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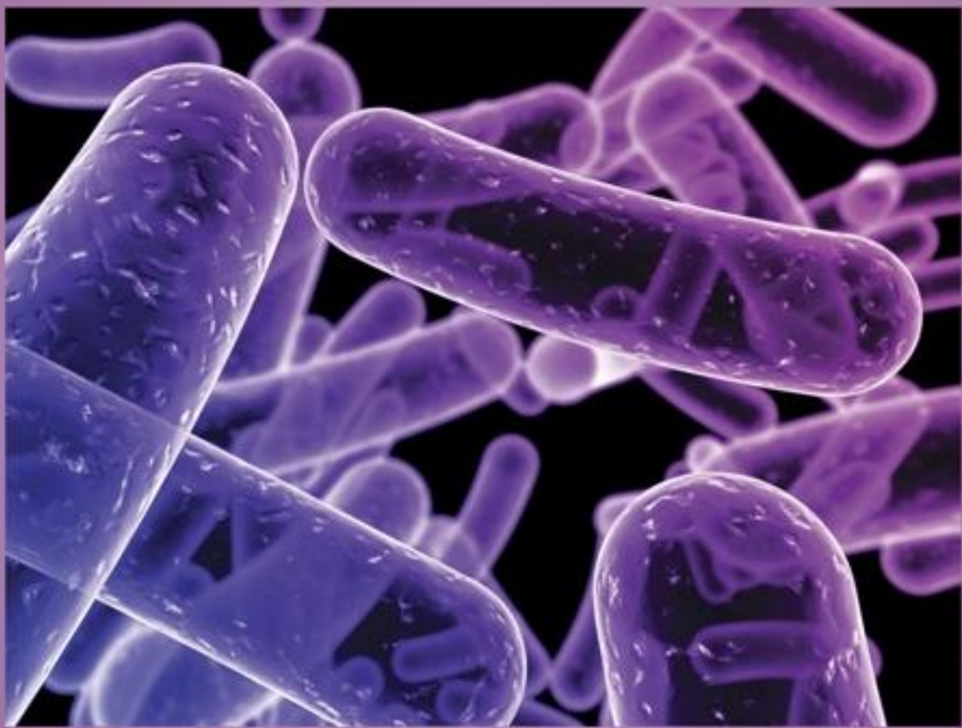

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Silver Oxide Nanoparticles-Modified Poly Vinyl Chloride Membranes to Enhance the Antibacterial Properties

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ABSTRACT

Develop nanocomposite membranes with antibacterial properties and other hydrophilic properties for usage in wastewater treatment. Co-precipitation was used to create nanoparticles of silver oxide (Ag_2O), which were then incorporated into polyvinyl chloride (PVC) flat sheet membranes made using the phase inversion procedure. Crystal structure and the production of Ag_2O 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 Sused to characterize both the unmodified membrane and the membrane that had been modified with Ag_2O in order to examine the distribution of the nanoparticles of Ag_2O 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 Ag_2O nanoparticles is a major contribution of our study. The mechanical strength of the created modified PVC membrane with Ag_2O 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 Ag_2O were observed.

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.

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 e*t 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; Aryanti1a *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 Ag_2O nanoparticles. First, Ag_2O 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/ Ag_2O 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/Ag_2O membrane demonstrated potent antimicrobial properties. The PVC/Ag_2O 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. Ag_2O 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/Ag_2O 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/Ag_2O 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 Ag_2O 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/ Ag_2O	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):

$$\varepsilon\ (\%) = \left(\frac{W_{w-}w_d}{ALP_H}\right)100 \qquad (1)$$

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/cm^3, A and L are the membrane area (m^2) 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 cm^2 under pressure from 2 bars for 120 min.

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

$$Flux = \frac{Q}{AT} \qquad (2)$$

Q, A, and T are the amount of product water (L), film area (m^2), and collecting time (h), respectively. Salt separation efficiency was obtained by applying equation (3):

$$Removl\ (\%) = \left(1 - \left(\frac{C_P}{C_F}\right)\right)100 \qquad (3)$$

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 Ag_2O:

Figure 1 displays the XRD result of Ag_2O. 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 Ag_2O 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 conformed to the characteristic peaks of Ag, as shown in Figure 2b. A high-resolution image indicated that the Ag_2O 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_2O 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.

