



## The Coating Effect of PANI/Silver on Performance of Polysulfone Membrane Toward Protein Separation

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### ABSTRACT

The effects of different coating time of PANI/Silver into polysulfone (PSf) membrane surface were investigated according to the morphology, contact angle, surface roughness and BSA, pepsin and trypsin rejection. The membrane was prepared by employing the pressure deposition method toward phase inversion membrane. Thus, PANI particles were forced to adhere on membrane surface by pressure driven force. The duration of coated time was taken from 30 mins up to 120 mins. However, due to smooth surface of PSf, PANI particle was able to bounce back from the PSf surface. Furthermore, the presence of PANI/Ag were also hard to distinguished on the membrane surface. Clear observation was noticed with the changed of the membrane surface from smooth surface until rougher surface. EDS result using SEM data proved the presence of PANI and PANI/Ag on the surface membrane. The hydrophilicity of membrane was proved with decreasing of contact angle test from 75° to 40° for duration time from 0 min until 120 min for membrane coated with PANI. Meanwhile membrane coated with PANI/Ag also show a reduction from 75° to 50°. The result was in line with membrane surface roughness which is increased up to 79% after coating with PANI while 90% after coating with PANI/Ag after under effect of 120 min. Higher surface roughness had influenced membrane rejection performance toward BSA. For water rejection test, PSf membrane showed the rejection of 100%, 60.42% and 50% for BSA, pepsin and trypsin. After coating membrane for 30 min, 100%, 90.21% and 77.23% was obtained for PANI-coated and 100%, 92.30% and 80.30% after coating with PANI/Ag. For duration of 120 min, the result shows that coated membrane able to reject 100% BSA, for pepsin and trypsin it shows 96.15% and 87.98% while membrane coated with PANI/Ag, it shows 100% BSA and up to 98.41% and 90.60% pepsin and trypsin protein. In the end, study of membrane performance was improved with presence of PANI and silver on the protein separation process by using deposition method.

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## 1. INTRODUCTION

According to a report by newspaper "The Star", most of the main river water in Malaysia has turned out to be polluted [1]. This report was in line with some other researcher that reported the water in the main river in Malaysia is not all clean and some were polluted [2, 3]. Although our watershed still remains secured, this issue can become serious due to the fact that water was essential either for human or animal life. In order to improve water quality especially for drinking water, all

of our water supply company such as Syarikat Air Johor has conducted treatment on the water to ensure that water are safe to drink. Among the treatment involves chemical such as chlorine, zinc ferrite, sodium chloride and potassium chloride [4, 5]. This chemical although was reported safe to be used; however, if the quantity exceeded the limit, the human health will be affected. Membrane technology was one of the water treatment processes that can treat water without involving any chemical or additive. In membrane separation applications, the main objective are to only letting one

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element in the water mixture to go through the membrane freely without any resistance while preventing the other elements to pass through the membrane.

Polysulfone (PSf) is one of the most common materials that has been used as a membrane. PSf has the ability to sustain high temperature up to 190 °C, high resistance to chemical and low production cost that make this material among popular in water treatment industry [6]. However, PSf has low hydrophilicity which resulted in a fouling problem. Fouling can be defined as deposition of a particle on membrane surface and pores. The particle mentioned is normally hydrophobic substances which tend to interact with membrane hydrophobic surface and eventually block the membrane pores. As the membrane pore is blocked, water unable to penetrate the membrane, hence membrane performance will be declined.

In order to reduce membrane fouling, a lot of researchers has made modification on membrane surface. This is due to the fact that the main attribute to fouling is the attraction of hydrophobic particle on the membrane surface [7, 9]. The membrane surface modifications can be conducted either by combining or blending with the hydrophilic nano particle, grafting or surface coating with hydrophilic materials [10, 11, 12]. Coating hydrophilic material on the membrane surface was reported to able protect membrane surface and reduce membrane hydrophobicity.

