Enhancing Contaminant Removal in Sewage Treatment Plant Effluent with PES/Ag Membrane

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ABSTRACT

In this study, a silver-infused membrane was fabricated using an *ex-situ* method that involved blending silver nanoparticles (AgNPs) with Polyethersulfone (PES) as the base polymer in treating sewage treatment plant (STP) effluent. Three distinct membranes denoted as S1(Ag0), S2(Ag0.5) and S3(Ag2.0) were manufactured with varying weight percentages of polymer and silver (Ag) contents. The objective was to investigate the effect of the dosage of AgNPs on membrane characterization and performance, encompassing pure water flux filtration tests, organic rejection tests, and antibacterial properties. The results showed that all PES/Ag membranes demonstrated robust performance in removing total suspended solids (TSS), chemical oxygen demand (COD), apparent colour (Hazen unit), total dissolved solids (TDS), turbidity, conductivity, total Kjeldahl nitrogen (TKN), and *Escherichia coli (E. coli)*, complying to Class IIB (Recreational use with body contact) of the Interim National River Water Quality Standards (INWQS) for Malaysia ruled by the Department of Environment Malaysia (DOE). The results also highlighted that upon the addition of AgNPs, the *E. coli* removal of the membrane S3(Ag2.0) was further improved to 99.87%. The results have evidenced that the PES/Ag membranes could reject *E. coli* effectively, proving their value in treating bacteria-contaminated surface water. In conclusion, this study highlights the effectiveness of PES/Ag membranes as a viable solution for domestic sewage treatment, aligning with stringent water quality requirements.

Keywords: *E. coli* removal, sewage treatment plant effluent, membrane filtration, polyethersulfone, silver nanoparticle

1.0 INTRODUCTION

Water is a necessity for living organisms. Water has become a worrying issue due to water scarcity and water pollution, therefore, upgrading municipal wastewater for indirect drinking water and direct industrial reuse, and internal industrial reclamation have become a common means of increasing the existing water supply. As reported by Bell *et al.* [1], wastewater from commercial, agricultural, domestic, and industrial sectors contains a numberless of pollutants such as toxic compounds (solvents, polychlorinated biphenyls, and pesticides), pathogens (viruses, protozoa, and bacteria), heavy metals (cadmium, lead, mercury, and arsenic), nutrients (phosphorus and nitrogen) and chemical of emerging concerns (drugs, personal care products, and endocrine disrupting substances). These pollutants pose a significant threat to living organisms and the environment.

Removal of contaminants in wastewater can be treated by two conventional methods, chemical precipitation, and biological treatment.

However, the conventional method has some drawbacks. The problems associated with large amounts of sludge treatment [2, 3] and the high cost of operation and maintenance are the major drawbacks of chemical precipitation. For biological treatment, essentially slow process and large area requirements are its limitations [2, 4]. According to Ballet et al. [2], the common limitation is that these methods are not able to achieve sufficiently high efficiency (i.e., < 30% [2, 5]) for the removal of pollutants as stipulated in Class IIB of the Interim National River Water Quality Standards (INWQS) [6]. On the other hand, membrane filtration for wastewater treatment can achieve higher removal efficiency to produce better water quality with zero or less sludge production as stated by Dolar et al. [7]. In particular, nanofiltration (NF) provides higher fluxes at low operating pressure compared to RO which is high permeability and better rejection than ultrafiltration (UF) for lower molar mass molecules and ions [8]. In addition, the NF process also has the advantages of reliability, ease of operation, relatively low energy consumption, and effective removal of contaminants as reported by Izadpanah and Javidnia [9]. According to previous research conducted by Nguyen et al. [10], one major drawback of membrane application is that the membrane is easily contaminated with organic matter and biological species present in the treated wastewater. Membrane fouling is defined as the adsorption or accumulation of organic material in the pore or on the surface of the membrane that affects membrane properties, including rejection and permeability as mentioned by Mohammad et al. [11] and Sun et al. [12]. Thus, Shon et al. [13] emphasized that there is a need to have proper and innovative treatment technology to ensure membranes will

have longevity and low operational costs.

