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Original Article

Synthesis of Hollow Fiber Membrane PES-Chitosan-$Mg(OH)_2$ as a Candidate for Hemodialysis Membrane

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ABSTRACT

Hemodialysis is a therapy for patients with chronic kidney failure using hollow fiber membranes. The use of hollow fiber membranes has encouraged the search for optimal, effective, and biologically safe formulations. This study aims to create, characterize, review the effectiveness and safety of PES-chitosan-$Mg(OH)_2$ hollow fiber membranes as candidates for hemodialysis membranes. The combination of these three materials is expected to improve the effectiveness and safety of hemodialysis. This membrane was made using a phase inversion method with varying concentrations of chitosan-$Mg(OH)_2$, then tested with FTIR, SEM, contact angle testing, flux, and BSA, urea, and creatinine rejection capabilities. FTIR results showed a shift in the –OH and –NH bands, indicating hydrogen interaction between PES and chitosan, as well as Mg^{2+} coordination with the chitosan amine group. Sample F3 with a 1% chitosan content showed the most optimal results with a porosity of 50.54%, a contact angle of 68.39°, a flux of 54.39 L/m^2h, BSA rejection of 96.26%, and urea and creatinine rejection of 57.74% and 48.96%, respectively. APTT, PT, and hemolysis tests confirmed good biocompatibility (<2% hemolysis). Overall, the PES–chitosan–$Mg(OH)_2$ membrane shows potential as an alternative material for effective and biologically safe hemodialysis applications.

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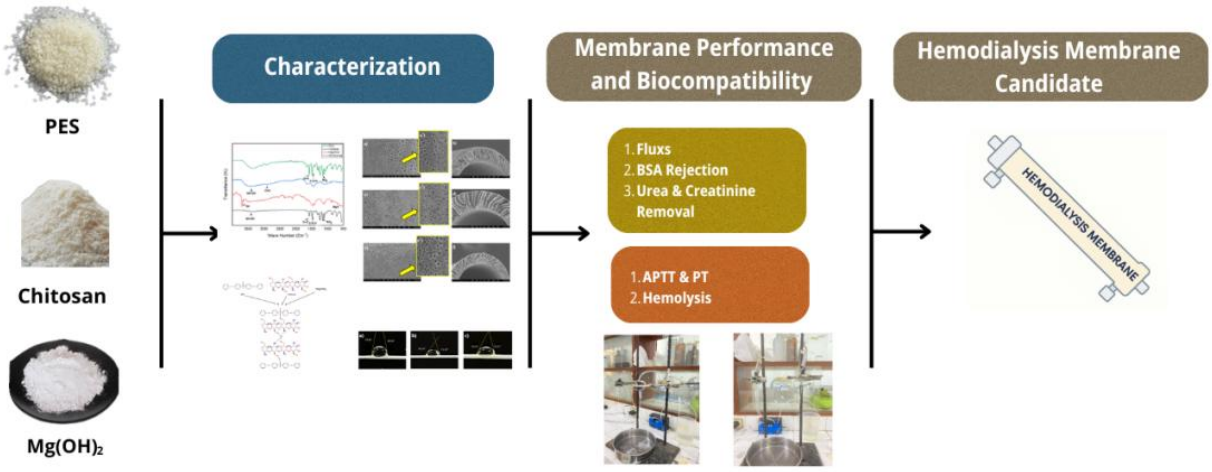
GRAPHICAL ABSTRACT



Introduction

Hemodialysis is the primary therapy performed by patients with chronic kidney disease (CKD). This therapy aims to remove toxic waste, excess fluid, and balance electrolytes using the principles of osmosis diffusion [1]. In this process, toxic uremic molecules are filtered through a semipermeable membrane, which is the main component of the hemodialysis system. CKD blood is spread across the membrane, while a low-concentration dialysate solution flows in the opposite direction [2] and removes toxins through substance exchange [3].

Hollow fiber membranes are used for hemodialysis. These membranes are chosen because they utilize diffusion and ultrafiltration mechanisms that enable the removal of uremic substances and are effective in removing excess fluid from the body [4] . In addition, their larger surface area and high flexibility, and low energy requirements, support filtration selectivity using horizontal flow, unlike flat sheet membranes with vertical flow [5].

