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# Surface Modification of Chitosan Membrane with Ag as Additive

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**Abstract**: The flat sheet membrane of chitosan/silver membrane was prepared via phase inversion technique. In this study, the membrane was added with different composition of silver nitrate as an additive. The analysis includes Scanning Electron Microscopy (SEM), elemental analysis via Energy Dispersive X-Ray (EDX), membrane porosity (%) and Fourier-Transform Infrared Spectroscopy (FTIR). Results showed that the addition of silver nitrate promotes the formation of dense spongy structure of the membrane and hence increases the macrovoids formation. These phenomena then lead to the decreasing in membrane porosity. The addition of silver nitrate reduced the membrane porosity and increased the mean pore size of the fabricated membrane. The chitosan membrane without silver nitrate has the highest membrane porosity which is 38.58 % while membrane with 0.75 wt.% of silver nitrate has the lowest which is 14.38%. Overall, the study shows that silver nitrate addition exhibited chitosan-based membrane enhancement in term of its morphology.

**Keywords**: Chitosan, Silver Nitrate, Phase Inversion Technique, Morphology

## 1. Introduction

In the classification of membrane technology, four systems that widely discussed were reverse osmosis (RO), nanofiltration (NF), microfiltration (MF) and ultrafiltration (UF). The systems selectively separate components over a wide range of particle sizes and molecular weights, from macromolecular materials such as starch and protein to monovalent ions [1]. UF has a broad function in water treatment such as drinking water purification, wastewater treatment, industrial wastewater treatment, seawater treatment and water reuse [2]. The most common types of membrane materials are polysulfone (PS), poly (vinylidene fluoride) (PVDF), polyethylene (PE), polydimethylsiloxane (PDMS), polyimide, poly (vinyl alcohol) and polyethersulfone [3]. This small size pore diameter membrane provides an effective barrier for suspended particles, colloids and pathogens while keeping the mineral content in water intake [4]. It can also remove almost all bacteria, viruses, algae and aquatic

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organisms and claimed to be the most effective technology to guarantee the microbiological safety of water at present [4].

Chitosan is a biopolymer produced from alkaline deacetylation of chitin [5-7]. Chitosan has been used in various fields especially in the water treatment process. In water purification process, chitosan has been used for the removal of oils, grease, heavy metal and fine particulate matter and thus reduced the turbidity of wastewater [5] [8]. Previous study of chitosan membrane discussed on asymmetric membrane [9] with a sponge like structure with large voids [10]. However, the chitosan-based membrane tends to have low water flux and permeation. This has to do with the hydrophilicity characteristic of the chitosan which affect the membrane structure and performance [11].

In order to enhance the effectiveness of hydrophilic chitosan-based membrane, various additives were studied in order to improve the membrane's flux characteristic and membrane fouling [12]. The additives are such as glycerin [9], polyethylene glycol [12] and cellulose acetate [10]. In this study, the effect of silver nitrate on the chitosan membrane has been explored. According to Said *et al*. [13] the addition of silver nitrate into polysulfone membrane has increased the macrovoids of the membrane. The effectiveness of silver nitrate in increasing the macrovoids of the membrane also been stated in Vatanpour et al. [14]. The macrovoids were expanded in number and size with an increasing amount of silver. The usage of silver nitrate as additives is able to fabricate a membrane with dense and spongy morphology which then reduce the pore size of the membrane.

## 2. Materials and Methods

### 2.1 Materials

The casting solutions were made from various ratio of $AgNO_3$ as in Table 1. The low molecular weight chitosan powder was obtained from Sigma Aldrich Co. The analytical reagent grade of 100% glacial acetic acid and sodium hydroxide from EMPARTA have been used as solvent and non-solvent. While, for an additive, silver nitrate solution from QREC (Asia) Sdn. Bhd. was used as an additive. Deionized water was used throughout the study for cleaning, dilution and analytical purposes.