Polyethersulfone (PES) is one of the most commonly used polymers in the preparation of commercial and laboratory NF membranes (Artuğ et al. [14], Van der Bruggen [15] and Vatanpour et al. [16]). However, Zinadini et al. [17] stated fouling is the major problem in polymeric membranes owing to its hydrophobic nature, which allows the accumulation of hydrophobic organic compounds. The repulsion between the hydrophobic foulants and water-repellent membrane can lead to the formation of a stubborn fouling layer thereby decreasing water permeability and rejection performance. To mitigate membrane fouling, an approach of embedding hydrophilic inorganic nanoparticles has been suggested by Vatanpour et al. [16, 18]. In addition, it is believed that the incorporation of inorganic nanoparticles into the membrane matrix can reduce fouling [19] in the membrane due to increased hydrophilicity or changes in membrane morphology as reported by Wu et al. [20]. This is because the modification of membranes incorporated with nanoparticles can trap and deactivate contaminants in the aqueous streams.

While past studies have delved into the application of PES membranes in water treatment, there is a limited exploration of the PES/Ag membranes specifically for the treatment of domestic STP effluent. Notably, earlier research predominantly relied on synthetic wastewater such as humic acid, bovine serum albumin (BSA), and synthetic dye solutions. The actual performance of the membrane in treating STP effluent may differ due to interactions with various pollutants, making it a critical research gap. This presents a challenging yet crucial area that requires further investigation in the realm of wastewater treatment.

Therefore, this research addresses a significant gap in the current understanding of water quality management by focusing on the novel application of PES/Ag membranes. Given the limited scope of existing research on advanced membrane technologies for effectively removing contaminants from STP effluent, this study focuses on evaluating the efficacy of PES/Ag membranes. Specifically, we assess their ability to remove targeted contaminants such as total suspended solids (TSS), chemical oxygen demand (COD), apparent colour (Hazen unit), total dissolved solids (TDS), turbidity, conductivity, total Kjeldahl nitrogen (TKN), and *Escherichia coli (E. coli)* from STP effluent. Additionally, the study examines how varying dope compositions of PES/Ag membranes influence their contaminant removal performance when treating STP effluent. The novelty of this study lies in the innovative use of PES/Ag membranes, offering a unique approach to enhance contaminant removal efficiency. Besides, this study also contributes valuable insights that could pave the way for more effective and sustainable solutions in STP effluent treatment, addressing the growing demand for advanced water quality management practices.

2.0 METHODS

2.1 Materials

Polyethersulfone (PES) granules, sourced from Solvay, UK, with a molecular weight (MW) of 35,000 g/mol, were utilized as the base polymer for the synthesis of the PES/Ag membrane. N-methylpyrrolidinone (NMP) with a minimum purity of 99.5% from Merck, Germany, was chosen as the solvent to dissolve the

PES polymer. Nanoscale silver nanoparticles (AgNPs) at a maximum particle size of 100 nm, consisting of 5 wt% in ethylene glycol, and polyvinylpyrrolidone (PVP) with a molecular weight of 29,000 g/mol were acquired from Sigma-Aldrich, Germany. The PVP and AgNPs were added to the membrane dope solution to control the membrane size and to improve the bacterial removal effectiveness, respectively. In this study, the distilled water was obtained from a laboratory distillation unit (Favorit W4L Water Stills, Malaysia).

2.2 Membrane Fabrication

The composition of the PES membrane dope is outlined in Table 1. Both the PES and PVP were dried in an oven at 60 ºC overnight before dissolving in the NMP solution [21]. The membrane dope solution was poured onto a glass plate and cast using a film applicator at 250 μm [22]. The wet film was left for solvent evaporation at room temperature for 30 s before being immersed in a distilled water bath for phase inversion. The flat-sheet polymeric membrane was then stored wet at room temperature to remove residual solvent.

