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Incorporation of Antifouling Based Nanoparticles in Ultrafiltration Membrane for Improving Water Permeability and Mitigate Microbial Fouling

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ABSTRACT

Membrane biofouling is one of the major problems that persist in separation field. Biofouling contributes to flux decline and energy consumption in these processes. In order to minimize bio fouling, this study deals to obtain both organic antifouling and anti-microbial properties in PVDF membrane, where silver and gold nanoparticles were incorporated. The microbial fouling properties of this membranes was investigated using as *E. coli, Pseudomonas aeruginosa, Staphylococcus aureus, and Bacillus subtilis* model foulants. The zone of inhibition obtained for each microorganism shows the microbial resistance of the silver and gold nanoparticles incorporated membranes. The experimental results confirm that the incorporation of silver and gold nanoparticles in PVDF membrane can mitigate biofouling.

Keywords: Ag nanoparticles, Au nanoparticles, PVDF membrane, antifouling, membrane biofouling

1.0 INTRODUCTION

Membrane technology is widely used in industries for the past few years due to its reliable effluent quality, and improved membrane design [1-3]. Membrane separation is very promising in water treatment, because of its potential to remove particles like micro- organisms, organic pollutants, and inorganic compounds. However, the major operational problem occurring in membrane separation is fouling. Membrane fouling causes flux decline and energy consumption which may lead to the decrease of the life of the membrane [4]. Biofouling is caused by organic matter derived from microbial cellular debris. It occurs due to the complex interactions between the micro-organisms, membrane materials, dissolved substances and the flow parameters [5]. Marshall and Blainey studied the force which transport micro-organism to a surface. The major mechanism for the transport of micro-organism is fluid dynamic forces [6]. Attachment of micro-organisms to the surface is the first step of membrane fouling [7]. This attachment process is affected by factors such as membrane material [8], hydrophobicity [9], membrane surface change [9] and roughness of membrane surface [10]. Biofouling contributes to more than 45% of membrane fouling [11] and has been reported as a major problem in different membrane filtration processes [12]. Therefore, finding efficient way to control biofouling is technologically significant. Nanotechnology is a rapidly growing field with applications in science and technology for the

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manufacture of new materials [13]. Metal nanoparticles have been studied extensively because of their unique physicochemical characteristics which include catalytic activity, optical properties, electronic properties anti-microbial activity and magnetic properties [14], [15].

Incorporation of these nanoparticles into polymeric matrix has attracted the interest because of the efficient performances of the resultant membrane like higher selectivity and permeability for separation of liquids [16] and the reduction of formation of biofilm. It has also been reported that addition of nanoparticles increased the permeability, strength and hydrophilicity of the membranes [17], [18]. Silver and gold nanoparticles have become a typical example of biocides, due to their effective antibacterial properties [19], [20] and they are also been used extensively in many microbicidal fields. Their anti-microbial activity attributed to their strong cytotoxicity against several bacterial cells [21]. These particles can interact with the functional groups on the bacterial cell surface and inactivate them [22], [23]. Silver nanoparticles incorporated into polysulfone membranes showed excellent anti-microbial properties towards variety of bacteria including *E. coli* [24] and *Pseudomonas Mendocino KR1* [25]. Similarly, the incorporation of silver nanoparticles into poly ether sulfone membrane also exhibited good anti biofouling and hydrophilicity performance [26]. Polyamide [27], chitosan [28] and polyether sulfone [29] membranes are also been synthesized with silver nanoparticles. There are limited researches over the use of silver nanoparticles onto PVDF membranes. Similarly, gold nanoparticles are also rarely involved in the process of incorporation into membranes. Here, we introduced silver and gold nanoparticles into PVDF membrane and studied the extent to which biofouling is controlled. These nanoparticles were synthesized from bamboo leaves. The phase inversion technique of nanoparticles was combined with membrane. The silver and gold nanoparticles were synthesized from Bamboo leaves. UV Spectroscopy confirmed the synthesized silver and gold nanoparticles. The microbial fouling properties of this membrane was investigated using *E. coli, Pseudomonas aeruginosa, Staphylococcus aureus, and Bacillus subtilis* as model foulants. The effect of pure water flux, filtration performance and fouling analysis were systematically investigated. XRD was done to analyze and confirm the incorporation of nanoparticles onto the membrane. Further SEM was also carried out to investigate the membrane surface morphology and its structure.

