Effect of Polyether Sulfone Modification on Membrane Performance and Biocompatibility for Haemodialysis Applications

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Haemodialysis is the process of purifying the blood of a person whose kidneys do not function normally. It removes urine toxins and regulates electrolytes in patients with kidney failure. Haemodialysis uses polyethersulfone (PES) membranes which are hydrophobic, so they are susceptible to fouling. Modified methods generally mix these with hydrophilic polymers. The hydrophilic material used as an anti-fouling agent is polyvinylpyrrolidone (PVP), which is able to increase the hydrophilicity of the membraneby increasing porosity, and removing uremic solutes. Another hydrophilic polymer is chitosan (Cs), which is able to increase hydrophilicity with high antifouling properties, as indicated by the reduced water contact angle. The modified hollow fibrefibre membrane can increase 324.5% compared to the unmodified one with a water flux value of 11.42 $L \cdot m^{-2}h^{-1}$. This is also in line with the removal values of urea and creatinine (51.14% and 50.06%). This is a promising result, similar to those obtained with commercial membranes. The resulting membrane is also good in blood applications which shows results less than 2% that correspond to the range which is considered non-hemolytic.

Keywords: Hemodialysis, biocompability, performance, Membrane Hollow Fibre PES-PVP-Cs, uremic toxines

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Chronic Kidney Disease (CKD) refers to the inability of the kidneys to perform the important task of removing the body's metabolic water products such as urea, creatinine, and excess water, in addition to maintaining the balance of electrolyte in the body and other substance, namely by removing excess sodium [1]. There are 66,000 new patients and 132,000 active patients registered in Indonesia. This number is increasing every year [2]. The widely used therapy for kidney failure patients is haemodialysis using a dialysis membrane. Through this therapy, water-soluble uremic toxins such as urea and creatinine can be removed [3].

Haemodialysis is the process of purifying the blood of a person whose kidneys do not function normally to remove urine toxins and regulate electrolytes. This External and internal dialysis systems can eliminate toxic residues and excess fluid while correcting electrolyte imbalances using the principle of osmosis and diffusion [4-5]. In haemodialysis, hollow fibre membranes are the most widely used. Hollow fibre membranes have high flexibility and a large surface area [6]. They are usually used for haemodialysis membranes that function as blood filters. The patient's blood flows through the fibres, while the dialysate fluid flows outside the fibres. The process of diffusion and ultrafiltration removes toxic substances such as urea and creatinine [7]. In the world of health, the materials used for hollow fibre membranes haemodialysis are PES, cellulose triacetate, polysulfone, polyamide, polyacrylonitrile, and polymethylmethacrylate [1, 8-9]. Such materials must meet the main requirements for membrane haemodialysis in terms of biocompatibility and ability to remove uremic toxins. Polyethersulfone (PES) membranes are hydrophobic, and thus susceptible to fouling [1, 10]. Fouling is the retention of a compound on the surface of the membrane due to its inability to pass through the hydrophobic membrane so that the membrane permeability becomes low and the membrane performance is reduced [11, 12]. Therefore, some modification is needed to prevent fouling. Modification is done by mixing with hydrophilic polymers. The hydrophilic material commonly used as an anti-fouling agent is polyvinylpyrrolidone (PVP). Previous findings have shown that addition of PVP as a membrane additive increased the hydrophilicity of the membrane by increasing porosity, removing uremic solutes, and enhancing biocompatibility performance as the main polymer [13-16].

Currently, the use of biopolymers in membrane forming materials has attracted a lot of attention, one of which is the chitosan biopolymer. Modified membrane materials are chosen because they are non-toxic, bioavailable, biocompatible and biodegradable. Chitosan (Cs) is a biopolymer that has the unique characteristics of being biodegradable, biocompatible, bioactive, porous and non-toxic [17-18]. Previous studies showed that a modified membrane with

chitosan had increased hydrophilicity with high antifouling properties, as indicated by the reduced water contact angle. This improvement is attributed to the amide and hydroxyl groups in chitosan, which also led to a substantial increase in pure water permeability. Cs-modified membranes exhibit an effective surface with smaller pore sizes compared to unmodified membranes [19-21].