**Table 1: The different composition of casting solution**

| Sample | Composition in casting solution | | |
|---|---|---|---|
| | Chitosan (g) | Acetic acid (ml) | Silver Nitrate (wt. %) |
| 1 | 1 | 50 | 0 |
| 2 | 1 | 50 | 0.5 |
| 3 | 1 | 50 | 0.75 |
| 4 | 1 | 50 | 1.0 |
| 5 | 1 | 50 | 1.5 |

### 2.2 Membrane Modification

The flat sheet of Chitosan/Silver Nitrate membrane was prepared by using 2 % acetic acid as solvent and silver nitrate as the additive. The casting solution was prepared by mixing chitosan subsequently into 2 % v/v acetic acid with continuous magnetic stirring and heating at 60 °C for 4 hours. The silver nitrate was only added when the chitosan was homogenously mixed in the acetic acid. The casting solution was magnetically stirred for another 30 minutes. The casting solution was casted onto glass plate and left for overnight. Glass plate with solution was then immersed into 4 % sodium hydroxide solution for 3 hours. The membrane was then washed with deionized water before it was separated from the glass plate and dried for overnight at room temperature.

2.3 Characterization of Membrane

Membranes were characterized in terms of morphology and elemental content. The common techniques performed for membrane characterization are Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray (EDX), membrane porosity (%) and Fourier-Transform Infrared Spectroscopy (FTIR). Each of the analysis represents the specific function and character of the membrane.

2.3.1 Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) Analysis

The morphological studies of membrane are important as it will represent the structure of the membrane itself. It allows the observation of the pores structure of the membrane prepared [15]. SEM is one of the method that analyze top surface and cross section of the solidified membrane [16] by using gold sputter method with acceleration voltage of 10.0 k [17].

The membrane was freezed in cryogenic condition using liquid nitrogen and then coated with gold [16]. Sputtering coater provides a thin layer acted as conductive material [15] that improve the conductivity of the sample surfaces. If the surface is not conducting, electron charge will be bombarded with the sample and the accumulate charge will create a charging effect that blurred the image formation. The freezed membrane was then broken into pieces [17] and by pasted onto the holder using double sided adhesive carbon tape [18] and transferred into microscopic chamber with sample holder.

EDX is the analysis that integrated with SEM which allow the detection of each element present in the piece of membrane and its mass fraction [22]. The sample was prepared same as the SEM analysis with accelerating voltage 15 kV. The composition of the membrane can be obtained in the form of weight and atomic percentage.

2.4 Membrane Porosity (%)

The ratio of pore volume to geometrical volume of the membrane was used to calculate the membrane porosity [18]. The membrane was cut into 4×4 $cm^2$ and they were immersed into deionized water for 24 hours at 25 °C. Then, the weight of immersed membrane was recorded. The wet membrane pieces then dried in oven with temperatures 60 °C for another 24 hours. The dry state of membrane was weighed. Measurement for their thickness, length, and width of the membrane in wet and dry state were taken by using digital vernier caliper and ruler. The membrane porosity was then calculated using Equation 1.

$$\varepsilon = \frac{(Wwet - Wdry)}{\rho water - Vwet} \qquad \text{Eq.1}$$

Where $W_{wet}$ is the weight of wet membrane (g), $W_{dry}$ is the weight of dry membrane (g), $\rho_{wate}r$ is density of pure water at room temperature (g/$cm^3$) and $V_{wet}$ is the volume of membrane in wet state ($cm^3$).

Mean pore radius was determined by using filtration velocity method as stated by Guerout-Elford-Ferry (Equation 2).

$$R_m = \sqrt{\frac{(2.9 - 1.75\varepsilon) \times 8\eta lQ}{\varepsilon \times A \times \Delta P}} \qquad \text{Eq.2}$$

Where η is the water viscosity, l is the membrane thickness, Q is the volume of permeated pure water per unit time ($m^3 s^{-1}$), Ɛ is the membrane porosity, A is the membrane area and ΔP is the operational pressure.