The development of hollow fiber membranes currently still requires evaluation of their biocompatibility and antifouling properties. Biocompatibility is a crucial factor because membranes with low biological compatibility can trigger complement system activation, proinflammatory cytokine formation, and oxidative stress, which worsen the condition of chronic kidney failure patients and accelerate cardiovascular complications [6]. On the other hand, antifouling properties are also very important to maintain filtration effectiveness and long-term membrane stability, considering that the accumulation of proteins and microorganisms on the membrane surface can cause pore blockage, increased flow resistance, and a decrease in selectivity and service life [7]. Therefore, the selection of materials with biocompatible and antifouling properties is key to developing optimal hemodialysis membranes.

Polyethersulfone (PES) is one of the most commonly used membrane-forming polymers for hemodialysis. This is due to its biocompatibility, stability, and high thermal resistance, as well as its low resistance to coagulation and hemolysis [8]. However, PES is naturally a hydrophobic material, which is one of the causes of an increased risk of fouling due to the accumulation of proteins and particles in the blood, thereby reducing its effectiveness as a biocompatible membrane for hemodialysis [9]. Therefore, modification is needed by adding hydrophilic additives to enhance its hydrophilic properties and reduce the fouling effect on PES material. One of these additives is the biopolymer chitosan.

chitosan is one of the commonly used additives for forming hydrophilic membranes. This is supported by its superior properties such as biocompatibility, biodegradability, antibacterial properties, and non-toxicity [10,11]. The presence of hydrophilic groups in chitosan, amine and hydroxyl groups, renders the material more

hydrophilic, enabling it to form hydrogen bonds with water and enhancing its effectiveness in filtering cationic materials and organic compounds. Several studies have reported that the addition of chitosan as an additive can significantly influence membrane permeability, hydrophilicity, rejection coefficient, and mechanical strength [12,13]. Furthermore, the addition of metal materials has also been found to increase the stability and hydrophilicity of chitosan through cross-linking agent modification. magnesium hydroxide ($Mg(OH)_2$) is an inorganic material that has been widely used as a membrane additive. This is because $Mg(OH)_2$ contains hydroxyl groups that enhance interactions with water, thereby increasing hydrophilicity and reducing the risk of fouling on the membrane surface [13]. Furthermore, the addition of $Mg(OH)_2$ can also produce a membrane morphological structure with more dispersed pores in the form of a finger-like structure that facilitates water flow through the membrane surface [14-16].

Therefore, this study focuses on the development of PES-chitosan-$Mg(OH)_2$ hollow fiber membranes as candidate materials for hemodialysis membranes. This material combination is expected to overcome the problems of conventional hemodialysis membranes, especially in terms of membrane performance and biocompatibility. Studies on the combination of PES-chitosan-$Mg(OH)_2$ are still very limited, so this research can be an important step in developing effective and biologically safe membranes for hemodialysis therapy.

Experimental

Materials

The equipment used included chemical beakers, measuring cups, watch glasses, droppers, 100 mL glass bottles, stands, clamps, aluminum foil micro syringes, OHAUS Pioneer TM analytical balances, magnetic stirrers (DLAB MS7-H550 -Pro), and general glassware used in laboratories (Iwaki, Pyrex, and Dhuran). The instruments used were FTIR Shmadzu IRTracer-100, SEM ThermoFisher Scientific Phenom Series ProX, Shimadzu 1800 UV-Vis Spectrophotometer, 24V DC Adjustable Peristaltic Pump, and Casting Machine. The materials used were chitosan obtained from CV. ChiMultiguna (DD 85%), 1% acetic acid (v/v), PES (Radel A-300 Resin) obtained from Solvay Advanced Polymer (AS), NMP (purity 99.5%, MW=99.1 g/mol) obtained from Acros Organics, $Mg(OH)_2$ and urea obtained from Merck, BSA with a purity of >98% from Sigma Aldrich, creatinine, PBS obtained from MaxLab, picric acid, NaOH obtained from Merck, glycerol obtained from Merck, p-dimethylamino benzaldehyde, HCl 37% obtained from MaxLab.