Therefore, this study investigates the effects of adding chitosan and PVP to a PES membrane to increase its biocompatibility.

EXPERIMENTAL

Chemicals and Materials

The materials and reagents used in this research include Aquadest, 85% Chitosan (CV. ChiMultiguna, Indonesia), 100% glacial acetic acid (Sigma Aldrich, USA), Polyethersulfone (Radel A-300 Resin, Solvay Advanced Polymer (AS)), 99,5% MW=99,1 g/mol N-methyl-2 pyrolidone (Acros Organics), >98% Bovine Serum Albumine (Sigma Aldrich), creatinine (merck), urea (merck), PBS (MaxLab), Piric acid (merck), NaOH (Merck), glycerol (Merck), p-dimethylamino benzaldehyde, and HCl 37% (MaxLab). The equipment used include a magnetic stirrer (DLAB MS7-H550-Pro), analytical balance (Ohaus Adventurer AR2140, UK), Fourier Transform Infra Red spectrophotometer (FTIR) (PerkinElmer with wide range (4,000-400 cm^{-1})), UV-Vis spectrophotometer (Shimadzu 1800), peristaltic pump 24V DC Adjustable, casting machine and Scanning Electron Microscope (SEM) (TM3000, Hitachi, USA)

Synthesis of Hollow Fibre Membranes

The hollow fibre membrane solution was synthesised by adding PES, PVP, 0.5% Cs, and NMP solution additional compotition membrane was added with following the varians (Table 1). A chitosan concentration of 0.5% was chosen because at this concentration, chitosan was able to increase the hydrophilic properties of the membrane without causing a significant increase in the viscosity of the dope solution. Furthermore, the use of a 1% w/w additive aimed to achieve a balance between increasing porosity and the stability of the hollow fibre morphology structure, so that damage to the finger-like macrovoid structure did not occur. The solution was stirred with a magnetic stirrer for ±24 hours or until homogeneous at room temperature (25 °C). Then, the membrane solution was printed to produce hollow fibres. The membrane was made using a Hollow Fibre Casting machine with specifications of 50 cm air gap, 3mL/min polymer flow, 1.5 mL/min bore fluid flow, 0.1 bar N_2 pressure, and dimension of spinneret 0.6 mm OD and 0.35 mm ID. The spun hollow fibres were collected by a drum collector at the speed of 4.5 RPM. The spinning process was done at room temperature (29 °C). After the membrane was molded, it was washed with water for 3 days to removed residual NMP. Finally, the fabricated hollow fibres were kept in 10 wt.% glycerol for 1 day to prevent membrane structure collapse. The purpose of washing with water and adding glycerol was to remove toxins/chemical residues and stabilize the pores, while glycerol keeps the pore structure open so the membrane is not damaged when dry. Then the membrane was dried at room temperature [1].

Characterization of the Hollow Fibre Membrane

The porosity of the hollow fibres was analysed using the dry-wet weight method. The membrane was equilibrated in water for 24 h. The weights of the wet membrane, dry membrane, and its volume were measured. Themembrane porosity was calculated using Equation (1):

$$\varepsilon = \frac{M_1 - M_2}{V \times \delta_{water}}$$

where, M_1 and M_2 are the weights of wet and dry membrane (gram), respectively, V is the volume of the hollow fibre membrane (cm^3) and δ_{water} is the density of pure water (g/cm^3) [1].

Table 1. Formulation of hollow fibre membranes.