2.5 Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy can be used to obtain the infrared vibrational absorption spectrum of the membrane material [19] which will allow the identification of the spectra of different functional group present in the membrane [20]. From the FTIR analysis, the information such as chemical bonding and the characteristic of the membrane surface was obtained. During preparation step, sample were cut into 2×2 cm and dried overnight before it was tested [21].

## 3. Results and Discussion

3.1 Effect of Silver Nitrate on the membrane preparation

The preparation of chitosan/silver nitrate membrane was using immersion precipitation method whereby the casting solution immersed into non-solvent. From the process of casting solution preparation until the precipitation start to occur, the effect of silver nitrate was studied. From the observation, when silver nitrate is added, the casting solution becomes darker.

Other than that, the addition of silver nitrate causes a reduction on casting solution viscosity. The higher the amount of silver nitrate added, the lower the viscosity of the casting solution. This observation could be observed in the study by Maximous *et al*. [23] which this condition increases the precipitation rate and promote the formation of sponge-like structure membrane. Silver nitrate also reduced the formation of the air bubble which then will disturb the precipitation of the membrane.

The pH analysis of each casting solution was conducted using pH 700 meter. Overall, all of the casting solution classified in acidic range which below pH 4. Silver nitrate does not affect the pH of the casting solution. The chitosan casting solution is essentially made with 1 g of chitosan powder and 50 ml of acetic acid. The acidity of acetic acid made the casting solution become acidic and ensure the chitosan powder to completely dissolved. Thus, the addition of silver nitrate in the casting solution become more homogenous and resulting uniform distribution of silver nitrate inside the membrane.

3.2 Membrane morphological and Energy Dispersive X-Ray (EDX) Analysis

The cross-section of the chitosan/silver nitrate membrane formed a spongy like structure. As the addition of silver nitrate, the spongy porous structure becomes more stable and uniform. Silver nitrate enhanced the formation of macrovoids in the membrane. The macrovoids formation is increasing and become small in size when the silver nitrate is added. The cross-section of chitosan only membrane as in Figure 1 (a) has no macrovoids formation. The similar observation also has been seen in the study by Said et al. [13] and Hasbullah *et al*. [24].

Different amount of silver nitrate added also portrayed different cross-section images onto SEM analysis. The addition of 0.5 wt. % and 0.75 wt. % give a smooth spongy cross-section. However, chitosan membrane with 0.75 wt. % silver nitrate has pores and macrovoids structure that is smoother among all of the variations. The sponge-like structure of the chitosan/silver nitrate fabricated membrane is due to the higher precipitation rate [23]. The lower the precipitation rate, the lower the possibility for the instantaneous de-mixing occur upon the immersion into the non-solvent [25]. According to Said *et al*. [13] the silver nitrate has shown the increase in water flux through the formation of macrovoid. Not

just that, macrovoid formation also allows the separation to occur for a smaller size colloid molecule in water [26].