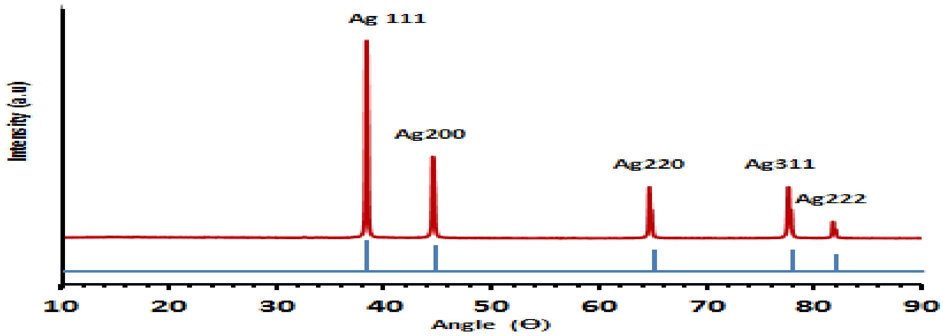


Fig.1.XRD for the prepared silver oxide nanoparticles.

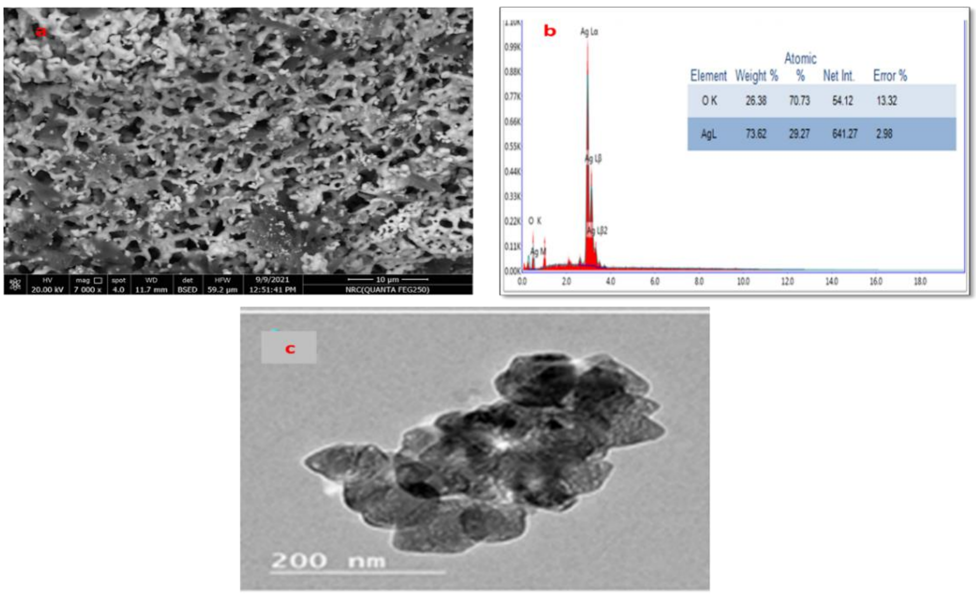


Fig.2. the morphology characteristics of the prepared silver oxide nanoparticles(a) SEM images (b) the EDAX analysis (c) the TEM image.

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

The FTIR spectra of Ag_2O was presented in Fig.3. The peaks at 1458.84 and 3466.13 cm^{-1} represent the C=Ostretching of carbonyl content and hydroxyl groups respectively, which indicated the hydrophilicity of Ag_2O (Zhang *et al*.,2012; Wu *et al*.,2022).