Material	Formulation (%w/w)		
	PES (F1)	PES-Chitosan 0.5% (F2)	PES-PVP-Chitosan 0.5% (F3)
PES	18	18	18
PVP	0	0	1
Chitosan 0.5%	0	1	1
NMP	82	81	80

Hydrophilicity testing was carried out to determine the contact angle of the material using a sessile method. The smaller the contact angle, the more hydrophilic it is. Hydrophilicity testing was carried out by dropping 3 µL of water which was then analyzed using image J. And then, the hollow fibre was analysed by FT-IR to identify the functional groups present in the hollowfibres. The morphology of the membrane was determined using Field-emission SEM (TM3000, Hitachi, USA) with 600x, 1000x, and 2000x magnification. For performance, the sample was analyzed using a flux test with a single strain method. Flux tests on hollow fibre membranes aim to ensure the operational performance of the membrane in specific applications, such as water filtration and separation. Another flux test was used to determine how much urea and creatininecould be lost. The Rejection of Bovine Serum Albumin (BSA) test was also performed. For biocompatibility, the sample was tested for Prothrombin Time (PT) and Activated Partial Thromboplastin Time (APTT) with the aim of measuring the length of time it takes for blood to clot when in contact with the membrane [14]. Finally, hemocompatibility was tested to check that the membrane would not cause blood clotting if it cames into contact with blood. Ethical clearance was granted by the Ethical Committee of Universitas Negeri Surabaya (035/UN38.III.1/DL.01.02/2025).

RESULTS AND DISCUSSION

Characterization of Hollow Fibre Membrane

The results of the FTIR analysis are shown in Figure 1. In the PES spectrum, the peak at 1231 cm^{-1} indicates the presence of a sulfone group (O=S=O), 1319 cm^{-1} indicates an aromatic ether (C-O-C), a methyl (C-H) at 1483 cm^{1}, and an aromatic ring cluster C=C at 1574 cm^{-1}. This is in accordance with the work of Machodi & Daramola (2019) showing three groups typical of PES [19].

In the Cs spectrum, OH^- and NH_2 functional groups are indicated by the peaks at 3410 cm^{-1}, while the CH groups are indicated by the peaks at 2922 cm^{-1}. The peaks at 1643 cm^{-1} and 875 cm^{-1}indicate the presence of amide and methyl groups, respectivelyare. The results of the characterization of chitosan are in line with the research conducted by Abriyanto et al. (2022) which found that the typical functional groups present in chitosan were OH-, C=O, C-H, and NH.

Furthermore, the modified membrane with the addition of PVP saw the emergence of a significant new peak at 1680 cm^{-1} which is due to a primary amide (NH). The same finding was also reported by previous researchers, namely the presence of PVP in the PES membrane at 1678 cm^{-1} [17]. According to Ahmad et al. (2019), the presence of OH groups can improve the hydrophilicity of the membrane and help improve its anti-fouling properties [22]. In another study, this improvement was attributed to the amide and hydroxyl groups in chitosan, which also led to a substantial increase in pure water permeability [20]. On the other hand, the presence of other stretches on the 1662 cm^{-1} at PVP. FTIR spectrum data showed the presence of new functional groups after the addition of additives to polymer solutions. This spectrum indicates the presence of PVP in the modified PES membrane.

In the PES-PVP-Cs membrane, is in line with the findings of another study [14], O-H and amine functional groups (NH) were detected at 3392 cm^{-1}. This indicates the presence of chitosan in the PES-PVP-Cs membrane.