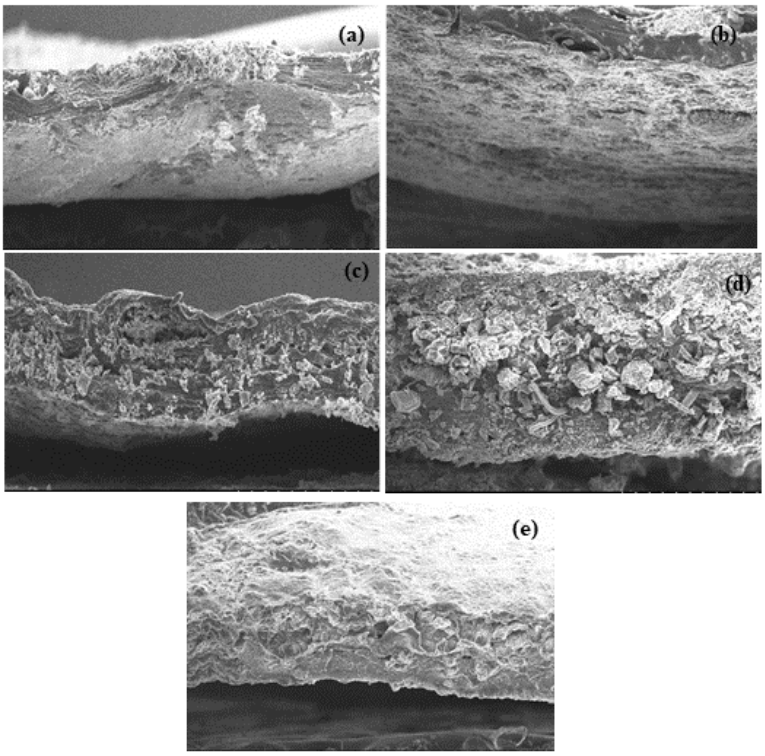


**Figure 1: The cross-section image of chitosan/silver nitrate from SEM analysis with different concentration of silver nitrate;** (a) 0 wt%, (b) 0.5 wt%, (c) 0.75 wt%, (d) 1 wt% and (e) 1.5 wt%.

Analysis of the element in the fabricated membrane is via EDX analysis. This analysis provided the weight in percent of the element presented in the membranes. From the analysis, the result obtained is showed in Table 2.

**Table 2: The element detected by EDX Analysis**

| Element | Weight (%) | | | | |
|---|---|---|---|---|---|
| | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 |
| Carbon | 49.35 | 28.61 | 21.85 | 13.46 | 21.94 |
| Oxygen | 66.62 | 65.85 | 49.66 | 51.75 | 49.95 |
| Nitrogen | - | 2.12 | 12.36 | 3.51 | 9.35 |
| Silver | - | 3.42 | 0.14 | 0.69 | 1.58 |
| Sodium | - | - | 15.99 | 30.59 | 17.18 |

From the analysis, the major element presents in each variation is oxygen. The percentage of their weight made up half of the composition. This is due to each compound that is being blended into casting solution. The main composition of the membrane is chitosan and acetic acid with molecular structure $C_{56}H_{10}N_9O_{39}$ and $CH_3COOH$. For those structure of each compound added for the membrane casting solution, the most element detected by the analysis is carbon and oxygen. As the addition of silver nitrate involved, the silver element also found. However, Sample 2 has a high silver weight which is 3.42% compared to the other variations. The area of analyzed membrane during the EDX might be focused on-site with high silver nitrate since the particles of silver nitrate were scattered in the membrane.

As the detection of sodium in sample 3, 4 and 5 it could be caused by the remaining sodium hydroxide that left during the immersion. The membrane was initially rinsed with deionized water before peeling process. During the process, there must be part of the membrane which is not rinsed properly and cause the sodium to remained and detected by the EDX analyser.

### 3.2 Membrane porosity

The study of membrane porosity (%) is shown in Table 3. The addition of silver nitrate has caused a decrease in the porosity (%) of chitosan/silver nitrate membrane. Without any addition of silver nitrate, sample 1 has the highest porosity which is 38.58%. the addition of silver nitrate in sample 2 may not higher, this is why it has 30.99% which is no big gap as sample 1. However, when the amount of silver nitrate goes up to 0.75 wt. % the porosity reduces almost into half. sample 3, 4 and 5 has porosity 14.38%, 15.36% and 20.16% respectively. The porosity reduction in the membrane is may cause by the distribution of small silver nitrate particle all over the fabricated membrane. This could also be observed in Figure 2 the SEM images show the particle that incorporated in between the membrane dense spongy layer.

According to Sukor. [27], the mean pore size obtained as in Table 3 is classified as ultrafiltration membrane. Based on Table 3, sample 3 shows the bigger pore size which the mean pore size for each membrane increasing from sample 1, 2, 5, 4 and 3. Sample 3 with 0.75 wt.% of silver nitrate has the highest mean pore size which is $2.06 \times 10^{-7}$ μm. The addition of silver nitrate increased the mean pore size of the membrane. The result improved the permeability behavior and its hydrophilicity of the membrane [28].