2.0 MATERIALS AND METHODS

Commercial grade PVDF was procured from Solvay Process India Ltd, was used without any further treatment. N-Methyl-2-pyrrolidone (NMP) was obtained from Qalinge’s Fine Chemicals, Glaxo India Ltd., India, is used as a solvent to dissolve the polymer. Silver nitrate and Auric chloride were purchased from Sigma-Aldrich Ltd India. Deionized and distilled water was used for the recrystallization of CA, preparation of dextran and for the preparation of gelation bath.

2.1 Synthesis of Gold and Silver Nanoparticles

Bamboo leaves were removed from

stems and washed with distilled water. The 20 g of *bambusa bamboos* leaves were added to 100 ml of distilled water and boiled for 60 min. The *bambusa bamboos* broth was centrifuged at 10000 rpm for 10 min. Then 100 ml of 1 mM silver nitrate solution was prepared, the leaf extract was mixed with 1mM of silver nitrate solution and allowed to react at 30°C. The color was changed from white color to dark brown color, which appearance confirms the formation of silver nanoparticles. Similarly, the gold nanoparticles synthesized by the obtained *bambusa bamboos* broth react with 1 mM Auric chloride solution and allowed the solution to react at 30°C.

Ultra filtration equipment used here is Dead-End Ultra Filtration Cell of total capacity 300 ml, which can withstand a pressure of 6 bars and 500 rpm with a magnetic stirrer and a temperature probe. Bamboo leaves were separated from stems and washed with distilled water. 200 ml of distilled water was added to 40 gm of leaves and boiled for 60 min. The leaf broth was centrifuged at 10,000 rpm for 10 mins. Then 100 ml of 1 mM silver nitrate solution and 1 mM Auric chloride solutions were prepared, and the leaf extract was mixed with the desired solutions separately and allowed to react at room temperature. The color change from white to dark brown, indicated the visual confirmation of silver nanoparticles, and the color change from pale yellow to light reddish brown indicated the confirmation of gold nanoparticles.

2.2 Characterization of Nanoparticles

UV Spectroscopy Provides information about the presence and absence of silver and gold nanoparticles. The absorption or reflectance in the visible range directly affects the perceived color of the chemicals involved. The presences of silver nanoparticles were confirmed by observing the peak in the range of 400 nm to 450 nm. The presences of gold nanoparticles were observed in the range of 500-530 nm.

2.3 Preparation of PVDF Membrane

PVDF membranes using silver and gold nanoparticles were prepared by phase inversion method. Initially silver and gold nanoparticles were sonicated the casting solution was prepared by dissolving PVDF in NMP solvent in a round bottom flask and subjected to constant stirring for 4hrs at room temperature to obtain a homogenous solution.

Different concentrations of biopolymers are mixed with 10% of PVDF in the range of 2.5% of nanoparticles. The casting solution for membrane without silver and gold nanoparticles was prepared by mixing PVDF (wt%) in NMP (wt%). The cast solution was drawn manually with a speed of 20 mm/s using doctor’s blade with gap 150µm. The ratio of PVDF/Solvents studied is labeled as AgNPs coated membrane, AuNPs coated membrane and uncoated neat PVDF membrane. The fabricated membranes were washed with distilled water and stored in the 0.1% w/w formalin solution to avoid microbial attack.