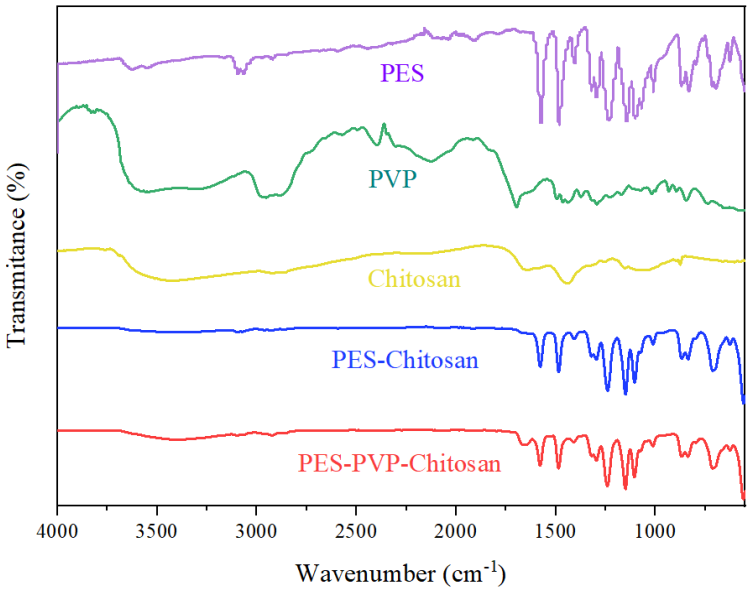

Figure 1. FTIR spectra of hollow fibre membranes.

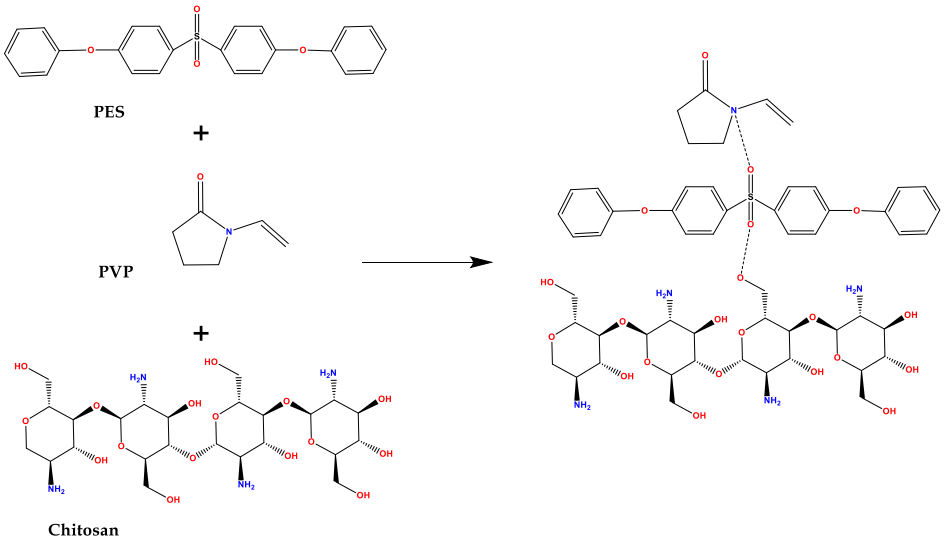

Figure 2. Hypothetical PES-PVP-Chitosan Reaction.

A comparison of the FTIR spectra show that the interaction between PES-PVP-Cs was indicated by an increase in the transmittance of the O-H group at 3390 cm^{-1}. It is likely that the bonds between the OH and NH_2 groups are chitosan, and PVP serves as the C=O-binding additives to PVP (Figure 2).

Based on this analysis, the hypothetical reaction of the PES-PVP-Chitosan combination shown in Figure 2 can be assumed. This is because the PES ether group is able to interact hydrogenically with amine and hydroxyl chitosan groups, while PVP is able to interact weakly through its hydrogen with oxygen atoms with sulfone groups in PES.

Permeability and anti-fouling are membrane performance parameters that are influenced by the hydrophilic properties of the membrane. This can be determined by measuring the water contact angle. A membrane is said to be hydrophobic if water does not spread over the surface of the membrane with a contact angle of more than 90°. A hydrophilic membrane has a contact angle of less than 90°, because water droplets are adsorbed to the surface of the membrane [23]. The lower the value of the water contact angle, the more hydrophilic the membrane [24].