**Table 3: Membrane porosity and mean pore size**

| Sample | Membrane porosity (%) | Mean pore size (μm) |
|---|---|---|
| 1 | 38.58 | $1.43 \times 10^{-7}$ |
| 2 | 30.99 | $1.66 \times 10^{-7}$ |
| 3 | 14.38 | $2.06 \times 10^{-7}$ |
| 4 | 15.36 | $1.87 \times 10^{-7}$ |
| 5 | 20.16 | $1.72 \times 10^{-7}$ |

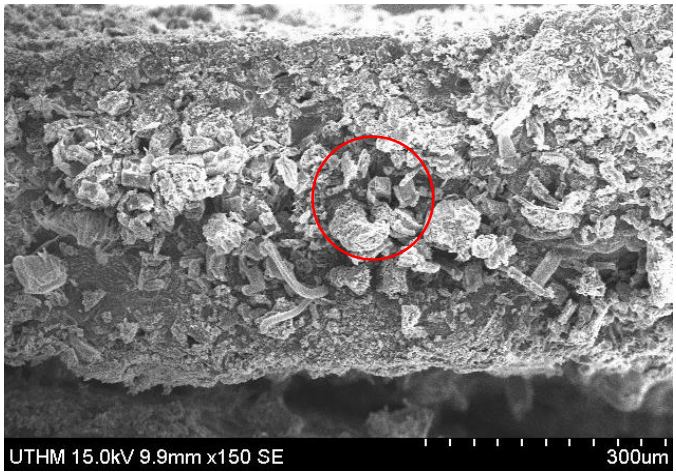


**Figure 2: Silver particle in cross-section of the chitosan/silver nitrate membrane**

3.3 Fourier-Transform Infrared Spectroscopy Field (FTIR)

The studies of the interaction between a chemical compound in the membrane have been done by using FTIR. This analysis also allows the determination of the functional group in a membrane by the spectra as in Figure 3. Apparently, there are no difference spectra at a range of 3300-400 $cm^{-1}$. Even the addition of silver nitrate does not emit different spectra. This shows that silver nitrate does not form chemical bonds with chitosan. The absorption band at 3284.77 $cm^{-1}$, 3275.16 $cm^{-1}$, 3203.37 $cm^{-1}$, 3190.65 $cm^{-1}$ and 3271.19 $cm^{-1}$ for sample 1 until 5 respectively shows the hydroxyl group (-OH) [28]. On the other hand, the peak absorbance at the range 1600-1490 $cm^{-1}$ indicates the amine and carboxamide bonds [29].

The C–O–C linkages in the saccharide structure of the glucosamine also appears in each of the membrane variations. In sample 1, the peak of absorbance appears at 1026.26 $cm^{-1}$ while in sample 2, 3 and 4 is 1068.50 $cm^{-1}$, 1067.78 $cm^{-1}$ and 1027.53 $cm^{-1}$ respectively. Sample 5 has three absorption peak that indicates this linkage structure which is at 1150.71 $cm^{-1}$, 1068.81 $cm^{-1}$ and 899.85 $cm^{-1}$ [30]. According to El-hefian *et al*. [31], the bond of C-C in chitosan bended film can be found at peak around 850 $cm^{-1}$. At the range 550 $cm^{-1}$ the out-of-plane bending NH, out-of-plane bending C−O could be observed. This band appear at all sample [32]. The simplify of all the bands is as in Table 4.

According to Phanjom & Ahmed. [33], the carbonyl group of amino acid and peptide have strong binding ability towards silver. Free amine group or cysteine group also has the ability for the binding. The ability of chitosan to bind with other molecules due to the numerous amounts of amine group [33]. As in Table 4, there is possible binding between chitosan molecules and silver nitrate at absorption peak near 1580 $cm^{-1}$ and also at the range 550 $cm^{-1}$ which is the peak for $NH_2$ functional group.