Polyaniline (PANI) is a hydrophilic polymer that is used as coating material on the membrane [13]. A lot of researches have focused on blending properties of PANI in a polymer membrane. Although the result was promising, there are concerns arise especially on the miscibility of PANI in polymer dope solution. It was reported that PANI was unable to be diluted due to strong chemical resistance on the solvent. Consequently, mixing PANI in polymer solution will produce a poor dispersion of PANI in the membrane. Thus, the alternative way are to coat membrane surface with PANI. However, coating PANI on membrane surface does not solve the biofouling problem. Compared to normal fouling, biofouling involved attachments of bacteria on membrane surface. The bacteria tend to accumulate on membrane pores and produce protein layer on the top of membrane surface. To overcome this problem, addition of antibacterial agents such as silver, zinc oxide or titanium dioxide is expected to be able to reduce biofouling problem. Silver is a common antibacterial agent that widely used in medical, others and able to attack several types of bacteria [14]. Silver is also among the materials that is mostly used in membrane because it is less toxic to human body cells and has excellent antibacterial properties. Thus, in this study, the effect of coating PANI and silver on PSf membrane was investigated. The prepared membranes were characterized with respect to its morphology, fouling properties. The objective for this paper are to

investigate the effect of coating time on Polyaniline (PANI) / Silver (Ag) on PSf membrane surface using BSA solution as foulant.

## 2. MATERIALS METHOD

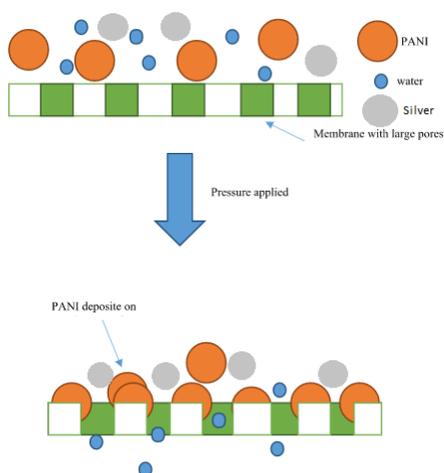
**2. 1. Materials** The materials used in this study include polyaniline (PANI), silver nitrate ( $\text{AgNO}_3$ ), Polyethylene Glycols (PEG), Bovine Serum Albumin Fraction V (BSA), pepsin, trypsin and N-methyl-2-pyrrolidone (NMP) were purchased and used to produce membrane PANI/Ag.

**2. 2. Manufacturing Membrane PANI And PANI/Ag Membranes** were prepared by immersion phase inversion method. Firstly, the polymer solution was prepared by dissolving NMP along with PSf. By mechanical stirrer, the solution was stirred for 4 hours. The solution was stirred at a temperature of 80°C at the speed of 500 rpm. Vigorous stir was carried out at room temperature to prepare PSf pristine membrane. By completing the stirring process, the dope solution was poured into the bottle. Before casting, the solution was sonicated for about 15 minutes using an ultrasonic device (KQ-100VDE, China). This is to ensure that the dope solution was dispersed well and the bubbles formed during the stirring process was eliminated. Then, the membrane was casted using casting knife with 150  $\mu\text{m}$  gap between casting solution and knife. Then, the dope was immersed into a water bath to solidify the dope solution and form a membrane. Next, the membrane was immediately proceeds to be coated with PANI and Ag.

The coating process was done by employing the pressure deposition method using ultrafiltration permeate machine. The concept of coating deposition is shown in Figure 1. Figure 1 shows that PANI and silver was acting as foulant and deposited on the membrane after pressure been applied. Theoretically, the deposition depends on time PANI and silver deposited on the membrane surface. The design of experiment was focused on the effect of time chemical additive deposited on the membrane surface. The reaction of deposition area depends on the time coating. The time was started from 30 min to 120 min.

**2. 3. Characterization of PANI And PANI/Ag Membrane** A Hitachi S-4800 field-emission scanning electron microscope (SEM, Japan) was used to study the microscopic structures of various membranes. The microscope has an energy dispersive X-ray spectrograph (EDX) detector.

The membranes were dried in vacuum, and then mounted on the samples support for the morphology observation.



**Figure 1.** Schematic diagram using deposition method

When studying the inner structure of a membrane, a cross-section was obtained by fracturing the membrane using liquid nitrogen. The sample was cut in 3 cm x 1 cm for this test.