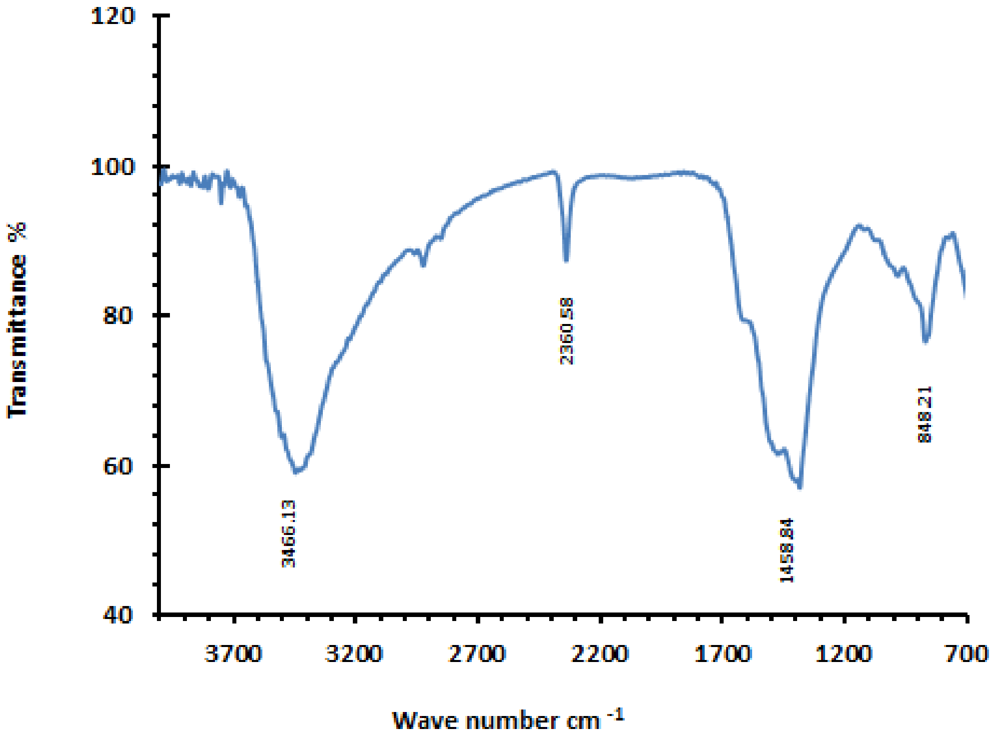


Fig.3. The FTIR of the prepared silver oxide.

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_2O nanoparticles into the polymeric solution, it is possible to observe that the quantity of macrovoids in the modified membrane with Ag_2O was reduced, but that the size of the macroviods and the average size of voids were both increased. It has been hypothesised that Ag_2O's existence as a hydrophilic additive during phase inversion accounts for this pattern. The triangle phase diagram's liquid-liquid de-mixing 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_2O. So, during phase separation in a water bath, the inclusion of Ag_2O nanoparticles in the casting solution accelerates the rate at which water penetrates the cast film. Therefore, Ag_2O 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_2O nanoparticles were found to be distributed regularly across the membranes (Behboudi *et al.*,2016).

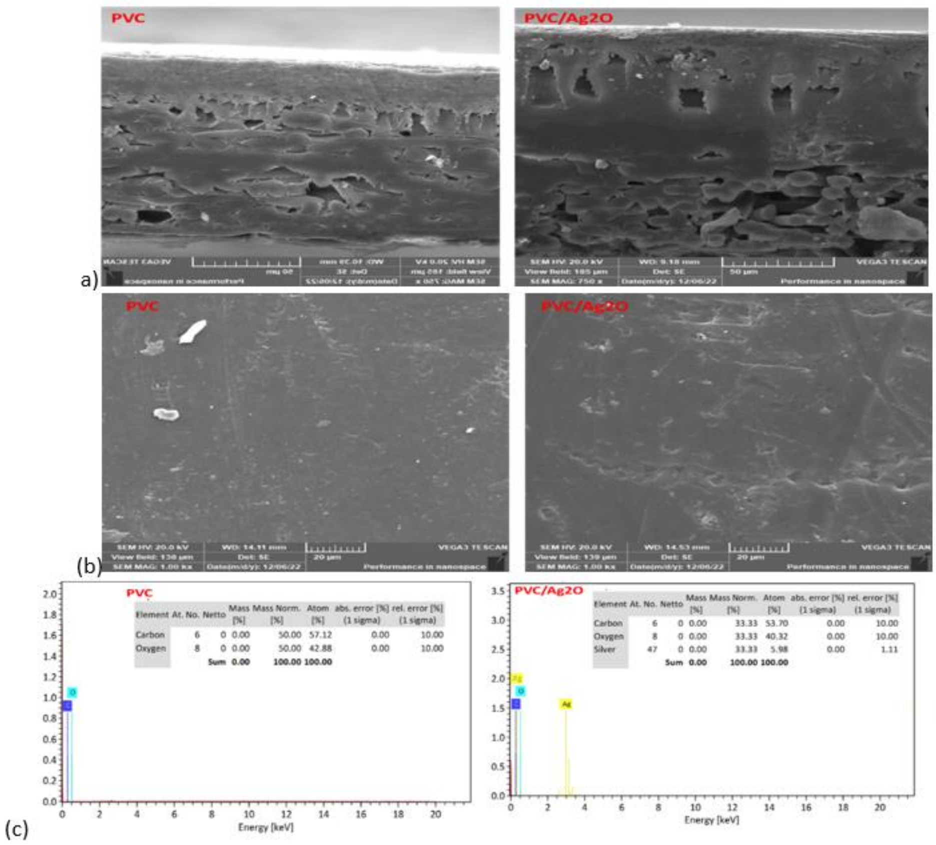


Fig.4: The SEM images of the fabricated membranes, the neat PVC membrane and the modified with Ag_2O (a) cross-section image (b) top surface image (c) EDAX analysis**.**

4.Mechanical Properties:

Both tensile strength and elongation decreased as the Ag_2O 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_2O 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_2O leads to decreased flexibility of PVC chains (Balta *et al*.,2012).