2.4 Characterization of Membrane

2.4.1 Membrane Filtration Studies

The pure water flux of the membrane was measured at 500 KPa trans membrane pressure in a dead-end type ultra-filtration cell (Model: XFUF076-01, Millipore, USA) with an effective filtration area of 38cm^2.water flux was calculated over measured time intervals using the following equation

$$J_w = \frac{Q}{A \cdot \Delta T} \quad (1)$$

Where,

Jw = Pure water flux, $lm^{-2}h^{-1}$;
Q = Quantity of pure water permeated, l;
A = Membrane area, m^2;
ΔT = Sampling time, h.

The permeation and separation properties of membranes were investigated with a dead-end ultra-filtration cell at room temperature. Pure water and *Staphylococcus aureus* culture were employed as the feed for evaluation of membrane performance. In all experiments, distilled water was used to characterize the pure water flux of membranes. The membranes were pre-compressed with pure water at 500 KPa for 60 min. Then, the pure water flux and *Staphylococcus aureus* permeation was evaluated at 500 KPa for 60 min. Membranes were washed for 15 min after 60 min of culture filtration and the water flux of washed membranes was measured.

2.4.2 Morphological Studies

The morphological characterization of membranes was studied using scanning electron microscope (SEM). The membranes were cut into pieces of various sizes, mopped with a filter paper. The samples were mounted on the sample holders and platinum sputtered to provide electrical conductivity to the membranes. The top surface and cross-sectional views of membranes were observed by SEM under high vacuum condition at 25 KV and at different magnifications.

2.4.3 XRD

The distribution of nanoparticles onto PVDF was analyzed using X-Ray Diffractometer. (Rigaku, Miniflux-300). XRD was operated at 40 KV voltage and 40 mA current using Cu kλ radiation (λ=mm) from 10º to 90º (2θ) at scan speed of 10 per min with 0.010 step size.

2.4.4 FTIR

Before Fourier transform Infrared (FT-IR) measurement, the membranes were dried for 2 hrs. via exposure to air. FTIR spectra were obtained by the Fourier transform infrared spectroscopy (Thermoscientific, Nicolet iS5) instrument equipped with the horizontal attenuated total reflectance accessories. The spectrum of each membrane was observed in the wavenumber range $500cm^{-1}$ to 4500 cm^{-1} with the setup range of $500cm^{-1}$ the spectral measurements were used to analyze the functional groups on the membrane surfaces.

3.0 RESULTS AND DISCUSSION

3.1 Characterization of Nano Particles

3.1.1 UV Spectral Analysis of Silver and Gold Nanoparticles Synthesis

Figure 1 and Figure 2 illustrate the reading of UV-Vis spectra of silver and gold nanoparticles at the absorbance range from 0.7nm between the wavelengths of 10nm to 1000nm. The peak was obtained at 450nm indicating the presence of silver nanoparticles and spectra at the absorbance range from 0.5 nm between the wavelengths of 100 nm to 900 nm, peak was obtained at 500 nm indicating the presence of gold nanoparticles.

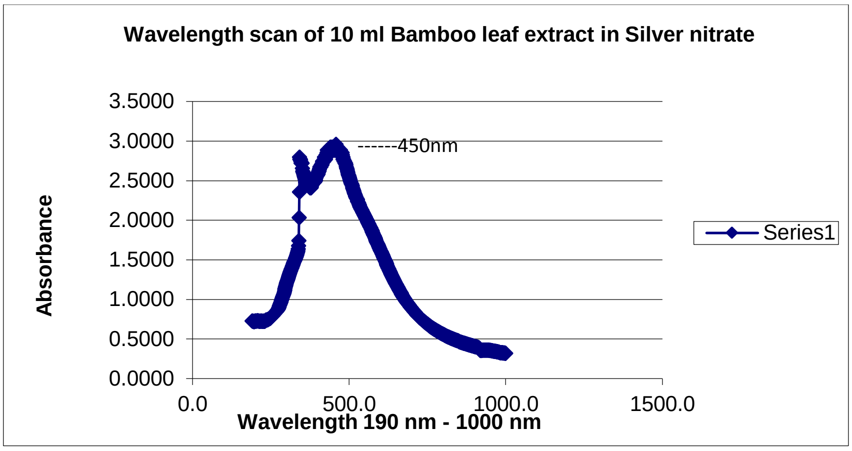

Figure 1 UV Visible absorption spectra indicating the presence of silver nanoparticle