Methods

This study consists of several stages, which are described as follows:

1. Synthesis of chitosan-$Mg(OH)_2$

The chitosan solution was mixed with $Mg(OH)_2$ solution at a volume ratio of 5:2 and homogenized at room temperature until homogeneous [1].

2. Fabrication of PES-chitosan-$Mg(OH)_2$ hollow fiber membranes

The PES solution was mixed with chitosan-$Mg(OH)_2$ solution for ± 24 h, with weight ratio is presented in Table 1, where F1 represents the PES membrane, F2 corresponds to the membrane with the addition of chitosan–$Mg(OH)_2$ containing 0.5% chitosan, and F3 represents the membrane with the addition of chitosan–$Mg(OH)_2$ containing 1% chitosan. The fabrication membrane was carried out using a hollow fiber casting machine with specifications of 50 cm air gap, 3 mL/min polymer flow, 1.5 mL/min borefluid flow, 4.5 RPM drum collector speed, 0.1 bar N_2 pre-pressure, and spinneret dimensions of 0.6 mm OD and 0.35 mm ID. After the membrane was cast, it was washed and dried at room temperature [15].

Table 1: Hollow fiber membrane formulations

Sample	Material % (w/w)			
	PES	chitosan (0,5%) -$Mg(OH)_2$	chitosan (1%) -$Mg(OH)_2$	NMP
F1	18	-	-	82
F2	18	1	-	81
F3	18	-	1	81

Characterization

1. Functional group test

Functional group tests on PES, chitosan, $Mg(OH)_2$, and PES–chitosan–$Mg(OH)_2$ composite samples were conducted using FTIR instrumentation.

2. Hydrophilicity Test

The hydrophilicity test was performed by measuring the contact angle of water on the membrane. The hydrophilicity of the membrane can be analyzed through the contact angle with the following angles:

Hydrophilic < 90° < Hydrophobic [15]

3. Porosity test

The porosity of the hollow fiber membrane is determined by soaking the membrane in distilled water for 24 h, and then weighing the membrane and recording its mass. After that, the membrane is dried in an oven at 105 °C for 24 h, and then weighed again. The porosity value can be calculated using the following formula 1, where ε is porosity (%), M1 is Wett Mass (g), M2 is Dry Mass (g), V is Volume (cm^3), and ρ is Density (g/cm^3) (Equation 1) [15].

$$\varepsilon\ (\%) = \frac{M1-M2}{V\times\rho} \times 100 \qquad (1)$$

4. Surface Morphology Test

Surface morphology tests were conducted using a SEM instrument with surface and cross-sectional analysis.

Membrane Performance

1. Flux

The flux test was conducted using the single strand method at a speed of 75 mL/min for 1 h. The flux value can be cal culated using the following Equation 3, where J is fluid flux (L/m^2h), V is permeate volume (L), t is permeate time (h), and A is membrane surface area (m^2) (Equation 2) [15].

$$J = \frac{V}{At} \qquad (2)$$

2. BSA rejection, urea, and creatinine removal

BSA rejection testing was performed by comparing the permeate concentration and feed concentration using a single strand method at a speed of 75 mL/min for 1 h, then analyzed with a UV-Vis spectrophotometer at a wavelength of 278 nm, and then calculated using Equation 4. Similarly, the urea removal test was performed by adding Erlich's reagent and analyzing it at a wavelength of 415 nm, while creatinine removal was analyzed by adding picric acid reagent and analyzing it at a wavelength of 485 nm. The urea and creatinine removal tests were calculated using Equation 5, where R is rejection (%), C_p is permeate concentration (ppm), C_f is feed concentration (ppm) [15].

$$Rejection\ (\%) = \left(1 - \frac{Cp}{Cf}\right) \times 100 \qquad (3)$$

$$Removal\ (\%) = \left(\frac{Cf-Cp}{Cf}\right) \times 100 \qquad (4)$$

Biocompatibility Test

1. APTT and PT

The activated partial thromboplastin time (APTT) and prothrombin time (PT) tests were performed by immersing the samples in PBS solution, and then incubating them with PPP, followed by mixing them with PPP, Actin FS, and adding $CaCl_2$ for APTT and Thromborel S for PT testing. After that, the clotting time was observed [16].

