption bands in the wavenumber range is indicative of the presence of the SiO_2 particles that were embedded inside the membrane structure.

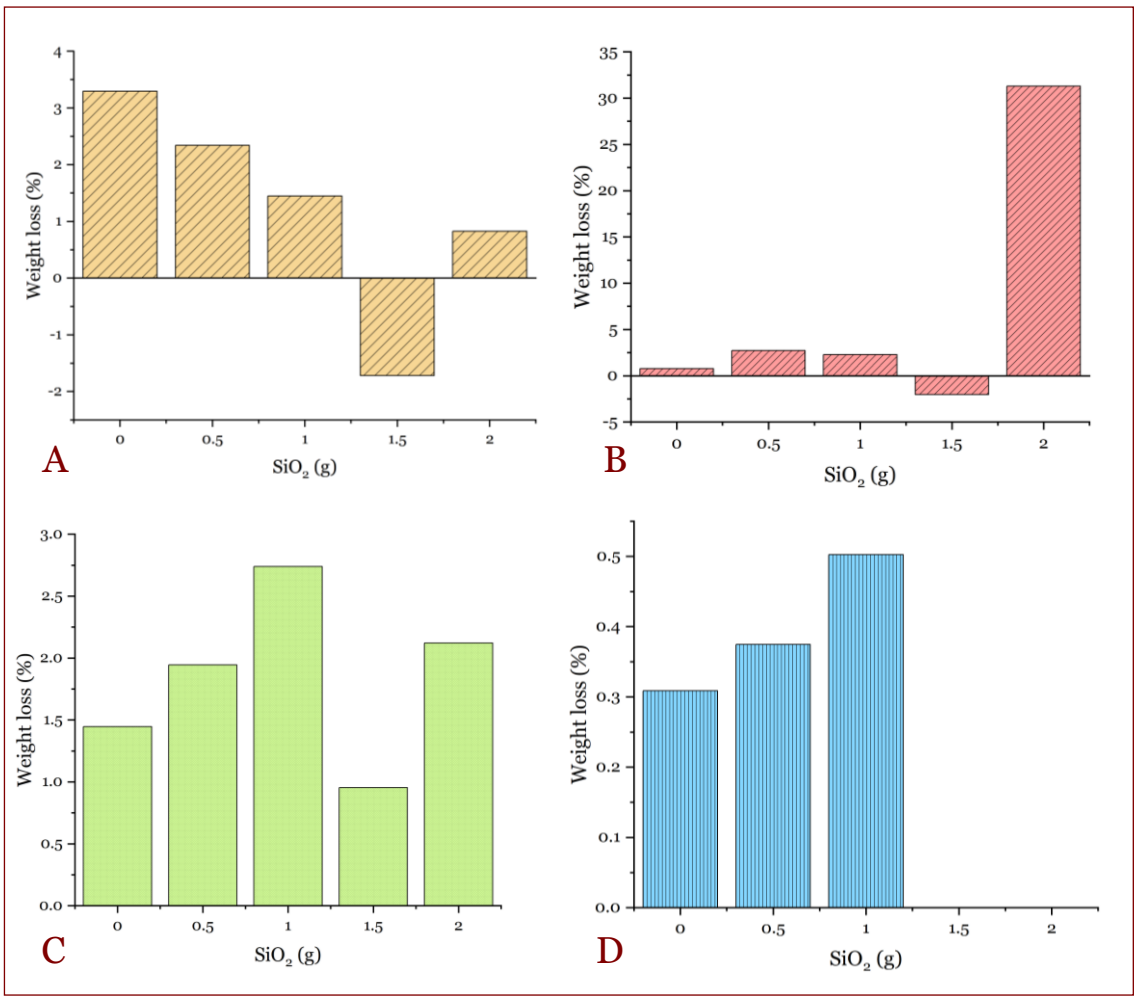

Figure 1. Effect of the SiO_2 addition on the chemical resistance of the membranes against NaOH (A), KOH (B), ethanol (C), and H_2SO_4 (D).