Table 2. The mechanical properties values of the fabricated membranes**.**

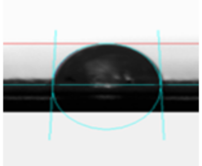
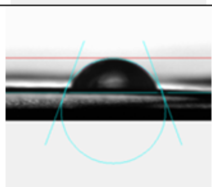
Samples	Strength (MPa)	Elongation at break (%)
PVC	4.599606	1.428571
PVC-Ag_2O	3.536842	1.342857

5.Contact Angle:

In order to investigate the effect of Ag_2O 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_2O nanoparticles were added to the polymeric solution. For the neat PVC membrane contact angle is around 87.5º and it reduces to 69.5 º for 0.2 wt. % Ag_2O embedded PVC membrane which indicates that incorporation of Ag_2O nanoparticles increases the hydrophilicity of PVC/Ag_2O membrane. This is due to the hydrophilic nature of Ag_2O 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

(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		37
Ag_2O	69.5		55

6.Impact of Ag_2O 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 Ag_2O in the membrane solution increased the water flux. The addition of Ag_2O by 0.2 wt.% in the PVC solution produced a membrane with a water flux of 5.1 L/m^{2}h at the beginning of the filtration process. The presence of Ag_2O 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 Ag_2O was added to the polymer solution (Putri *et al.*,2021).

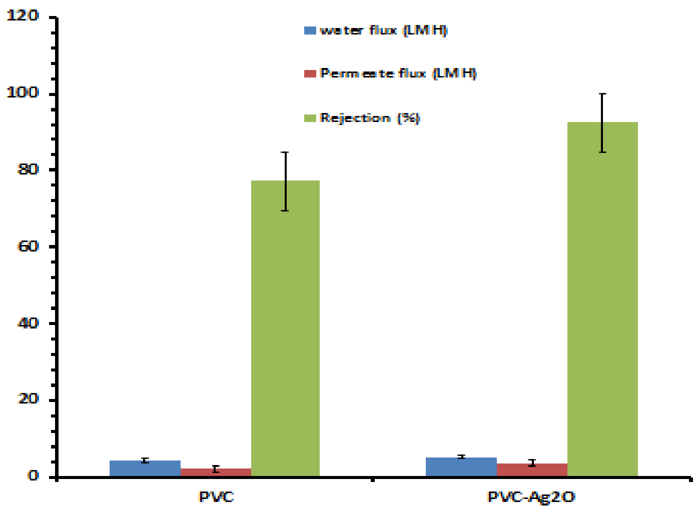


Fig.5. The performance of the fabricated membranes in terms of flux and rejection**.**

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 Ag_2O 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 Ag_2O is preferable to use for wastewater treatment (Kacprzyńska-Gołacka *et al.*,2020).

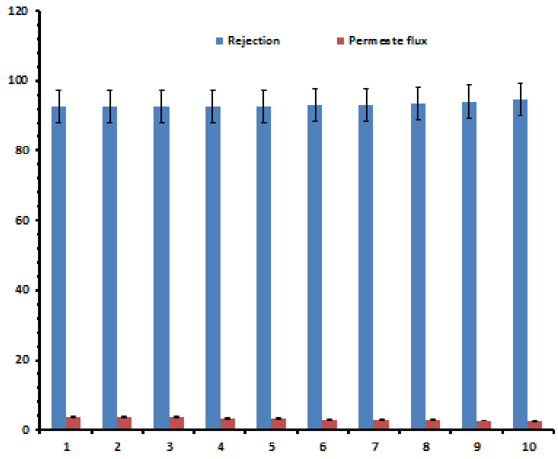


Fig. 6. The stability of the fabricated membranes

8.Antibacterial Properties:

In our results, Ag_2O nanoparticles loaded in the modified PVC membrane were most effective against E. coli (Fig. 7). The inhibition of PVC/Ag_2O membrane against E. coli may be estimated between 2.2 nm and 4.4 nm. For S. aureus, however, Ag_2O nanoparticles showed a growth-inhibitory effect inhibitory effect compared with the control unmodified PVC in this condition. The inhibition of PVC/ Ag_2O membrane against S. aureus was estimated to be more than 3 nm. Also, there is no antimicrobial activity in the plate devoid of Ag_2O nanoparticles used as a vehicle containing non-modified PVC, reflecting that antimicrobial activity was directly related to the Ag_2O nanoparticles (Kim *et al*.,2007)**.**