2.3 Membrane Characterization and Performance Testing

The analysis tests, such as the rejection test and pure water flux rate, were examined using a dead-end filtration unit. The dead-end filtration unit consisted of a nitrogen (N_2) cylinder tank, a high-pressure regulator, a stirred cell (Sterlitech HP4750), a magnetic stirrer, and an electronic balance with a data logging system. Sterlitech HP4750 is a high-pressure (capable of withstanding a maximum of 69 bar) and chemical-resistant stirred cell used in performing a wide variety of membrane

performance tests. The effective membrane area of the stirred cell was 14.60 cm^2. N$_2$ was used to pressurize the cell to the operating pressure. The feed solution (pure water or STP effluent) was fed continuously from the stirred cell to produce the permeate. Meanwhile, the permeate flux was continuously monitored and measured gravimetrically by the electronic balance. The membranes were pressurized or underwent compaction with distilled water at a pressure of 12 bars. These pre-pressurized membranes were tested to determine the pure water flux rate at a pressure of 10 bars membranes S1(Ag0), S2(Ag0.5), and S3(Ag2.0). The pure water flux of the membranes was calculated using Equation (1) [23]:

$$J_w = \frac{Q}{A \times \Delta t \times \Delta P} \qquad (1)$$

Where J_w presents the pure water flux (LMH/bar), Q is the quantity of permeate (L), A is the effective membrane area (m^2), Δt is the sampling time (h) and ΔP presents transmembrane pressure (bar).

The porosity of the membranes was examined using Equation (2) [23].

$$\varepsilon = \frac{(W_w - W_d)\rho_w}{\frac{W_w - W_d}{\rho_w} + \frac{W_d}{\rho_m}} \qquad (2)$$

Where ε is the membrane porosity, W_w represents the weight of wet membrane (g), W_d is the weight of dry membrane (g), ρ_w is the density of water (g/cm^3) and ρ_m is the density of PES polymer (g/cm^3). Meanwhile, the pore radius (r_m) of the membrane was calculated using Equation (3) [24].

$$r_m = \sqrt{\frac{(2.9 - 1.75\varepsilon) \times 8\eta LQ}{\varepsilon \times A \times \Delta P}} \qquad (3)$$

Where r_m is the mean pore radius (nm), ε is the membrane porosity which is obtained from Equation (2), η represents the water viscosity (bar.s), L is the membrane thickness (cm), Q is water flowrate (cm^3/s), A represents membrane effective area (cm^2) and ΔP is transmembrane pressure (Pa).

Table 1 Composition of the PES membrane dope

Membranes ID	Composition (weight %)			
	PES	AgNPs	PVP	NMP
S1(Ag0)	15.0	-	-	85.0
S2(Ag0.5)	15.0	0.5	0.5	84.0
S3(Ag2.0)	20.0	2.0	2.0	76.0

2.4 Sample Collection and Water Quality Assessment

The STP effluent samples were collected from an STP (3.031125N, 101.785005E) located at Taman Suria, Kajang, Malaysia. Upon collection, the samples were immediately transported to the laboratory in a temperature-controlled environment to maintain sample integrity. The water quality and bacterial content of the STP effluent were assessed before and after filtration to investigate the effect of AgNPs incorporated into the modified membranes. Several analytical methods were employed to measure various parameters, including total suspended solids (TSS), chemical oxygen demand (COD), apparent colour (Hazen unit),

total dissolved solids (TDS), turbidity, conductivity, total Kjeldahl nitrogen (TKN) and *E. coli.*

Firstly, the pH value was determined by employing a portable pH meter (Mettler Toledo, LP115 pH meter, USA). To determine the TSS in the water samples, the APHA Standard Methods 2540D was implemented. The COD and TKN values of the water samples were measured using the HACH Standard Method 8000 for Low Range COD and simplified TNTplus 880 s-TKN, respectively with a digestion technique. Meanwhile, the apparent colour was measured using the Platinum-Cobalt Standard Method. All the analyses mentioned above were examined with a spectrophotometer (HACH, DR3900, USA). The TDS value of the water samples was measured with the help of a benchtop conductivity/TDS meter (Jenway, Model 4510, UK). Turbidity measurements were taken using a turbidimeter (Eutech Instruments, TN-100, Singapore). All reagents used in this research were of analytical grade and used as received. The water sample quality evaluation test was conducted three times for each membrane sample to verify accuracy, and the results were then averaged to obtain a mean value.