2. Hemolysis

The hemolysis test was performed by incubating red blood cell suspensions with membrane samples and controls, and then centrifuging them

before measuring the absorbance of the supernatant at a wavelength of 570 nm. The percentage of hemolysis was calculated using the following Equation 5, where SA is sample absorbance, PA is positive control absorbance, and NA is negative control absorbance.

$$HR\ (\%) = \frac{SA-NA}{PA-NA} \times 100 \quad (5)$$

Results and Discussion

Based on the FTIR results in Figure 1, the PES spectrum shows a sulfonyl band (–SO_2) at 1,231 cm^{-1}, aromatic C–O–C at 1,319 cm^{-1}, and asymmetric C=C bonds at 1,574 cm^{-1}, in accordance with Razi *et al.* [13]. In the chitosan spectrum, O–H and N–H stretching vibrations are observed at 3,410.26 cm^{-1}, C–H stretching at 2,922 cm^{-1}, and the presence of carbonyl and amide groups [19]. The $Mg(OH)_2$ spectrum shows a sharp –OH peak at 3,665 cm^{-1} and a MgO peak at 1,631 cm^{-1} [17]. In the PES/chitosan/Mg composite, the shift of the –OH and –NH bands to 3,365 cm^{-1} indicates hydrogen interaction between chitosan and the PES functional group [18], while the shift of the chitosan amide band to 1,698 cm^{-1} indicates coordination between chitosan and Mg^{2+} ions [19]. Accordingly, the combination of PES-chitosan-$Mg(OH)_2$ can be described by the hypothetical reaction shown in Figure 2.