VCA Optima contact angle machine was used to investigate hydrophilicity of the membrane. Wettability studies are defined as the measurement of contact angle as a primary data, which indicated the degree of wetting when the solid and liquid interact. Hydrophobic will occur when Contact angle 90 degrees and above while hydrophilic occur less than 90 degrees. The membrane was cut into 5 cm x 2 cm size. To measure the degree of contact angle, 2  $\mu$  L of water was carefully injected on to the membrane surface. After the water drop to the tip of the needle, the position of the moving plate was adjusted to fit with the scale in the screen monitor.

The membrane specimen was cut into the size of 2 cm x 2 cm and placed on to scanner tube was prepared. AFM was used to measure membrane surface roughness and the type of machine used is E-100 Park system. The value of average surface roughness was then recorded.

Rejection test for  $\text{AgNO}_3$ /PANI/PSf membrane was tested using Pepsin, Trypsin, and BSA. The sample was cut in round shape and place it in the permeate machine. The solution for 3 type protein was added with 1 litter distilled water. The liquid will permeate though the membrane by driven pressure. After finish the permeate, the sample of water permeate was taken and store in the container. Each sample was taken to measure the rejected using UV – vis spectrophotometer.

### 3. RESULTS AND DISCUSSION

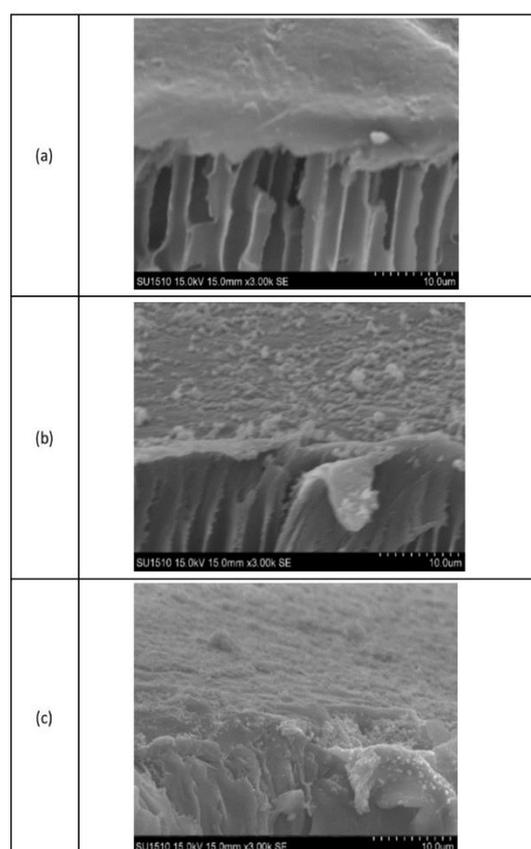
#### 3. 1. Characterization Properties Of PANI And PANI/Ag Membrane

SEM images of PSf, PANI and PANI/Ag microspheres are shown in Figure 2. As can be seen from the Figure 2(a) the membrane has no additive on

the surface. Figure 2(b) shows the membrane coating with PANI on the surface. The white and small particles on the Figure 2(b) show the presence of PANI element on the membrane surface. Figure 2(c) also show the presence of PANI/Ag with the existence of smaller particles on top of membrane surface both coated membrane show the formation of thin layers of non-homogenous structure with rougher surface area. This result is in line with previous research that showed the non-homogenous surface was formed on the membrane after several cycles of depositions technique [15].

Further information on the elemental composition of the coated samples can be determined by EDX analysis. Figure 3 also shows the EDX spectrum of membrane not only PSf but with additive PANI and Ag. The EDX results in Figure 3(a) confirmed the presence of only oxygen, O, carbon, C and sulphur, S elements in the membrane. This proven the membrane are not coated with other chemical. The spectrum Figure 3 (b) shows that membrane coated with PANI consist of O, C, S and nitrogen, N elements. The present of nitrogen element where possessed by PANI are able to detect at 0.4 KeV [16].

The result also shows that the PANI tend to agglomerate and form a cake layer on the top membrane surface.