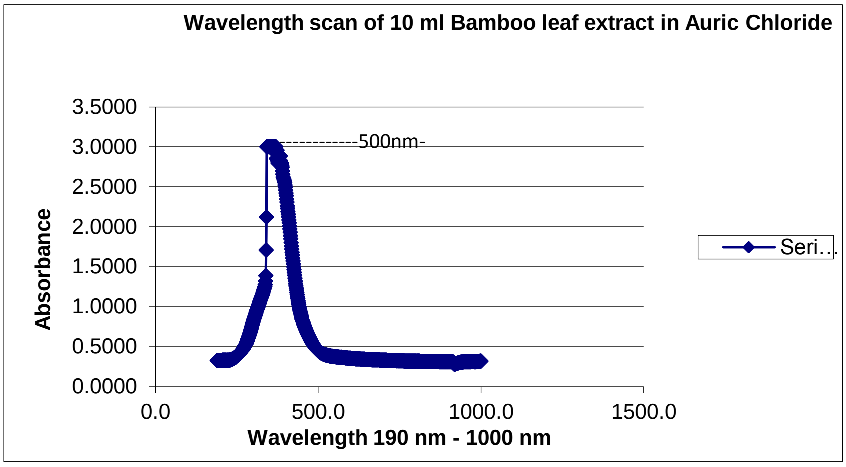

Figure 2 UV Visible absorption spectra indicating the presence of gold nanoparticle

3.2 Characterization of Membrane

3.2.1 Membrane Filtration Studies

The water flux for PVDF membrane was carried out under the trans membrane pressure of 500 KPa. The flow rate is calculated as per the above formula and the flux showed that the flow rate of nanoparticles coated membrane was found to be low when compared to Neat membrane. The Table 1 and Figure 3 representation explain the above interpretation. The concentration polarization effect due to the accumulation of the culture in the near of membrane surface appeared to be the dominant factor controlling the flux of the membranes. Resistance-in series model was applied to analyze resistances that lead to flux decline during ultrafiltration process. Fouling is governed by cake layer on the membrane surface. Thus, the adhesion force of foulants against the surface of membranes is an important parameter that allows direct assessment of culture adsorption behavior at the interface. The stronger the adhesion force of the membrane-foulants, the more severe flux decline in permeation process. This suggests that the adhesion force of membrane-foulants could be used to predict the flux decline rate and extent of membrane fouling in the permeation process. Figure 4 shows the graphical representation of the fouling activity of PVDF, PVDF/ AgNPs and

PVDF/AuNPs incorporated membranes. The above said points are interpreted in the form of Table 1 and it arrives to the point that the flux rate directly affects the fouling factor of membranes. To a membrane to have low fouling rate, the flux rate recorded is of higher value. From the table, the flux rate of PVDF membrane is higher, pertaining the membrane to be easily prone to foul.

Table 1 Water flux, contact angle, fouling, membrane resistance of membranes

Sample name	Pure water flux (l/m^2h)	Fouling (%)	Contact Angle (°)	Membrane Resistance ($kPa\ l^{-1}.m^2.h$)
PVDF	46.4	25.4	68	18.1
PVDF-Gold	45.4	6.08	72	9.3
PVDF-Silver	40.8	10.1	78	8.5

