Silver Oxide Nanoparticles-Modified Poly Vinyl Chloride Membranes to Enhance the Antimicrobial Properties

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INTRODUCTION

As the population grows and more people depend on fewer water sources, the development of a method to reuse wastewater is crucial. Filtration methods, such as membrane technology, play a more crucial role. One technique of physicochemical purification that may be used to remove contaminants is membrane filtration.

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ABSTRACT

Develop nanocomposite membranes with antimicrobial properties and other hydrophilic properties for usage in wastewater treatment. Co-precipitation was used to create nanoparticles of silver oxide (Ag₂O), which were then incorporated into polyvinyl chloride (PVC) flat sheet membranes made using the phase inversion procedure. Crystal structure and the production of Ag₂O nanoparticles may be confirmed by X-ray diffraction (XRD) investigations, Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Scanning electron microscopy (SEM), Attenuated Total Reflection- Fourier transform infrared spectroscopy (ATR-FTIR), Mechanical properties, and contact angle were used to characterize both the unmodified membrane and the membrane that had been modified with Ag₂O in order to examine the distribution of the nanoparticles of Ag₂O within the PVC polymeric solution and the surface properties, thus highlighting the effect of the polymer/filler interfacial interactions. Improving the mechanical and wettability characteristics of the pristine PVC membrane with the insertion of Ag₂O nanoparticles is a major contribution of our study. The mechanical strength of the created modified PVC membrane with Ag₂O was high, with a young’s modulus of 5.5 MPa, while the pristine PVC membrane has a modulus of 3.5 MPa; this is an important characteristic for the application of this type of membrane in a Holding Company of water and wastewater purification. At a permeate water productivity of 3.5 LMH, 92 percent of a target NOC concentration of 50 mg/L of humic acid was removed. Positive antibacterial effects of the modified membranes with Ag₂O were observed.
Membrane technology is a rapid and cheap alternative to traditional chemical treatment procedures including evaporation and thermal treatment (Jaramillo et al., 2017; Sadeghi et al., 2019). Depending on the operational circumstances, the membrane performance might be enhanced by including different materials within the membrane matrix. It is possible to classify the processes used in membrane production, such as TIPS, NIPS, VIPS, and EIPS (Hosseini et al., 2016; Liu et al., 2011). The membranes’ hydrophilicity is enhanced by using hydrophilic nanoparticles. The physical and chemical characteristics of an approach are two examples of how physical-chemical methods might be shown (Hendricks et al., 2018). Nanoparticles may also enhance anti-fouling capabilities. The adaptability of polymer membranes makes them useful in numerous applications, including biosensing (Osaki et al., 2017), ion exchange, and many more (Ran et al., 2017). Membrane distillation (Drioli et al., 2015; Sadeghi et al., 2019) and biological applications (Voicu et al., 2016) have improved performance in purifying water. Polyvinyl chloride (PVC) is one of the most widely used polymers because it has useful qualities and is widely available at a low cost (10% PSf price). PVC’s hydrophobicity makes it inefficient as a filtering medium, decreasing permeation flow, decreasing system efficiency, and raising the cost of membrane cleaning. For this reason, industrial applications need to modify such a membrane (Fang et al., 2017; Aryanti et al., 2015). Filter parameters like hydrophilicity and porosity may be altered and enhanced with the help of nanoparticles due to their distinctive characteristics (Xu et al., 2019). By using a two-step production technique, we were able to effectively create a modified PVC polymer containing Ag2O nanoparticles. First, Ag2O nanoparticles were synthesized by a simple co-precipitation approach; next, a PVC flat sheet membrane with a uniform dispersion of the nanoparticles was created via the phase inversion technique. The PVC/Ag2O membrane that was manufactured had improved properties over those of the neat PVC membrane, including increased stability, consistent morphology, mechanical strength, hydrophilicity, and humic material removal. In addition, the PVC/Ag2O membrane demonstrated potent antimicrobial properties. The PVC/Ag2O membranes that were manufactured have the potential to be used as membranes in wastewater treatment.

**MATERIALS AND METHODS**

Silver nitrate, polyvinyl alcohol, sodium hydroxide, lactose, polyvinyl chloride, and N,N-dimethylacetamide are the first set of materials. All of the chemicals and reagents were from Sigma Aldrich and were utilized in their purest forms.

1. **Ag2O Nanoparticles Preparation:**

At first, 5 g of polyvinyl alcohol was dissolved in distilled water (100 mL) at 80 °C until all crystals dissolved. Silver nitrate (0.33864 g) dissolved in distilled water constitutes the second solution (10 mL). There is also a third solution, which is 10 grammes of sodium hydroxide dissolved in water (30mL). The fourth solution contains 14.418 grammes of lactose dissolved in water (20mL). The first solution was diluted with the second solution, which was diluted with the third solution, which was diluted with the fourth solution. The ingredients were agitated for 18 hours, at which point a thick off-brown colloidal semi-gel formed; this was then rinsed multiple times with distilled water. The precipitate was dried at 80 degrees Celsius for 2 hours, then at 100 degrees Celsius for 1 hour.