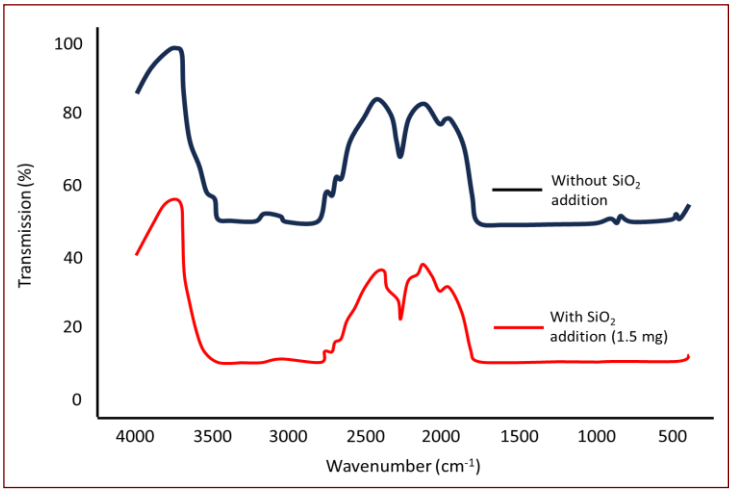

Figure 2. FTIR spectra of the castor oil-based polyurethane membranes with and without the addition of SiO_2.

Thermal behavior

The thermal analysis of castor oil-based polyurethane membranes, both with and without silica, are presented in **Table 2**. The initial decomposition of the neat polyurethane membrane occurred between 258°C and 332°C. With the addition of SiO_2, this range shifted slightly, starting at 270°C and ending at 329°C. The shifted temperature range was also observed in the second decomposition. In the neat membrane, the decomposition is observed at the range between 414°C

and 480°C. The addition of SiO_2 also increased the temperature range (406°C to 511°C). An increase in temperature was also observed at 50% weight loss from 410°C for the membrane without SiO_2 and 422°C for the membrane with SiO_2. Similarly, the temperature at 90% weight loss was 480°C without SiO_2 and 490°C with SiO_2. Additionally, the residue percentage after decomposition was 3.2% for the membrane without SiO_2 and 1.8% for the membrane with SiO_2.

Table 2. Thermal gravimetry analysis (TGA) profiles of the castor oil-based polyurethane membranes with and without the addition of SiO2

TGA parameters	Temperature (°C)	
	Without SiO_2	With SiO_2 (1.5 mg)
T1 (initial decomposition temperature)		
Start	258	270
End	332	329
T2 (second decomposition temperature)		
Start	414	406
End	480	511
T 50% (temperature at 50% weight loss)	410	422
T 90% (temperature at 90% weight loss)	480	490
Residue (%)	3.2	1.8

Mechanical strength

The mechanical strength parameters of castor oil-based polyurethane membranes with and without SiO_2 are presented in **Table 3**. The maximum force the membrane could withstand was significantly higher with SiO_2 as compared to those without SiO_2 (8.0 kgf versus 5.9 kgf). The 0.2% yield strength also improved with SiO_2, increasing from 0.24 kgf/mm² to 0.41 kgf/mm². Conversely, the yield strength of membranes without SiO_2 is considerably higher at 3.39 kgf/mm² compared to just 0.89 kgf/mm² with SiO_2. The SiO_2-embedded membrane had a higher tensile strength as compared to that of the neat membrane (0.56 kgf/mm² versus 1.14 kgf/mm²). However, elongation was reduced in the presence of SiO_2, decreasing from 103.25% to 49.38%.

Table 3. Mechanical properties of the polyurethane membrane with and without SiO_2

Mechanical strength parameters	Without SiO_2	With SiO_2 (1.5 mg)
Area (mm²)	10.5	7.0
Maximum force (kgf)	5.9	8.0
0.2% yield strength (kgf/mm²)	0.24	0.41
Yield strength (kgf/mm²)	3.39	0.89
Tensile strength (kgf/mm²)	0.56	1.14
Elongation (%)	103.25	49.38

Surface morphology

The SEM images show differences in surface morphology between the neat polyurethane membrane and the polyurethane membrane embedded with SiO_2 (**Figure 3**). The membrane with SiO_2 addition exhibited distinct surface features with visible particles or clusters measuring approximately 2.37 µm and 1.35 µm. The different particle sizes suggest the presence of SiO_2 as individual particles or agglomerates, where they appeared in irregular shapes. As the consequence, the membrane with SiO_2 addition has a more heterogeneous surface structure. In contrast, the neat polyurethane membrane displayed a smoother and more homogeneous surface with no visible particulate features at the same magnification of 5,000× (**Figure 3**).