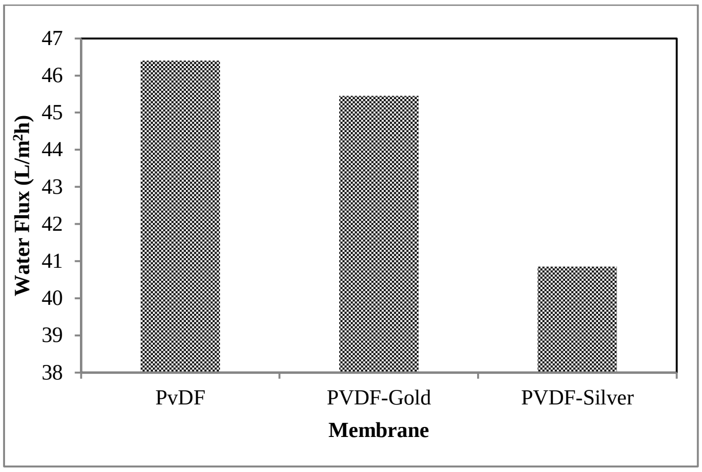

Figure 3 Water flux of Neat PVDF, PVDF/Gold and PVDF/ Silver membranes

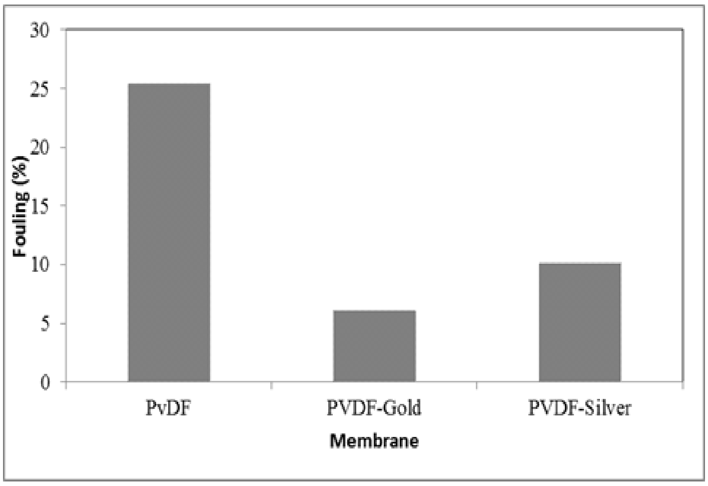

Figure 4 Fouling activity of PVDF, PVDF/Gold and PVDF/ Silver membranes

3.2.2 Morphological Analysis of Membrane

Figure 5 and Figure 6 shows the SEM images of the top view and surface area of the gold PVDF and silver PVDF membranes. It also indicates the characterized channel of the silver/ PVDF and gold/ PVDF composite membrane. It can be observed from that silver and gold nanoparticles exists in the PVDF membrane and the dispersion of silver is more uniform. In addition, many filamentary connections are formed between silver and PVDF polymers. The phenomena can improve the compatibility between silver nanoparticles and PVDF Polymer chains, which enhances the integrative properties of the membranes.