**Figure 2.** SEM images of (a) PSf (b) PSf / PANI and (c) PSf/PANI/Ag membrane

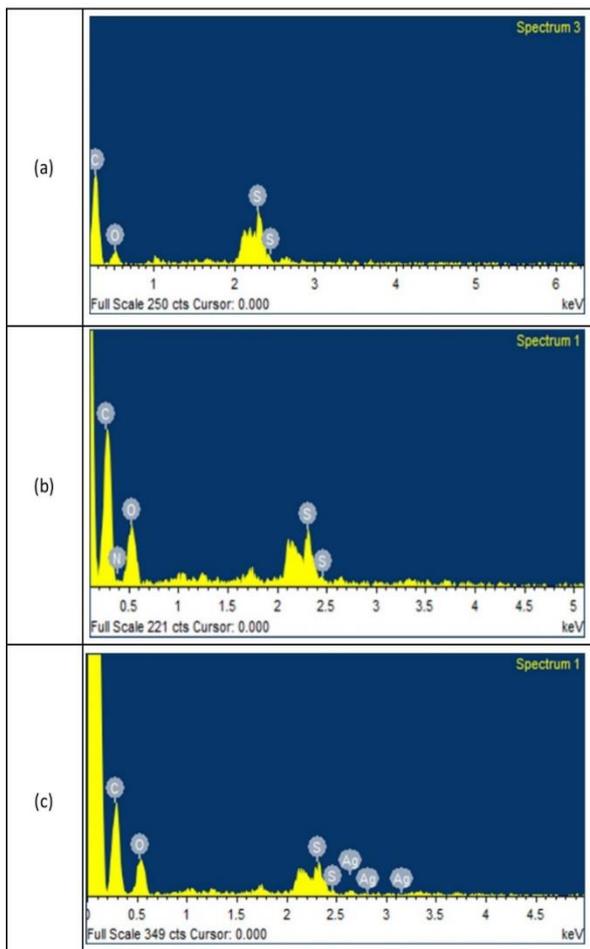
This is because of strong interaction between PANI and membrane surface and no anti – agglomeration agent was added to prevent such phenomena happen. The distribution peak of silver, Ag in membrane was confirmed by the EDX elemental mapping as shown in Figure 3(c). The EDX elemental mapping clearly shows the peak of Ag eventhough slightly low in concentration revealed that the membrane was sucessfully coated using deposited method.

AFM test was conducted to inspect the effect of the additive on the surface roughness for all the coated membrane. The surface roughness,  $R_a$  parameters of the membranes were calculated using XE software Figure 4 shows the surface roughness of membrane that prepared in this area study. The result clearly showed the roughness on membrane increased as coating time increases for both coating materials. This is due to the presence of PANI and silver particles on the membrane surface.

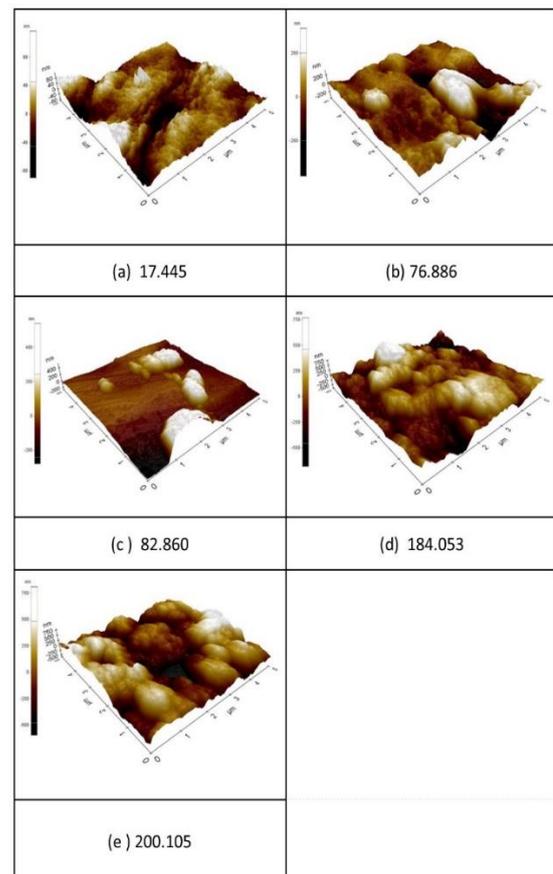
As coating time increases, the surface roughness of

the membrane also increases due to increment of the PANI and PANI/silver particles. As compared with membrane coating with PANI only, the membrane coating using PANI/silver shows higher surface roughness. This might be due to the presence of different particles geometry between silver and PANI increased the membrane surface roughness. According to Mollahosseini et al. [15], the presence of silver able to transform the membrane surface became rougher due to the incompatibility of silver with polymeric membrane. Thus, as coating time increased, the amount of silver on membrane surface will increase and this will attribute to higher surface roughness.