Table 2 shows that the amount of material added to the hollow fibre membrane affects its hydrophilicity. This is due to the addition of chitosan as a hydrophilic material [21] and PVP as a hydrophilic material that can reduce hydrophobic properties [14]. Therefore, the combination of the three materials creates a synergy that can increase its hydrophilic properties. Machodi & Daramola (2019) showed that the angle of contact decreased with an increase in chitosan content, hence the influence of chitosan as a hydrophilic agent in increasing the hydrophilicity of the membrane surface. This results in increased transport of water molecules through the membrane [19]. Wu et al. (2020) indicated that the surface hydrophilicity of a membrane increased gradually with PVP content. The increased membrane hydrophilicity can be attributed to the polar groups in the PVP molecule [16].

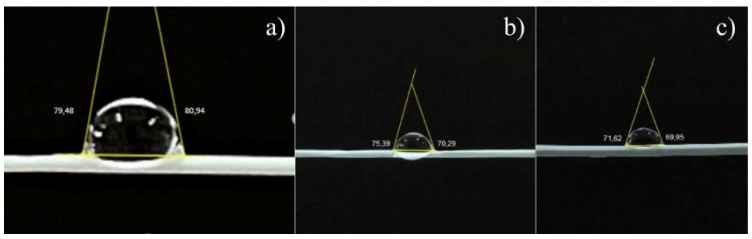

Figure 3. Contact angles of hollow fibre membranes for (a) F1, (b) F2, (c) F3.

Table 2. Contact angle of hollow fibre membranes.

Sample	Contact Angle (^{O})
F1	80.21 ± 1.03
F2	72.80 ± 3.61
F3	70.78 ± 1.18

Table 3. Porosity of hollow fibre membranes.

Sample	% Porosity
F1	32.20 ± 2.28
F2	41.60 ± 0.54
F3	62.42 ± 0.01

In porous membranes, the contact angle depends on the porosity and energy of the membrane surface [15]. The lower the porosity, the higher the angle of contact [25]. Table 3 shows the results of the membrane porosity test. The membrane with chitosan and PVP had its porosity increased to 62.42% which implies there is more space on the membrane. The high porosity of the membrane allows it to have evenly distributed pores so that it can filter out elements that are not needed in the water [13]. The statistical test results show that the relationship between contact angle and porosity gave a result of less than 0.05, namely a significant inverse relationship.

When the blood filtration process takes place, the membrane cavity would be filled with uremic substances. With a large number of membrane pores, many uremic substances that are toxic to the body can be removed. This would optimally reduce the uremic content in the blood.

Figure 4 shows the morphological structures of PES, PES-Cs, and PES-PVP-Cs on their surfaces and cross sections. These indicate that the hollow fibre membranes are uniformly porous. The morphological structure of the membrane shows that it is dense and porous. This suggests that the addition of chitosan makes the membrane dense and porous. This is in accordance with research by Iman, et al. (2024) which suggests that the morphological structure of the chitosan and PES membranes results in a uniform dense, small, porous membrane [20]. Whereas membranes with PVP (Figure 4(c)) were more dense and porous membranes. It can also serve as a release of uremic toxins. This is also in accordance with the research of Sofyana et al. (2020) which shows that the morphological structure of the PES-PVP membrane produce membranes, namely pores that form more and spread. Pore formation increases membrane permeability [13].

In figure 4 Morphology of membranes in transverse cross-sections, porous structures and cavity structures such as finger-like void which is almost similar for various variations. All membranes contain structures macrovoids. In Figure 4a has finger-like macrovoids. However, the modified membrane has larger pores and more regular channels. This is based on the Hagen-Poiseuille relationship [21].

Flux is one of the most important parameters carried out to determine the performance or ability of the membrane in the separation process. Flux measurement in this study was carried out using aquadest [13]. The membrane performance test was carried out with a single-strand motode using a 24 DC peristaltic pump at a speed of 75 mL/h for 60 minutes [1]. The pure water flux test was performed using aqueducts. In Figure 5, the flux of the modified hollow fibre membrane increased 324.5% compared to the unmodified one with a value of 11.42 $L \cdot m^{-2}h^{-1}$. This is influenced by several factors related to membrane structure such as increased pore size, increased porosity, and decreased membrane thickness. It may also be associated with increased hydrophilicity [26]. The statistical test results show that the relationship between contact angle and flux had a result of less than 0.05, namely a significant inverse relationship.