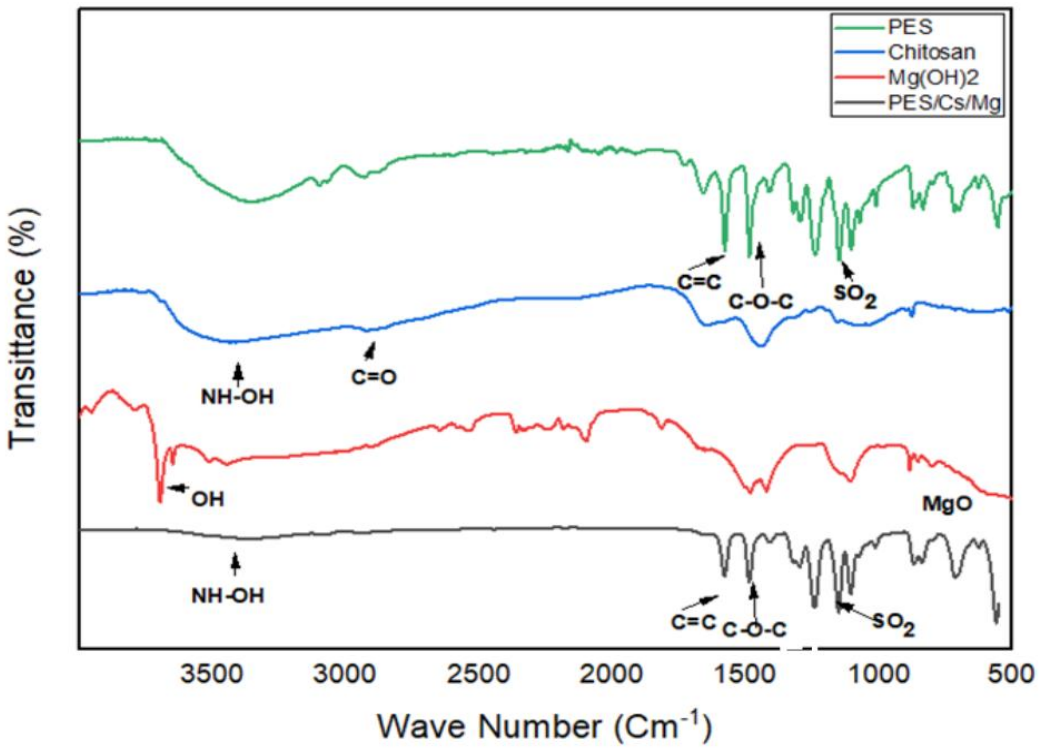


Figure 1: FTIR test result

Based on the hydrophilicity test in Figure 3 and Table 2, F3 showed the smallest box angle of 68.39 ± 3.98° compared to F1 and F2. This indicates that the addition of chitosan can produce a more hydrophilic membrane that is more effective in interacting with water [20], combined with the hydrophilic properties of $Mg(OH)_2$ which can also enhance membrane hydrophilicity [21]. According to research by Silitonga *et al.* [22], combination of chitosan-$Mg(OH)_2$ as a provider of hydrophilic groups, namely amine and hydroxyl, can increase membrane interaction with water [8].

In the porosity test in Table 2, F3 (50.54 ± 1.14%) was the highest compared to F2 (42.09 ± 2.86%) and F1 (32.20 ± 1.86%). This indicates that the addition of chitosan and $Mg(OH)_2$ as hydrophilic group donors can cause higher viscosity, and

solvent-additive interactions can change the solvent-non-solvent exchange profile so that the pores become larger and layered. These results are in accordance with Machodi and Daramola [20], where the addition of hydrophilic agents can increase membrane porosity.

Table 2 : Porosity, contact angel, APTT, PT, and hemolysis test results

	Porosity (%)	Contact angel (°)	APTT (s)	PT (s)	Hemolysis rate (%)
Control	-	-	25-35	11-15	-
Standart	-	-	32	32	-
F1	32.20 ± 1.86	80.21 ± 0,73	31.8	11.3	0.379
F2	42.09 ± 2.86	69.40 ± 4.86	31.7	12.8	0.434
F3	50.54 ± 1.14	68.39 ± 3.98	32.3	11.8	1.302