The contact angle measurement was used to investigate the adhesion properties of PANI with water on the membrane surface. Figure 5 show the resulting effect of PSf membrane coated with PANI based on different coating times. As shown in Figure 5, the pristine membrane possesses high contact angle as compared to coated membrane.



**Figure 3.** EDX result for (a) PSf (b) PSf / PANI and (c) PSf/PANI/Ag membrane.



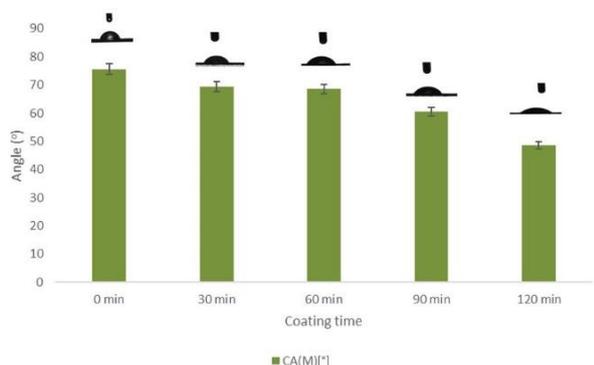
**Figure 4.** SEM images of surface roughness of the membrane (a) PSf only, (b) PSf coating with PANI 30 min, (c) PSf coating with PANI 120 min, (d) PSf coating with PANI/Ag 30 min, (e) PSf coating with PANI/Ag 120 min.



**Figure 5.** Result of water contact angle on PSf membrane coated with PANI

The hydrophilicity of the membrane depends on the lowest possible angle of water as attached to the membrane surface. It was shown in that figure, as coating time increases, the contact angle of the membrane with water also increased. This result might occur due to hydrophilic properties of PANI on top of membrane surface promoted the reduction of contact angle. Furthermore, similar result was observed by Razali et al. [10], which found that PANI tend to reduce membrane contact angle value as its changed the surface of the membrane to become more hydrophilic. Another possibility was due to high surface roughness due to the accumulation of PANI on the membrane surface and provides cake layer formation with non-uniform roughness. Hence, the contact angle of water was affected and water tend to diffuse in the membrane.

The effect of PANI/Ag coated on PSf on the contact angle was depicted in Figure 6. As shown in this figure the contact angle of the membrane was reduced as the coating time increases. However, the result shows that contact angle of the membrane coated with PANI / Ag slightly higher as compared with a membrane coated with PANI only. The result implies that addition of silver decreased hydrophilicity properties of the membrane, although silver was known as hydrophilic materials [17].



**Figure 6.** Result of water contact angle on PSf membrane coated with PANI/Ag

This may be due to the surface properties of solid silver particles which is quite shiny and reflected, that make water to penetrate or adhere on the surface. These results indicates that PANI was more hydrophilic as compared to silver. Thus, the addition of silver has not enhanced the hydrophilicity of membrane.

### 3. 2. Performance of the Membrane Additive Toward 3 Type Protein

The influence of coating time for PANI and PANI/Ag particles on the rejection performance of the membrane is shown in Table 1. As shown in this table, the rejection percentage of the membrane were increased as coating time increases. The result showed that membrane can completely reject BSA. This result might be due to the prepared membrane has a smaller pore size as compared to BSA. The table also shows that coated membrane able to reject pepsin 40 and 30% for trypsin as to compared with the pristine membrane. This may be due to the pore size of the membrane was reduced as the coating is applied on the membrane surface. This result was in line with Arthanareeswaran et al. [18], research stated that BSA was rejected more than Pepsin and Trypsin due to the effect of additive that able to reduce pore size.