Adsorptive removal of Fe

The removals of Fe^{3+} ions by castor oil membranes with varying SiO_2 content over different contact times (0, 2, 4, and 6 hours) are presented in **Figure 4**. After 2-hour batch adsorption, regardless of SiO_2 content, there was a reduction in Fe^{3+} concentrations, with values ranging from 9.32×10^{-4} to 9.25×10^{-4} mol/L for lower SiO_2 contents (0–1.5 g) and 9.26×10^{-4} mol/L for the membrane with 2 g SiO_2. At 4 hours, the Fe^{3+} concentration continued to decrease, with the membrane without SiO_2 showing adsorption of 9.16×10^{-4} mol/L and the membrane with 2 g SiO_2 showing a further reduced concentration of 9.15×10^{-4} mol/L. After 6 hours, there was minimal change in Fe^{3+} removal for membranes with 0 g and 0.5 g of SiO_2. Membranes with higher SiO_2

content showed a gradual decrease in Fe^{3+} concentration, reaching 9.12×10^{-4}, 9.04×10^{-4}, and 9.08×10^{-4} mol/L for 1 g, 1.5 g, and 2 g SiO_2, respectively.

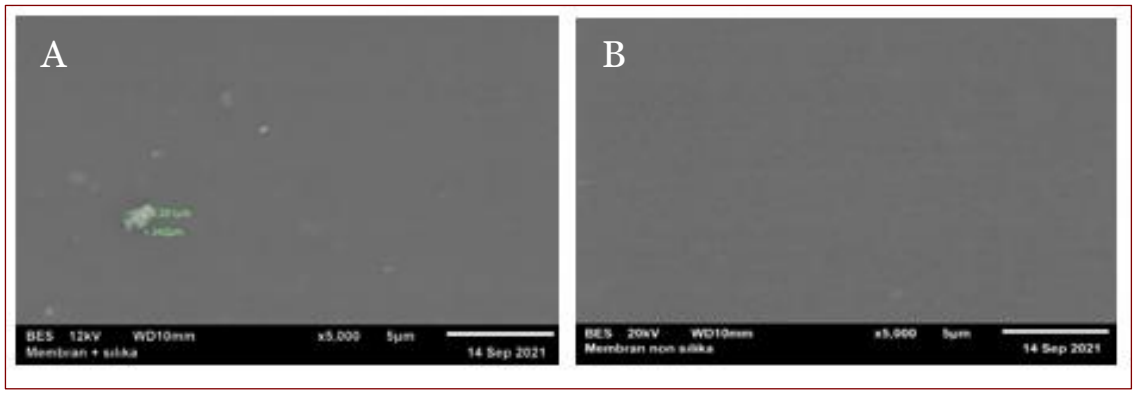

Figure 3. SEM images of the surface morphology of the polyurethane membrane with SiO_2 (A) and without SiO_2 (B).

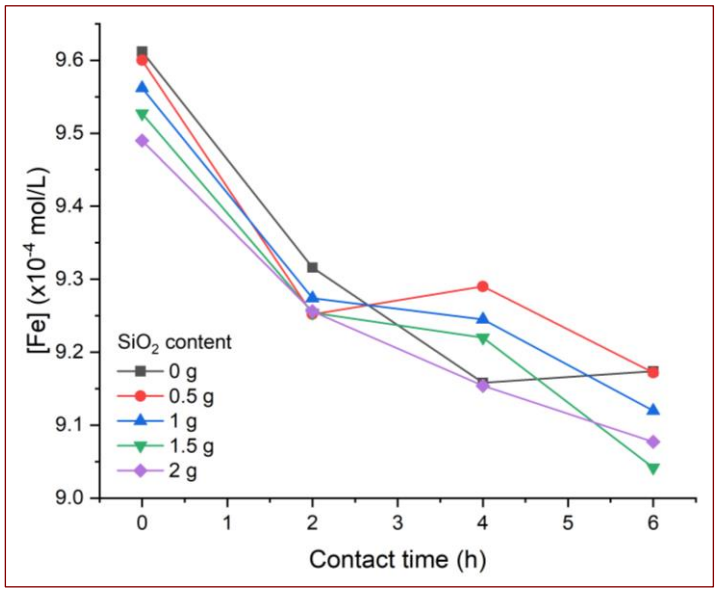

Figure 4. Effect of the SiO_2 addition on the Fe removal with a variation of contact time.