2. **PVC Membrane Preparation:**

At 90 °C, 16 g of PVC was added to 84 g of N,N-Dimethylacetamide and dissolved well. The polymer solution was poured onto non-woven polyester on a glass plate, and rinsed with distilled water after being stirred.

3. **PVC/Ag2O Membrane Preparation:**

The non-woven polyester served as the foundation material for all the mix membranes, which were otherwise
manufactured using the standard phase inversion technique.

Table 1 displays the ingredients used to make PVC/Ag₂O casting solutions. PVC polymer content to the total casting solution was 16 wt., with a solvent concentration of 84 wt., for all casting solutions. Once the 0.2 wt. of Ag₂O nanoparticles in the dope were dissolved in N, N-dimethyl acetamide, 16 g. of PVC were added. After that, sonicate all of the solutions at 50 KHZ for 30 minutes then, the polymer solution was mixed well. The polymer solution was left in an incubator overnight to allow the air bubbles to settle out. The polymeric solution was poured over a glass plate and soaked with distilled water. Membrane casting equipment was used to pour the solutions into nonwoven polyester at a thickness of 200 mm, and the polymeric sheets were then instantly cured to generate a wet solid membrane. Deionized water was used to store the membranes.

Table 1: The composition of the fabricated membranes

| Membrane    | Polymer composition | Solvent     |
|-------------|---------------------|-------------|
|             | PVC(wt.%)           | Nanoparticle(wt.%) | (DMAC) (wt.%) |
| Blank PVC   | 16                  | 0           | 84           |
| PVC/Ag₂O   | 16                  | 0.2        | 84           |

4. Characterization:

Scanning electron microscope (SEM, microscope (QUANTA FEG250) and transmission electron microscopes (TEM, Joel (HR)) were used to study the membrane morphologies. X-ray diffraction (XRD, Philips powder diffractometer) was performed to determine the structure of the nanoparticle. ATR-FTIR was measured using Bruker VERTEX 80 (Germany) combined Platinum Diamond ATR. Mechanical testing was applied on prepared membranes, where the Stress at break (σ), and elongation at break (ε br) were recorded on samples with a length of 100 mm and width of 25 mm, using an H5KS universal tensile testing machine.

The membrane porosity (ε) was calculated according to the gravimetric method as shown in equation (1):

\[ ε (%) = \left( \frac{W_w - W_d}{A L P_H} \right) \times 100 \]  

Where, \( W_w \) and \( W_d \) are the mass of wet and dry membranes (gm), respectively. The water density is PH and equal to 0.998 g/cm³, A and L are the membrane area (m²) and thickness (m) respectively.

5. Performance of the Fabricated Membrane in Terms of Flux and Rejection of Natural Organic Pollutants:

Membrane productivity was carried out by a dead-end setup, with an active exposed film area of 28 cm² under pressure from 2 bars for 120 min.

Next, flux of DW was measured from equation (2) (Abdallah et al. 2018):

\[ Flux = \frac{Q}{A T} \]  

Q, A, and T are the amount of product water (L), film area (m²), and collecting time (h), respectively. Salt separation efficiency was obtained by applying equation (3):

\[ Remvlp (%) = \left( 1 - \frac{C_p}{C_F} \right) \times 100 \]  

\( C_p \) and \( C_F \) are humic aqueous solution concentrations (mgL⁻¹) in product and feed, respectively which were specified by UV-Vis spectrophotometer (Agilent Cary 100).

RESULTS AND DISCUSSION

1. Characterization of the prepared Ag₂O:

Figure 1 displays the XRD result of Ag₂O. It could be observed that there were peaks at \( 2θ = 38.26^°, 44.47^°, 64.71^°, \) and \( 77.74^°, 81.91^°, \) which could be indexed as (111), (200), (220), (311) and (222) reflections of the face-centered cubic structure of metallic silver (Zhang et al., 2014; Sadeghi et al., 2015). The size and surface morphology of Ag₂O nanoparticles were determined by SEM, as shown in Figure 2a, and the size of silver nanoparticles was estimated, in the form of Nano crystallites. EDX carried out during the SEM analysis confirmed to the
characteristic peaks of Ag, as shown in Figure 2b. A high-resolution image indicated that the Ag\textsubscript{2}O NPs spread uniformly on the polymeric chain or were embedded in the outer layers of the PVA. Measured by the Nano Measurer software, the average diameter of AgNPs was 4.28:9.19 nm.