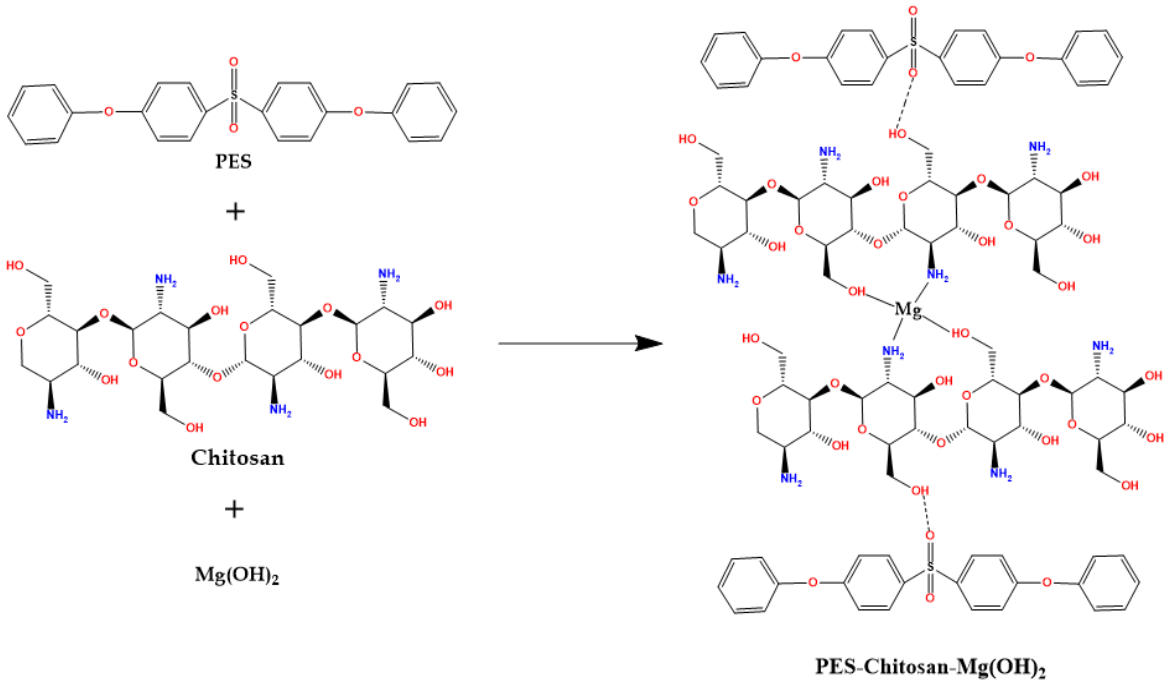


Figure 2: Hypothetical reaction of PES-chitosan-$Mg(OH)_2$

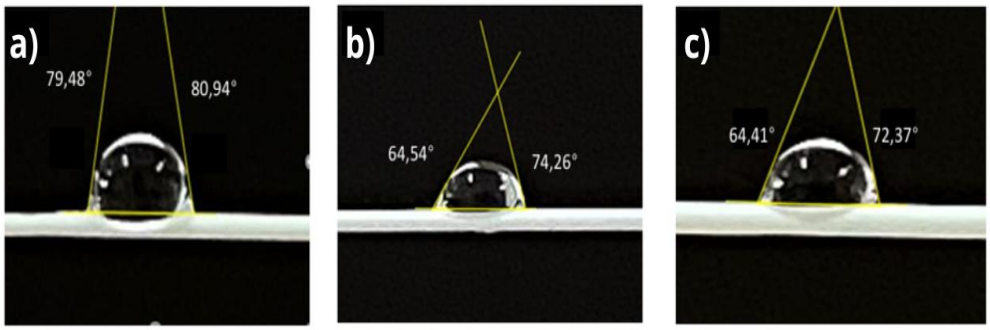


Figure 3: Contact angel result, a) F1, b) F2, and c) F3

Based on Figure 4, SEM analysis results show an increase in pore density and a clear finger-like structure with the addition of chitosan and $Mg(OH)_2$. F3 shows the most significant development, with wider and deeper surface pores (Figure 4e,e[1]) and a clearer and more evenly distributed finger-like macrovoid structure (Figure 4F). This increase occurs due to the nonsolvent-induced phase separation mechanism, which accelerates with the addition of chitosan

and $Mg(OH)_2$, resulting in more intense solvent-nonsolvent exchange and the formation of larger pores [23]. These results are consistent with the research by Fathanah *et al.* [21], where the combination of chitosan-$Mg(OH)_2$ was able to increase the formation of higher finger-like structures.

Based on the results of the flux performance test in Figure 5a, membrane F3 showed the highest result. This indicates that the addition of chitosan-$Mg(OH)_2$ can increase the hydrophilic properties of PES [21]. Chitosan has amino and hydroxyl groups that can form hydrogen bonds with water, thereby increasing water permeability and reducing the risk of fouling [24]. This is in accord with the research by Machodi and Daramola [20] and Abriyanto *et al.* [25], where an increase in hydrophilic properties can increase membrane flux. In the BSA rejection test for 1 h (Figure 5b), F3 also showed the highest results. This is important to note because it determines selectivity, fouling, biocompatibility, and prevents the loss of albumin protein in the hemodialysis process [26]. The addition of chitosan and $Mg(OH)_2$ acts as a hydrophilic agent for the membrane that does not alter protein conformation and functions as a protector or barrier against protein adsorption [14]. This surface also helps stabilize protein structure, unlike hydrophobic surfaces that can adsorb proteins, causing protein structure restructuring [27]. These results are consistent with the research by Zatadini *et al.* [28], where hydrophilic surfaces make it more difficult to adsorb BSA.