Discussion

The present study revealed that the embedment of SiO_2 into the polyurethane membrane matrix resulted in improved mechanical and thermal properties. The embedment of SiO_2 into the membrane matrix was associated with the appearance of irregular shape particles on the membrane surface. The addition of SiO_2 was not found to interact chemically due to the absence of vibrational bands corresponding to the Si-O-Si in the FTIR spectra. The fact that agglomerated SiO_2 particles were found on the membrane surface further suggests that the embedment occurred physically instead of chemically. In previous studies, similar changes in morphological, thermal, and mechanical properties were observed in the SiO_2-based composites [11-14]. Embedment of the SiO_2 nanoparticles into a bacterial fibrous matrix was reported to increase the mechanical strength, thermal, and water absorption [15]. Similar beneficial effects of the SiO_2 incorporation into a polypropylene-based matrix were also reported in a study aiming at fabricating thermoplastic material [16]. More mechanically and thermally stable properties were resulted from the SiO_2 due to the modifications in the adhesiveness of interfacial bonding. Filler addition-associated tensile strength improvement can be attributed to the interfacial bonding between fillers and matrix, which accommodates the transfer of load from polyurethane membrane into SiO_2 particles [17]. The structure of SiO_2 is also responsible for the increase in tensile strength as it prevents the slipping effect of the matrix due to stress [18].

Herein, the SiO_2 embedment into the castor oil-based polyurethane membranes resulted in an increased swelling degree. Moreover, we observed improved chemical resistance of the materials against various solvents such as NaOH, KOH, ethanol, and H_2SO_4. Interestingly, the increase in weight was observed when the SiO_2-embedded membranes were soaked in NaOH and KOH, suggesting a strong affinity for basic solutions. The resistance against ethanol and H_2SO_4 was particularly observable in membranes embedded with 1.5 g SiO_2. As suggested previously, an increase in swelling degree or water uptake is an indication of the increase in looser molecular structure and wider pores resulted from the SiO_2 embedment [19]. Swelling property is important in determining the quality of polymeric materials, particularly those used in adsorption. Water acts as a proton transport medium, but excessive absorption can weaken the membrane [20,21]. Regarding its effect on chemical resistance, silica was reported to have greater affinity for binding with salts and bases compared to acids and alcohols [22]. In previous studies, the castor oil-based polyurethane membrane was indeed having a weak resistance against acidic solutions [23-25].

The castor oil-based membrane was found to achieve higher Fe uptake when the embedded SiO_2 was 1.5 g, as observed after 6-h batch adsorption. At this point of observation, the Fe uptake was dose-dependent to the SiO_2 load, except when the load was increased to 2 g. In previous studies, the increase in impurities removal was associated with the SiO_2 load until it reached a certain level [5]. The effect of the SiO_2 addition was found to be beneficial for the multiple metal ions removal from water across the entire pH range [26]. As reported previously, the addition of SiO_2 could increase the porosity of the material, as the filler itself has a high porosity [27]. Further, the increased in swelling properties following the SiO_2 embedment also contributed to the increased surface area [28, 29]. Increased in affinity to positively charged heavy metal ions was also responsible to promote the electrostatic interaction between the adsorbent and Fe ions [30,31].

This study has several limitations, such as the absence of surface area and porosity analysis and a limited range of silica concentrations tested. The study also considers only specific contact times, which may not fully capture adsorption kinetics and equilibrium. Additionally, the controlled laboratory conditions do not reflect real-world environments, where factors like temperature, pH, and competing ions can impact the performance. Future studies employing large-scale production, more thorough characterization, and more adsorption parameters are warranted.

Conclusion

The embedment of SiO_2 into the polyurethane membrane prepared from castor oil resulted in higher thermal stability, mechanical strengths, chemical resistance, and swelling degree. The successful embedment was observable through the SEM images and spectral profiles of the membranes. More importantly, the addition of SiO_2 enhanced the ability of the membrane in the adsorptive removal of Fe ions. Overall, the SiO_2 embedment could increase the general properties of the material and improve its performance in water purification. Future studies to optimize the membrane compositions in a larger scale study whilst investigating the effect of different parameters affecting the performance (such as pH, temperature, competing ions, and adsorbent load) should be carried out.

Ethics approval

Not required.

Acknowledgments

None to declare.

Competing interests

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Underlying data

Data underlying the findings in this study could be requested to the corresponding author.

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