The filtration performance of the PES membranes in removing bacteria from STP effluent samples was compared. Each water sample, 200 mL in volume, was filtered through the membranes at 1 bar pressure using the dead-end filtration cell. After filtration, a spread plate count analysis was conducted on the filtered water, following the guidelines outlined in APHA Standard Methods 9215C. To perform the spread plate count, a 10× dilution of the filtered sample was prepared and 200 μL of the diluted sample was spread on sterilized agar plates. These plates were subsequently sealed with parafilm and placed in an incubator at 37°C for 24 h. After the incubation period, the count of bacterial colonies on each agar plate was visually assessed and recorded. This information was utilized to assess and compare the bacterial removal efficiency of the membranes in treating the STP effluent samples. The average values of the STP effluent (as the feed in the dead-end filtration unit) concentrations are tabulated in Section 3.0. The solute rejection was then calculated using Equation (4) [25] where C_p and C_f indicate the permeate and feed solute concentrations in ppm, respectively.

$$R\% = \left(1 - \frac{C_p}{C_f}\right) \times 100 \qquad (4)$$

3.0 RESULTS AND DISCUSSION

3.1 Membrane Characterization

All the PES membranes were tested with a pure water filtration test. Water permeability or pure water filtration was evaluated based on the pure water flux results, while porosity and pore size of membranes were calculated using the data obtained from the pure water filtration test. Results of average pure water flux, porosity and pore radius are presented in Table 2.

As previously reported by Wai *et al.* [21], the pure water flux values of all three membranes fall within the flux range of nanofiltration membranes indicating significant pores for efficient water permeability. Adding inorganic substances (i.e., AgNPs) and PVP usually enhances mass transfer during phase change, making larger pores, increased porosity [26], and better water interaction [27]. Yet, for the membrane S2(Ag0.5), adding 0.5wt% AgNPs and PVP slightly reduced porosity. The effect might be attributed to the silver nanoparticles filling the

membrane pores and evenly distributing them across the membrane structure, which resulted in a membrane with less porosity. Similar effects were seen by Mollahosseini *et al.* [28] with AgNPs in polysulfone substrate. Higher NP levels, however, enlarged pores due to clumping. Leaching these clumps later increased membrane porosity. Hence, the membrane S3(Ag2.0), with 2% AgNPs and PVP, displayed the largest pores and highest pure water flux which was in agreement with the statement reported by Acarer [26].

3.2 Membrane Performance Evaluation in terms of Water Quality Assessment

Major parameters of STP effluent after treatment with various PES membranes and their requirements in INWQS are presented in Table 3. The average feed solution concentrations of TSS, COD, apparent colour, TDS, turbidity, conductivity, TKN and *E. coli* are 313.33 mg/L, 90 mg/L, 32 Hazen unit, 147.1 mg/L, 19 NTU, 293.9 μS/cm, 23.9 mg/L, and 14800 colony forming unit (CFU)/mL, respectively.

The average permeate TSS concentrations of S1(Ag0), S2(Ag0.5) and S3(Ag2.0) were found 3.3 mg/L, 1.3 mg/L and 4.0 mg/L, respectively. These concentrations demonstrate compliance with the INWQS for TSS under Class IIB, which sets a maximum permissible limit of 50 mg/L. In all cases, the values of permeate TSS showed almost complete rejection compared to the values observed in the feed. The overall efficiency of TSS removal range is 98.72% - 99.59%. The removal of TSS occurred because the PES/Ag membranes have pore sizes from 19.92 – 25.25 nm which were much smaller than the filter paper used to perform the TSS test (0.45 μm). Thus, the size cutoff operated by the PES/Ag membranes worked effectively to remove the TSS originally present in the feed water and this size cutoff was independent of the particular phase being studied. Therefore, the TSS removal effectiveness was not affected by the specific fouling condition established when the membranes were used to filter the STP effluent in this study.

Table 2 Pure water flux, porosity and pore radius of the PES membranes

Membrane	Average Pure Water Flux (LMH/bar)	Porosity (%)	Pore radius (nm)
S1(Ag0)	2.56 ± 0.58	98.86	19.92
S2(Ag0.5)	2.45 ± 0.31	98.05	20.46
S3(Ag2.0)	3.32 ± 0.56	99.03	25.25

Regarding the examination of turbidity, the initial turbidity level of the feed sample was recorded as 19 NTU. Subsequent filtration through these membranes resulted in permeate that exhibited a turbidity removal rate of over 90%. Notably, the turbidity level of the permeate from membrane S2(Ag0.5) closely resembles the turbidity value of tap water from Kuala Lumpur, which stands at 0.86 NTU [29]. This enhanced performance is likely due to the presence of an Ag added that effectively obstructed membrane pores, thereby improving the rejection of particulates through molecular sieving.