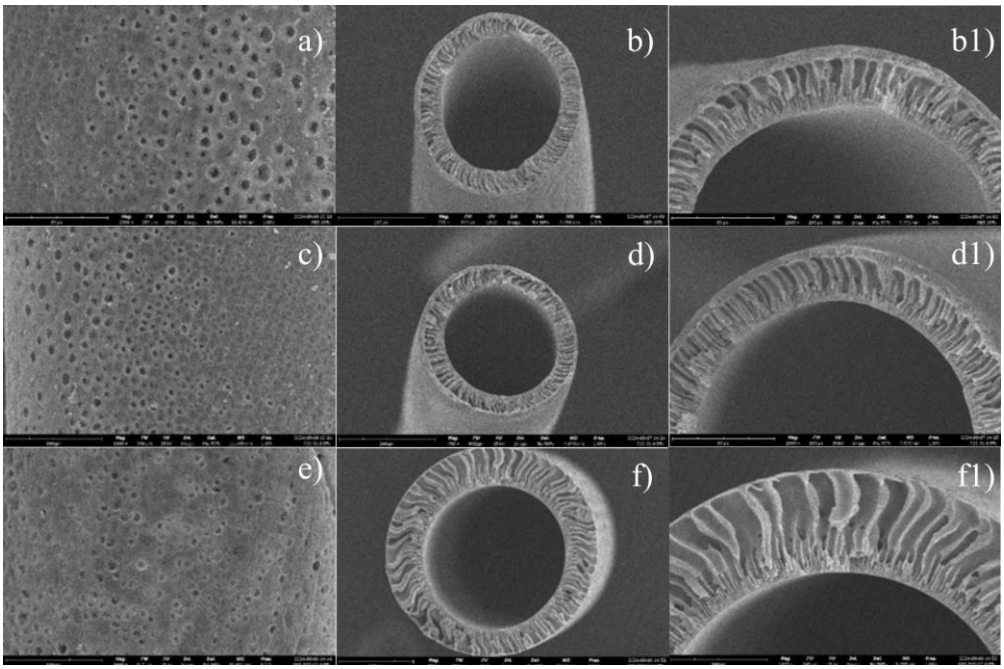

Figure 4. SEM Results for (a) F1, (b) cross section of F1, (b1) magnification of F1 cross section, (c) F2, (d) cross section of F2, (d1) magnification of F2 cross section, (e) F3, (f) cross section of F3, and (f1) magnification of F3 cross section.

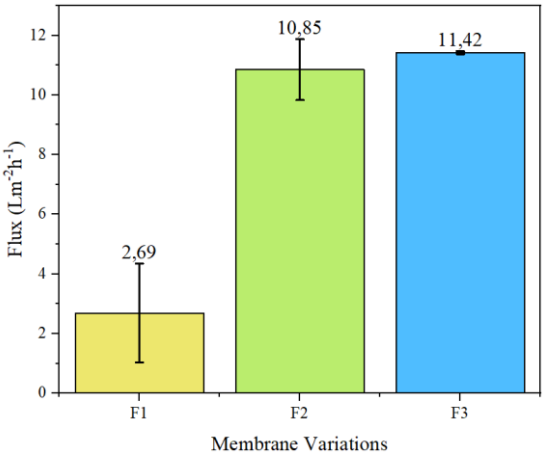

Figure 5. Flux of of hollow fibre membranes.