**TABLE 1.** Percentage rejected of the membrane against Pepsin, Trypsin, and BSA solution

Membrane	Coating time, t (min)	Percentage rejection (%)		
		Pepsin	Trypsin	BSA
Psf	0	60.42	50	100
Psf + PANI	30	90.21	77.23	100
	60	92.33	80.22	100
	90	96.15	85.23	100
	120	96.15	87.98	100
Psf + PANI + AgNO <sub>3</sub>	30	92.30	80.30	100
	60	96.15	88.40	100
	90	96.20	88.41	100
	120	98.41	90.6	100

## 4. CONCLUSION

In a nutshell, a deposition method was conducted to fabricated PSf membrane with coated layers. Two types of chemical additives that were one with PANI and the other one was PANI/Ag. Surface chemical composition analysis by EDX and morphological characterization confirmed that PANI/silver was deposited for both membrane on the top layer membrane of membrane surface. Further investigation of the surface roughness, shown that the coating with PANI and PANI / Ag result in higher surface roughness value compared to pristine membrane. Slightly higher surface roughness was

noticed in membrane coated with PANI / Ag compared to membrane coated with PANI only. Similar pattern was also observed in hydrophilicity as increased coating particles, hydrophilicity also increased for both coated membrane. This indirectly influence the membrane performance. All of the results regarding the permeation showed that coated membrane with PANI and PANI/Ag was able to enhance the improve the rejection toward protein solution which recorded the 100% BSA and up to 98.4% and 90% for pepsin and trypsin protein rejection.

Lastly, it was found that the membrane performance was improved with presence of PANI and silver on the protein separation process by deposition method.

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اثرات زمان پوشش مختلف PANI / نقره به سطح غشای پلی سولفون (PSf) بر اساس مورفولوژی، زاویه تماس، زبری سطح و BSA، پیپسین و تراوش تیتراپسین مورد بررسی قرار گرفت. غشا با استفاده از روش رسوب فشار به سمت غشاء غربالگری فاز تهیه شد. بنابراین، ذرات PANI مجبور شدند بر روی سطح غشا با نیروی فشار تحت فشار قرار گیرند. مدت زمان پوشش داده شده از 30 دقیقه تا 120 دقیقه لحاظ شد. با این حال، با توجه به سطح صاف PSf، ذرات PANI قادر به بازگشت از سطح PSf بودند. علاوه بر این، حضور PANI / Ag نیز بر روی سطح غشا متمایز بود. مشاهدات شفاف با توجه به تغییر سطح غشا از سطح صاف تا سطح شفاف مشاهده شد. نتایج EDS با استفاده از داده SEM حضور PANI و PANI / Ag را در غشای سطح نشان داد. هیدروفیل بودن غشا با کاهش زاویه تماس با دقت 75 تا 40 درجه برای مدت زمان از 0 تا 120 دقیقه برای غشای پوشش داده شده با PANI اثبات شد. در ضمن، غشای پوشش داده شده با PANI / Ag نیز کاهش را از 75 به 50<sup>o</sup> نشان می دهد. نتیجه این بود که با زبری سطح غشا که بعد از پوشش با PANI افزایش می یابد تا 79٪ و 90٪ پس از پوشش با PANI / Ag پس از اثر 120 دقیقه افزایش می یابد. زبری سطح بالاتر بر عملکرد غشای ریزنمونه تا BSA تأثیر گذاشته است. برای آزمون ردگیری آب، غشا PSf، رد 100٪، 60.42٪ و 50٪ برای BSA، پیپسین و تریپسین را نشان داد. پس از پوشش دادن غشا به مدت 30 دقیقه، 100٪، 90.21٪ و 77.23٪ برای پوشش PANI و 100٪، 92.30٪ و 80.30٪ پس از پوشش با PANI / Ag بدست آمد. برای مدت 120 دقیقه، نتایج نشان می دهد که غشای پوشش داده شده قادر به رد 100٪ BSA، برای پیپسین و تریپسین آن را نشان می دهد 96.15٪ و 87.98٪ در حالی که غشای پوشش داده شده با PANI / Ag، آن نشان می دهد 100٪ BSA و تا 98.41٪ و 90.60 درصد پروتئین پیپسین و تریپسین. در پایان، بررسی عملکرد غشا با حضور PANI و نقره بر روی فرآیند جداسازی پروتئین با استفاده از روش رسوب دهی انجام گردید.

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