TEM analysis of Ag\textsubscript{2}O nanoparticles was also carried out to estimate the size of silver nanoparticles. Particle size was estimated to be in the range of 18 to 50 nm, confirming the results already estimated by SEM and EDX. TEM images are shown in Figure 2c (Wu et al., 2022; Khan et al., 2013).

![Fig.1.XRD for the prepared silver oxide nanoparticles.](image1)

![Fig.2. the morphology characteristics of the prepared silver oxide nanoparticles(a) SEM images (b) the EDAX analysis (c) the TEM image.](image2)

### 2. Chemical Structure in Terms of FT-IR Spectra:

The FTIR spectra of Ag\textsubscript{2}O was presented in Fig.3. The peaks at 1458.84 and 3466.13 cm\(^{-1}\) represent the C=O stretching of carbonyl content and hydroxyl groups respectively, which indicated the hydrophilicity of Ag\textsubscript{2}O (Zhang et al., 2012; Wu et al., 2022).
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3. SEM Images of the Fabricated Membranes:

Figure 4 displays FESEM pictures of the produced membranes' surfaces and cross-sections. Membranes generated using the NIPS approach, in which the morphology of the membrane is impacted by the kinetics and thermodynamics of the polymer/solvent/non-solvent system, all have a finger-like structure with a very thin dense top layer. By loading Ag$_2$O nanoparticles into the polymeric solution, it is possible to observe that the quantity of macrovoids in the modified membrane with Ag$_2$O was reduced, but that the size of the macrovoids and the average size of voids were both increased. It has been hypothesised that Ag$_2$O's existence as a hydrophilic additive during phase inversion accounts for this pattern. The triangle phase diagram's liquid-liquid demixing gap area is typically determined by the interaction parameters between the polymer and solvent, polymer and non-solvent, and solvent and non-solvent (Wu et al., 2022). It is well-established that inorganic nanoparticles present in a material reduce the contact between the polymer and the solvent, hence facilitating the movement of solvent molecules through the material (Bae et al., 2005; Kim et al., 2001). Polymeric compounds like PVC have been shown to have a lower affinity to water than inorganic nanoparticles like Ag$_2$O. So, during phase separation in a water bath, the inclusion of Ag$_2$O nanoparticles in the casting solution accelerates the rate at which water penetrates the cast film. Therefore, Ag$_2$O nanoparticles are thought to enlarge both the surface pores and the void-like holes inside the membranes. EDX analysis was used to investigate how nanoparticles were distributed throughout the membranes. Fig. 4.c depicted the outcomes. Ag$_2$O nanoparticles were found to be distributed regularly across the membranes (Behboudi et al., 2016).
Fig.4: The SEM images of the fabricated membranes, the neat PVC membrane and the modified with Ag\textsubscript{2}O (a) cross-section image (b) top surface image (c) EDAX analysis.

4. Mechanical Properties:

Both tensile strength and elongation decreased as the Ag\textsubscript{2}O was loaded as shown in Table 2. Similar studies are reported (Zhang et al., 2016; Yue et al., 2012). Understandably, a thin layer is responsible for permeation and rejection, and the sublayer mainly acts as mechanical support. As the Ag\textsubscript{2}O increases, more macro voids appeared at the bottom of the sub-layer, reducing the mechanical support of the membrane. What's more, it seems probable that the interaction between PVC and Ag\textsubscript{2}O leads to decreased flexibility of PVC chains (Balta et al., 2012).

| Samples     | Strength (MPa) | Elongation at break (%) |
|-------------|----------------|-------------------------|
| PVC         | 4.599606       | 1.428571                |
| PVC-Ag\textsubscript{2}O | 3.536842       | 1.342857                |

5. Contact Angle:

In order to investigate the effect of Ag\textsubscript{2}O nanoparticles on the hydrophilicity of membranes, a static water contact angle test was applied and the results are shown in Table 3. As observed, the contact angle reduces continuously as Ag\textsubscript{2}O nanoparticles were added to the polymeric solution. For the neat PVC membrane contact angle is around 87.5° and it reduces to 69.5° for 0.2 wt. % Ag\textsubscript{2}O embedded PVC membrane which indicates that incorporation of Ag\textsubscript{2}O nanoparticles increases the hydrophilicity of PVC/Ag\textsubscript{2}O membrane. This is due to the hydrophilic nature of Ag\textsubscript{2}O nanoparticles present on the surfaces of the membrane. A decrease in the contact angle of polymeric membranes by the addition of inorganic nanoparticles has been reported in other studies (Liang et al., 2012; Yu et al., 2015) and the same results were reported in inorganic/PVC composite membranes.
Silver Oxide Nanoparticles-Modified Poly Vinyl Chloride Membranes to Enhance the Antibacterial Properties (Rabiee et al., 2014; Yuliwati et al., 2011). In general, increasing the hydrophilicity of membrane improves fouling resistance as well as water permeability causing water adsorption of hydrophilic membranes is higher than that of hydrophobic ones (Putri et al., 2021).