Considering the water quality parameters of COD, apparent color, TDS, turbidity, conductivity, and TKN,

membrane S2(Ag0.5) exhibits a notable removal efficiency compared to membranes S1(Ag0) and S3(Ag2.0), highlighting its efficacy in the process. Higher contaminant removal can be attributed to several reasons, one of which is the fact that the S2(Ag0.5) membrane has the smallest pore radius among all the membranes investigated. In all cases, the values of permeate for COD decreased compared to the values observed in the feed. The rejection of COD was found to be 77.78%, 80.0% and 73.33% for S1(Ag0), S2(Ag0.5) and S3(Ag2.0), respectively, which is less performed than TSS. In fact, small organic molecules, possessing molecular weights smaller than the size of the membrane pores, tend to permeate the membrane. This phenomenon is facilitated by the correlation of COD with the fraction associated with TSS. The removal of colour by the PES/Ag membranes is the utmost important aspect in the context of water reuse. A very high colour rejection is needed to reuse the treated water. In all cases, the values of effluent apparent colour decreased concerning the values observed in the feed. The rejection of apparent colour was found to be 87.5%, 90.63%, and 84.38% for S1(Ag0), S2(Ag0.5), and S3(Ag2.0), respectively.

TDS value in mg/L can be described in terms of specific conductivity in µS/cm multiplied by a factor of 0.64. The feed and permeate TDS concentrations, as well as conductivity, along with the removal efficiencies for various membrane types are presented in Table 3. Notably, higher TDS concentrations and conductivity were observed in the permeate of S3, signifying comparatively poorer performance compared to the other membranes. This outcome can be attributed to the larger pore size (25.25 nm) of membrane S3(Ag2.0), which results in reduced TDS rejection and an increase in conductivity concentration. The data from Table 3 indicate that the membranes exhibited satisfactory performance in removing TDS from surface water samples.

Table 3 Major parameters of STP effluent after treatment with various PES membranes and their requirements in INWQS

| Parameter	Feed Sample	Types of Membrane						INWQS (Class IIB)[6]
		S1(Ag0)		S2(Ag0.5)		S3(Ag2.0)		
		Permeate	Rejection (%)	Permeate	Rejection (%)	Permeate	Rejection (%)	
TSS (mg/L)	313.33	3.3	98.95	1.3	99.59	4.0	98.72	50
COD (mg/L)	90	20	77.78	18	80.00	24	73.33	25
Apparent colour (Hazen unit)	32	4	87.50	3	90.63	5	84.38	150
TDS (mg/L)	147.1	17.7	87.97	10.7	92.73	29.7	79.81	-
Turbidity (NTU)	19.0	1.1	94.21	0.8	95.79	1.2	93.68	50
Conductivity (µS/cm)	293.9	35.8	87.82	21.6	92.65	59.2	79.85	-
TKN (mg/L)	23.9	2.3	90.36	1.4	94.13	2.5	89.53	-
E. coli (CFU/mL)	14800	287	98.06	45	99.70	19	99.87	50*

*Total coliform
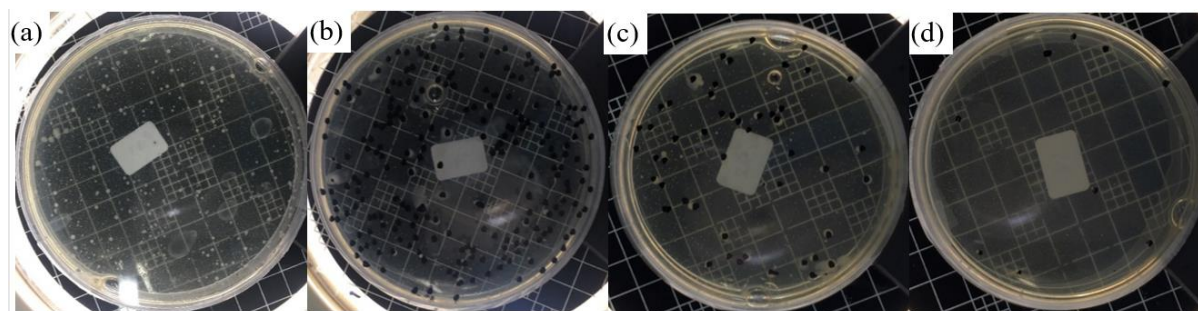
Figure 1 Bacteria count (CFU) in (a) Feed after a 1000× dilution (b) Permeate of S1(Ag0), (c) Permeate of S2(Ag0.5) and (d) Permeate of S3(Ag2.0)