**Table 4: FTIR spectra for chitosan-based membrane and their functional group**

| Sample | C-C | -OH | $NH_2$ | C-O-C | Out-of-plane NH and CO |
|---|---|---|---|---|---|
| 1 | 863.79 | 3284.77 | 1580.10 | 1026.26 | 554.54 |
| 2 | 863.10 | 3275.16 | 1583.93 | 1068.50 | 554.32 |
| 3 | 863.07 | 3203.37 | 1573.00 | 1067.78 | 554.93 |
| 4 | 863.22 | 3190.65 | 1581.24 | 1027.53 | 555.19 |
| 5 | 862.90 | 3271.19 | 1575.38 | 1150.71<br>1068.81<br>899.85 | 556.03 |

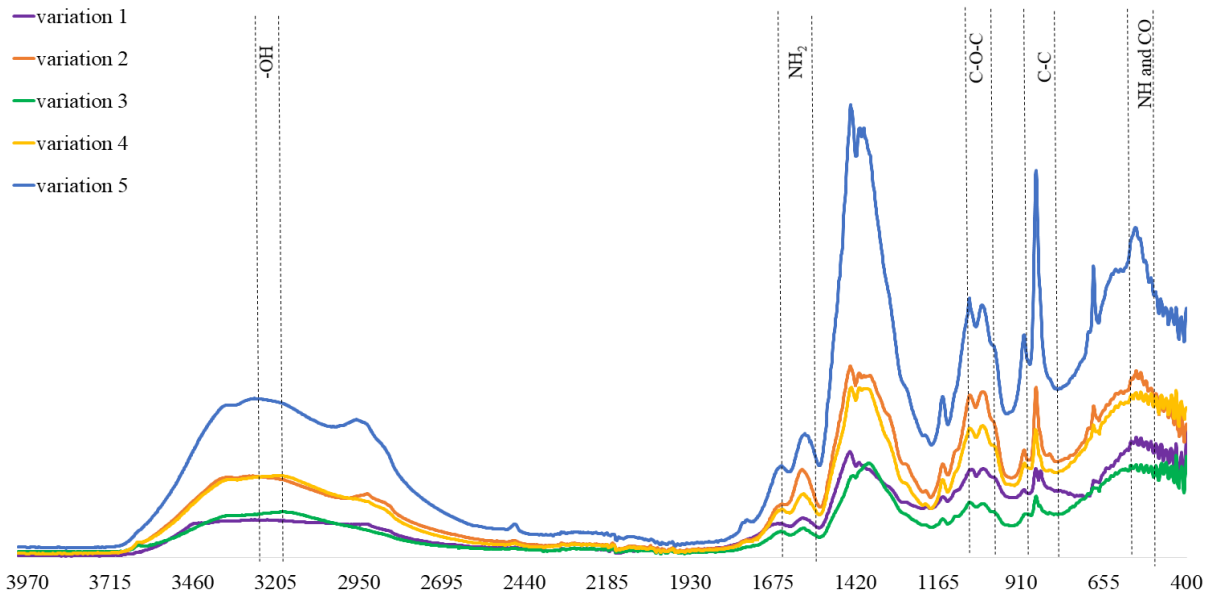


**Figure 3: FTIR spectra of chitosan/$AgNO_3$ with different concentration of $AgNO_3$**; (a) 0 wt%, (b) 0.5 wt%, (c) 0.75 wt%, (d) 1 wt% and (e) 1.5 wt%.

## 4. Conclusion

The flat sheet ultrafiltration chitosan/silver membrane has successfully been prepared using the phase inversion method. The addition of silver nitrate into the membrane does change several characteristics of the casting solution and the flat sheet membrane. Silver nitrate reduces the viscosity of the casting solution which reduce the rate of precipitation of membrane during immersion in non-solvent. As the composition of the silver nitrate increase, the macrovoids also increase. It does also affect the colour of the membrane which the high the composition gives a darker colour. In conclusion, the higher the silver nitrate composition, the better the membrane formed.

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