Haemodialysis therapy has been shown to remove at least 60% of toxins during the treatment process [27]. In addition, it has been proven that this treatment prevents albumin loss by up to 75%. The dialysis performance of pure PES and surface-modified membranes was evaluated by determining urea cleansing (60 Da) [28]. Figure 6 shows the hollow fibre membrane of PES-PVP-Chitosan showed a urea removal of 51.14, better than for PES membrane alone. This is in line with the SEM characterization which showed that the PES-PVP-Chitosan hollow fibre membrane had many even pores. It is also in harmony with the principle that the more hydrophilic the membrane, the easier it is for water and uremic toxins to dissolve and exit the hollow fibre membrane. The high porosity also supports the removal of uremic toxins during the dialysis process [29]. The statistical test results show that the relationship between contact angle and urea removal had a result of less than 0.05, namely a significant inverse relationship.The urea removal test used a 1000 ppm urea dialysate solution analyzed at 415 nm using Erlich reagents [9][30].

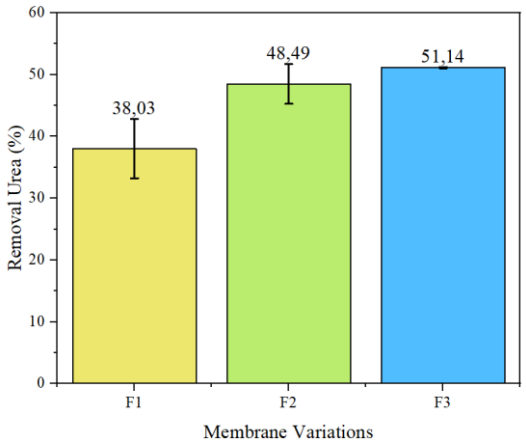

Figure 6. Urea removal by hollow fibre membranes.

Haemodialysis is an effective and commonly used treatment for chronic kidney failure [31]. According to Nguyen et al. (2021), haemodialysis can only remove about 75% of the accumulated urea and 66% of the creatinine in the body [32]. The creatine removal test used a 1000 ppm creatine dialysate solution at 485 nm using a picric acid reagent [9]. Figure 7 shows that the membrane with PVP was able to remove uremic toxins, namely creatinine by 50.06%. This is due to the formation of micropore cavities throughout the membrane structure. This increases the permeation of solutes, allowing larger molecules to pass through the membrane [33]. It is also in harmony with the principle that the more hydrophilic the membrane, the easier it is for water and uremic toxins to dissolve and exit the hollow fibre membrane. Its high porosity also supports the removal of uremic toxins during the dialysis process [29]. The statistical test results show that the relationship between contact angle and removal creatinine gave a result of less than 0.05, namely a significant inverse relationship.

The BSA rejection test used a 1000 ppm BSA dialysate solution at 280 nm [9]. The PES and PES-chitosan membranes were able to prevent albumin loss by 90%. This is due to the pore size being no larger than BSA at 68 kDa [34]. Figure 8 shows that the PES-Chitosan membrane was able to prevent the highest loss of albumin. This is because the PES-Chitosan membrane had a denser pore size than other membranes, but the results on the hollow fibre membrane are able to prevent albumin loss by 90%. It is also in harmony with the princile that the more hydrophilic the membrane, the easier it is for water and uremic toxins to dissolve and exit the hollow fibre membrane. Its high porosity also supports the removal of uremic toxins during dialysis [29]. The statistical test results show that the relationship between contact angle and rejection BSA gave a result of less than 0.05, namely a significant inverse relationship.

From the practical point of view, these results underline the necessity to find operating conditions (membrane composition, cleaning in place parameters) that can avoid additives leaching from the matrix body.

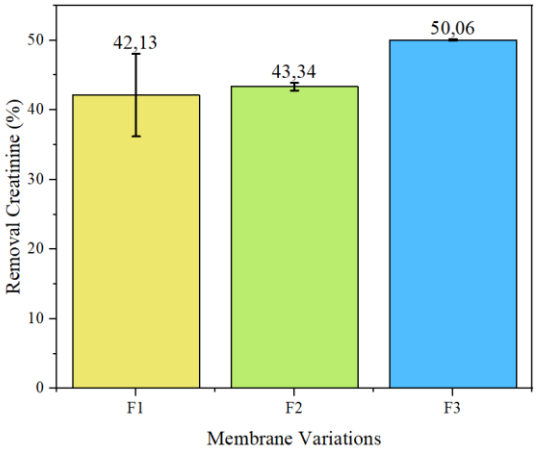

Figure 7. Removal creatinine of hollow fibre membranes.