Based on Figure 6a, the urea removal test showed that F3 had the highest result of 57.74%. This increase occurred because chitosan acts as a donor and acceptor of amino and hydroxyl groups [20], which are capable of forming hydrogen bonds with carbonyl groups (C=O) [29]. Similarly, in the creatinine removal test in Figure 6b, F3 showed the highest value of 48.96%. This was influenced by the formation of hydrogen bonds between the amine and hydroxyl groups of chitosan with the carbonyl and N-H groups in creatinine. These results are in accordance with Khabibi *et al.* [30], where the addition of amine and hydroxyl groups can increase the adsorption of urea and creatinine.

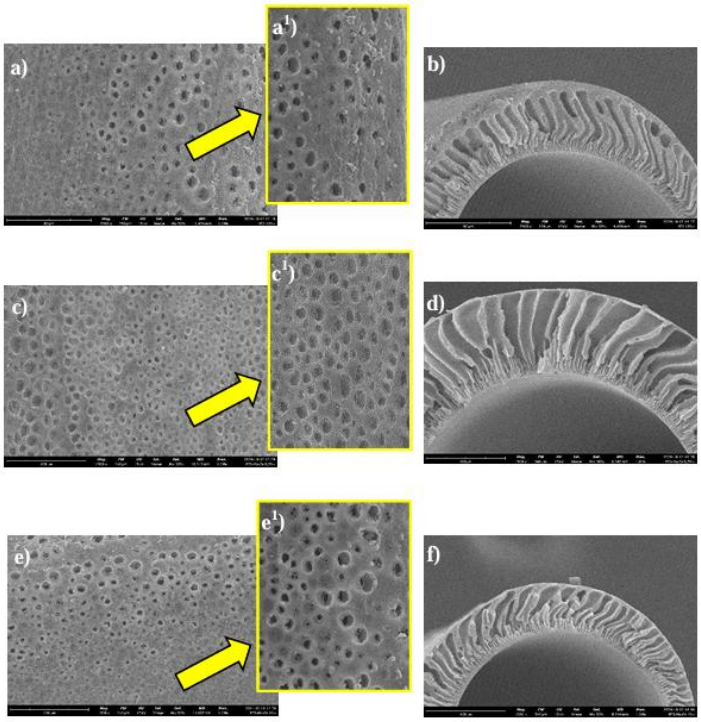


Figure 4: SEM results a) F1 surface 2,000x, a¹) magnification of F1 surface 2,000x, b) F2 cross section 2,000x, c) F2 surface 2,000x, c¹) magnification of F2 surface 2,000x, d) F2 cross section 2,000x, e) F3 surface 2,000x, e¹)magnification of F3 surface 2,000x, and f) F3 cross section 2,000x