All three membranes exhibited significant TKN rejection, with rejection rates ranging from 89.53% for the S3(Ag2.0) membrane to 94.13% for the S2(Ag0.5) membrane. In all instances, permeate TKN values were much lower than those in the feed, indicating the high effectiveness of PES/Ag membranes in removing TKN from STP effluent. The membrane S3(Ag2.0) with the largest pore size of 25.25 nm had the highest reading of pure water flux of 3.32±0.56 LMH/bar making membrane S3(Ag2.0) less susceptible to TKN rejection.

The effectiveness of removing *E. coli* from modified PES/Ag membranes S2(Ag0.5) and S3(Ag2.0) and unmodified PES membrane S1(Ag0) was compared. Table 3 shows that compared to membrane S1(Ag0), the addition of AgNPs into the membranes led to a noticeably better removal of *E. coli* from the STP effluent. Figure 1 illustrates the bacteria count (CFU) in (a) Feed after a 1000× dilution (b) Permeate of S1(Ag0), (c) Permeate of S2(Ag0.5) and (d) Permeate of S3(Ag2.0). Treatment with membrane S1(Ag0) revealed significant growth of *E. coli*, whereas membranes S2(Ag0.5) and S3(Ag2.0) exhibited considerably reduced *E. coli* growth. These findings align with expectations, with the pristine membrane S1(Ag0) showing the highest *E. coli* content, followed by S2(Ag0.5) and S3(Ag2.0).

The addition of AgNPs into the membranes S2(Ag0.5) and S3(Ag2.0) led to minimal *E. coli* growth, with the membrane S3(Ag2.0) showcasing the most effective inhibition. Furthermore, Andrade *et al.* [30] tested AgNP content from 0.5 wt% to 2 wt%. While 0.5 wt% showed minimal antibacterial activity, both 1 wt% and 2 wt% completely inhibited *E. coli*. This experimental evidence has demonstrated that AgNPs possess antibacterial properties, effectively impeding the growth of *E. coli* by the membrane S3(Ag2.0) with the higher AgNPs content [31]. Silver ions were found to damage bacterial protein structures, highlighting their toxicity and inhibitory effects on microorganisms. Moreover, silver ions were observed to disrupt electron density and cause DNA dimerization in bacteria, further underscoring their inhibitory mechanism [32].

4.0 CONCLUSION

In this research, the incorporation of AgNPs into the PES membrane was achieved through an *ex-situ* blending technique. Three distinct membranes denoted as S1(Ag0), S2(Ag0.5), and S3(Ag2.0), were fabricated, each varying in substrate concentrations and additive loadings of PVP and AgNPs. It was found that the permeability and pore size of the membrane are

positively correlated, for which Membrane S3(Ag2.0) with the largest pore size of 25.25 nm and exhibited the highest value of water flux of 3.32±0.56 LMH/bar. Besides, all fabricated membranes demonstrated excellent performance in treating STP effluent, reporting high removal efficiency for TSS, COD, TDS turbidity and TKN. Besides, the fabricated membranes also reported high *E. coli* removal efficiency, ranging from 98.06% to 99.87%. For the silver-impregnated membrane, the membrane S3(Ag2.0), characterized by the largest pore size, demonstrated slightly lower efficacy in removing most targeted contaminants from the STP effluent as compared to membrane S2(Ag0.5). However, membrane S3(Ag2.0) demonstrated enhanced removal efficiency for *E. coli* attributed to its higher concentration of loaded Ag. The incorporation of PVP and AgNPs into the PES polymeric membrane effectively enhances the filtration performance for both membranes S2(Ag0.5) and S3(Ag2.0), making them well-suited for water reclamation.

CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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