Table 3. the hydrophilic properties of the fabricated membranes.

| Membrane type | Contact angle value (°) | Contact angle image | Porosity (%) |
|---------------|-------------------------|---------------------|--------------|
| PVC           | 87.5                    | ![Contact angle image](image) | 37           |
| Ag₂O          | 69.5                    | ![Contact angle image](image) | 55           |

6. Impact of Ag₂O Loading Amount on Separation Performance:

The concentrations of PVC and DMF were fixed at 16, and 84 wt. %, respectively. It shows that the addition of Ag₂O in the membrane solution increased the water flux. The addition of Ag₂O by 0.2 wt.% in the PVC solution produced a membrane with a water flux of 5.1 L/m²h at the beginning of the filtration process. The presence of Ag₂O in the membrane structure improved the interaction between membrane and water, which resulted in the formation of greater membrane productivity and permeate flux. However, a higher compact layer in the membrane structure, particularly in the top layer, resulted in the higher rejection of humic substances. Humic substance rejection was achieved above 92% when 0.2 wt.% of Ag₂O was added to the polymer solution (Putri et al., 2021).

![Fig.5. The performance of the fabricated membranes in terms of flux and rejection.](image)

7. Membrane Stability and Recyclability:

The membrane was examined on the permeate flux and rejection during filtration of HA for 10 cycles as shown in Figure 6. It was found that the membrane using Ag₂O did not change the permeate flux and rejection...
values. In membrane filtration, it is preferable to keep a high permeate flux enabling the desired efficiency of the wastewater treatment process; thus, this modified membrane with Ag₃O is preferable to use for wastewater treatment (Kacprzyńska-Gołacka et al., 2020).

Fig. 6. The stability of the fabricated membranes

8. Antibacterial Properties:

In our results, Ag₃O nanoparticles loaded in the modified PVC membrane were most effective against E. coli (Fig. 7). The inhibition of PVC/Ag₃O membrane against E. coli may be estimated between 2.2 nm and 4.4 nm. For S. aureus, however, Ag₃O nanoparticles showed a growth-inhibitory effect inhibitory effect compared with the control unmodified PVC in this condition. The inhibition of PVC/ Ag₃O membrane against S. aureus was estimated to be more than 3 nm. Also, there is no antimicrobial activity in the plate devoid of Ag₃O nanoparticles used as a vehicle containing non-modified PVC, reflecting that antimicrobial activity was directly related to the Ag₃O nanoparticles (Kim et al., 2007).
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Fig. 7. The antibacterial properties of the modified membranes with Ag\(_2\)O and the unmodified membrane and the membrane that had been modified with Ag\(_2\)O in order to examine the distribution of the nanoparticles of Ag\(_2\)O within the PVC polymeric solution and the surface properties, thus highlighting the effect of the polymer/filler interfacial interactions. Adding Ag\(_2\)O nanoparticles successfully into the clean PVC membrane improved its mechanical and wettability qualities, highlighting the importance of this study. In addition to its superior antimicrobial activity, the Ag\(_2\)O-modified PVC membrane that we prepared showed impressive mechanical strength, with a young's modulus of 5.5 MPa compared to the neat PVC membrane's 3.5 MPa. This is an important quality for a membrane to possess in a wastewater purification-related Holding Company. Furthermore, with permeate water productivity of 3.5 LMH, NOC removal from 50 mg/L humic acids was maximized at 92%.

Conclusion:

Hydrophilic membranes modified with Phase inversion were used to create Ag\(_2\)O. Since these membranes are antimicrobial, they may be utilised to treat wastewater.

X-ray diffraction (XRD) analysis, Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) were used to characterize the produced Ag\(_2\)O nanoparticles and validate their crystal structure and shape, respectively. Scanning electron microscopy (SEM), Attenuated Total Reflection- Fourier transform infrared spectroscopy (ATR-FTIR), Mechanical properties, and contact angle was used to characterize both the unmodified membrane and the membrane that had been modified with Ag\(_2\)O in order to examine the distribution of the nanoparticles of Ag\(_2\)O within the PVC polymeric solution and the surface properties, thus highlighting the effect of the polymer/filler interfacial interactions. Adding Ag\(_2\)O nanoparticles successfully into the clean PVC membrane
improved its mechanical and wettability qualities, highlighting the importance of this study. In addition to its superior antimicrobial activity, the Ag₂O-modified PVC membrane that we prepared showed impressive mechanical strength, with a young's modulus of 5.5 MPa compared to the neat PVC membrane's 3.5 MPa. This is an important quality for a membrane to possess in a wastewater purification-related Holding Company. Furthermore, with permeate water productivity of 3.5 LMH, NOC removal from 50 mg/L humic acids was maximized at 92%.

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