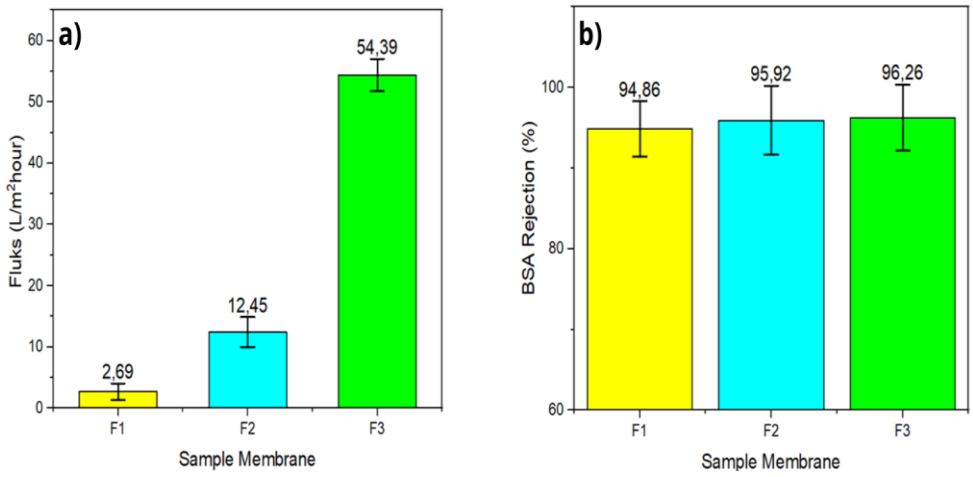


Figure 5: a) Flux and b) BSA results on hollow fiber rejection membranes

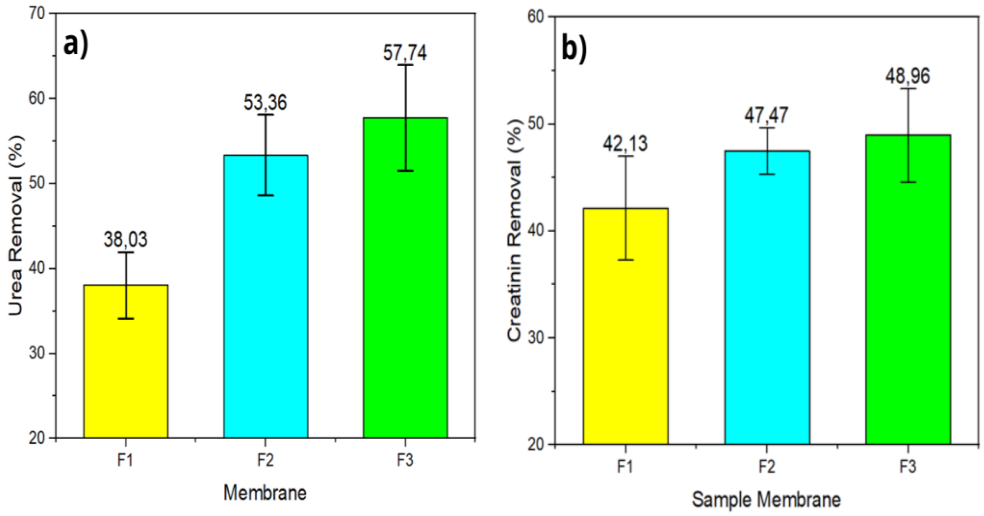


Figure 6: a) Urea removal results and b) creatinin removal results

In the APTT and PT tests aimed at testing blood coagulation in Table 2, samples F1, F2, and F3 were found to be biocompatible membranes and did not trigger abnormal blood clotting, with results still within the normal range of Airlangga University Hospital. The insignificant differences in these results are assumed to be influenced by uneven hydrophilic properties or platelet adhesion barriers [31]. Additionally, in the hemolysis test, samples F1, F2, and F3 were still below the <2% limit according to ASTM F756, which is classified as non-hemolytic. The increase in hemolysis in these results is assumed to be due to the charge and cationic properties of chitosan, which can increase electrostatic interactions, thereby increasing adhesion [32], as well as a mild hemolytic effect due to the presence of dissolved $Mg(OH)_2$, which causes pH changes or particle aggregation, increasing mechanical friction with blood cells [33].

Conclusion

Based on the results obtained, a PES-chitosan-$Mg(OH)_2$ hollow fiber membrane has been created, which shows a shift in the –OH and –NH bands toward lower values at a wavelength of 3,365.55 cm^{-1}, confirming the interaction between PES and chitosan. There is also a shift in the amide band at a wavelength of 1,698.56 cm^{-1}, which is related to chitosan and $Mg(OH)_2$. Among the synthesized membranes, membrane F3 was the most optimal, with the best hydrophilicity (68.39 ± 3.98°) and porosity (50.54 ± 1.14%) results, and produced the widest and most evenly distributed macrovoid-like finger structure on the membrane surface. In addition, membrane F3 showed the

most optimal results for flux (54.39 L/m²h), BSA rejection (96.26%), urea removal (57.74%), and creatinine removal (48.96%). All membrane formulations produced were biocompatible membranes, as indicated by APTT, PT, and hemolysis results that were still within the applicable standard range.

Conflict of Interest

The authors declared that there was no conflict of interest to disclose.

Consent for Publications

The authors have read and approved the final manuscript for publication.

Availability of Data and Material

If necessary, the authors have to declare that they embedded all data in the manuscript.

Authors' Contributions

Concept, design, and materials: SCH; supervision and resources: SCH; data collection or data processing and literature search: YBW; analysis or interpretation: YBW; manuscript writing: YBW; and critical review: SEC, PS, and AA.

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