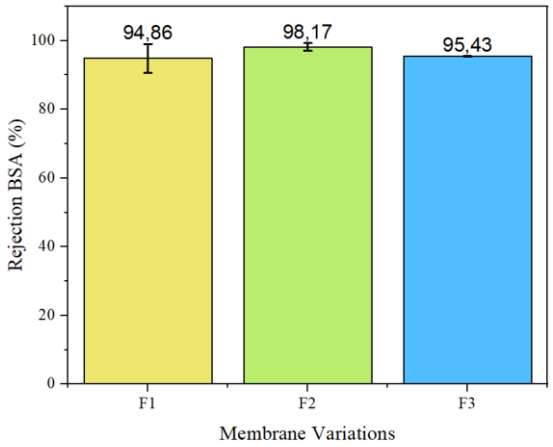

Figure 8. BSA rejection of hollow fibre membranes

The membranes were tested by PT and APTT with the aim of measuring the length of time it takes for blood to clot when in contact with the membrane. The results of the blood clotting time test can be seen in Table 4. The sample had a clotting time that was close to the control blood clotting time. The results for F1, F2, and F3 dropped significantly but were still within control limits. The results of the APTT test on the hollow fibre membrane showed a slightly faster blood clotting time (31.7 s) compared to the control (32 s). These results did not differ significantly compared to the control and were still in the normal range, which was 25 to 35 seconds for APTT and 11 to 15 seconds for PT. The results of the one way ANOVA test showed that there was a significant difference in APTT values between groups ($p < 0.05$). All formulations showed a lower clotting time than the controls. For PT, there was a significant difference between groups ($p < 0.05$), where the F3 formulation showed a higher clotting time than the control.

Based on these data, the PES-PVP-Chitosan membrane had good hemocompatibility because it did not cause blood clotting when in contact with blood. Based on this, biocompatible membranes are capable of interacting with blood without causing blood clotting effects [28]. Based on Table 5, a positive control with an absorbance value of 1.901, a negative control of 0.058, and an F1 membrane sample of 0.065 with an average haemolysis of 0.379 % were obtained. The F2 membrane sample was classified as good with an average haemolysis value of 0.542%, while the F3 membrane sample had an average haemolysis of 0.488% which is considered non-haemolytic based on ISO 10993-4:2017. This indicates that F1-F3 is biocompatible and would not cause damage to red blood cells.

Table 4. Coagulation test on blood (PT and APTT).

Sample	PT (s)	APTT (s)
Control	13.2 ± 0.20	32.0 ± 0.20
F1	11.3 ± 0.15	31.8 ± 0.10
F2	11.7 ± 0.20	31.7 ± 0.12
F3	12 ± 0.05	32.6 ± 0,10

Table 5. Haemolysis test.

Sample	Haemolysis (%)	Absorbance
Positive control		1.901
Negative control		0.058
F1	0.379 ± 0.21	0.065 ± 0.004
F2	0.542 ± 0.10	0.068 ± 0.002
F3	0.488 ± 0,11	0.067 ± 0.002

CONCLUSION

A PES membrane modified with PVP and Chitosan was successfully synthesized. The resulting membrane had good characteristics as a haemodialysis membrane. Hydrophilic membranes were produced by adding PVP and chitosan as hydrophilic polymers. In the performance of the membrane by the value of water flux is the best and can increase 324.5% compared to the unmodified with the addition of PVP and chitosan of 11.42 $L{\cdot}m^{-2}h^{-1}$. This is also in line with the urea and creatinine removal values (51.14% and 50.06 %) but not in the best BSA rejection with the addition of chitosan alone of 98.17%. The resulting membrane is also good in blood applications which shows results less than 2% that correspond to the range which is considered non-hemolytic. That matter improved toxin clearance without compromising hemocompatibility.

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