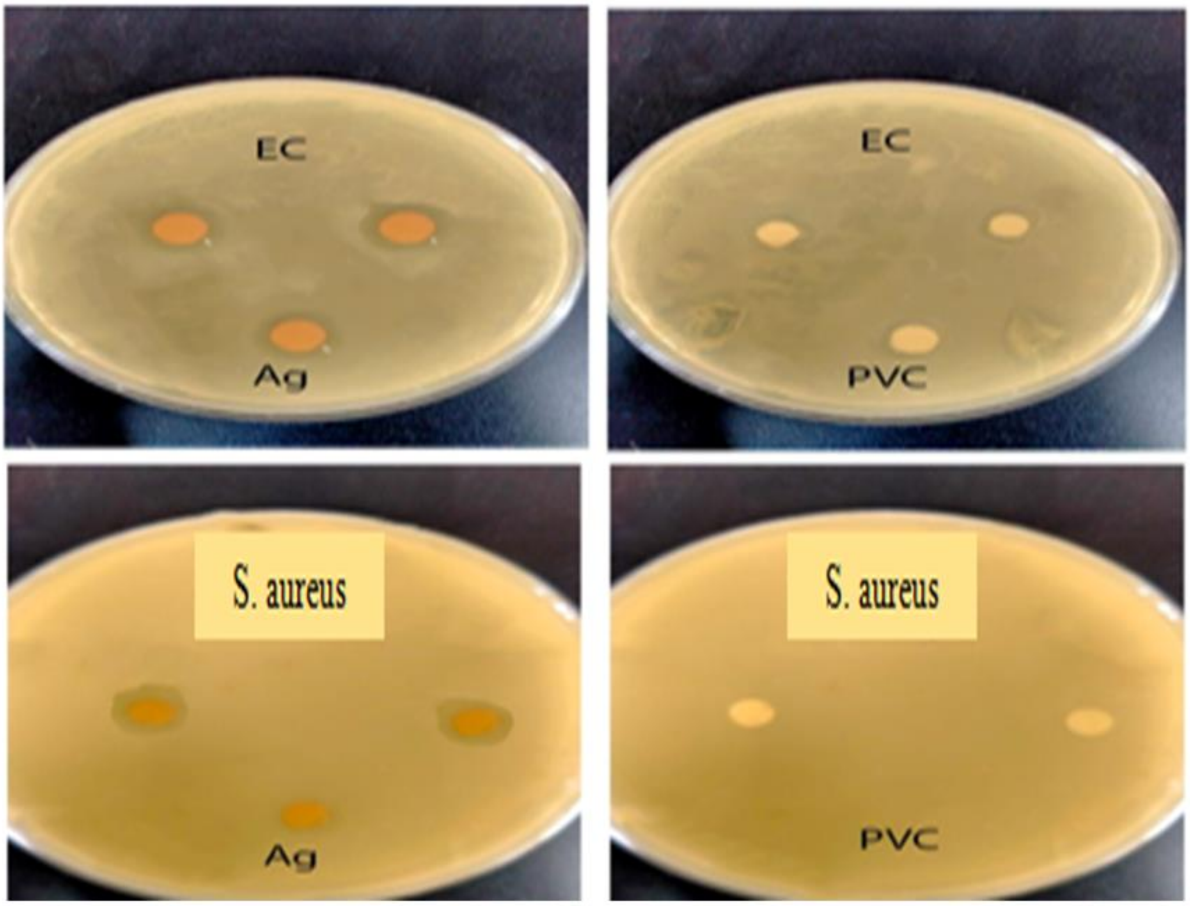


Fig.7. The antibacterial properties of the modified membranes with Ag_2O and the unmodified membrane and the membrane that had been modified with Ag_2O in order to examine the distribution of the nanoparticles of Ag_2O within the PVC polymeric solution and the surface properties, thus highlighting the effect of the polymer/filler interfacial interactions. Adding Ag_2O 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_2O-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_2O. 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_2O 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_2O in order to examine the distribution of the nanoparticles of Ag_2O within the PVC polymeric solution and the surface properties, thus highlighting the effect of the polymer/filler interfacial interactions. Adding Ag_2O 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_2O-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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