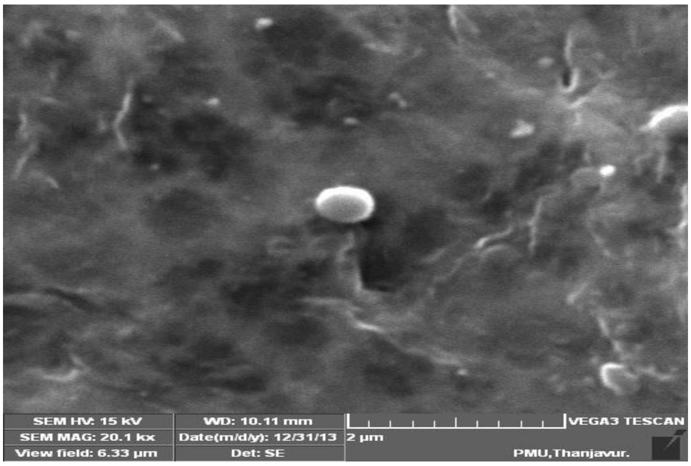

Figure 5 SEM image of silver nanoparticle incorporated membrane

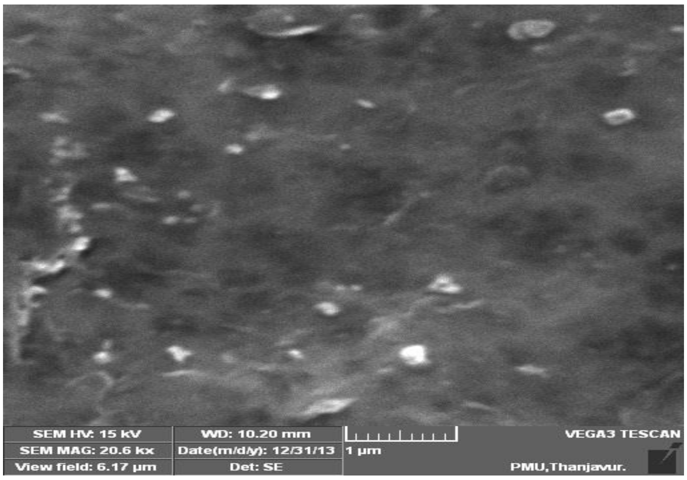

Figure 6 SEM image of gold nanoparticle incorporated membrane

3.2.3 XRD

Figure 7 shows the XRD patterns of neat PVDF, gold PVDF and silver PVDF membranes. According to Figure 7, Bragg reflections at 2θ values around 39.35º, 63.78º and 64.18º correspond to (111), (200), (220) sets of lattice planes of vibration which may be indexed for the face centered cubic structures of silver nanoparticles. Also, the intensity of (111) diffraction peak is much stronger than any other peaks. In case of silver incorporated PVDF membrane, in addition of the dispersion peak of base polymer, the peak at 41.12º, 49.86º and 67.57º had risen from silver with a slight shift as compared to the prepared gold nanoparticle. The analysis is revealed that the crystalline silver nanoparticles have an effective interaction with the PVDF matrix.

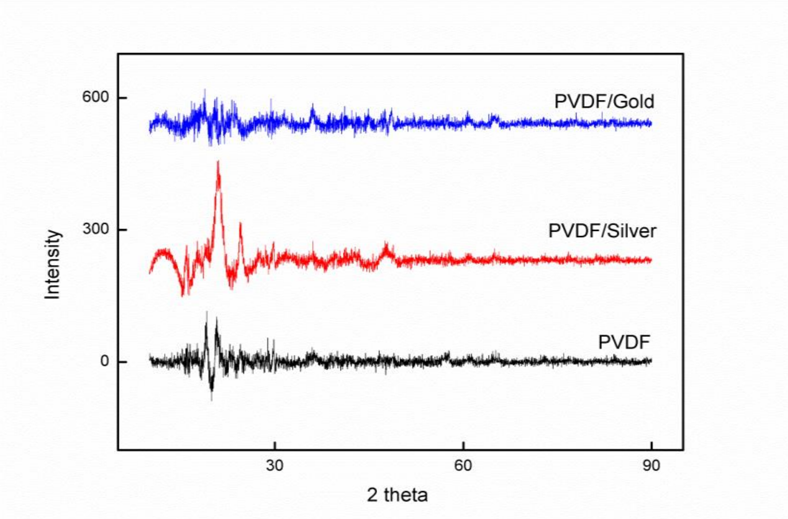

Figure 7 XRD analysis of PVDF, PVDF/Gold and PVDF/ Silver membranes

3.2.4 FTIR

Figure 8(a), 8(b) and 8(c) shows that the FTIR spectra of PVDF membrane, silver and gold nanoparticles composite membranes before and after fouling. In Figure 8(a) and 8(b) the intensity of OH stretching peak and bending peak at 3420 cm^{-1} and 1660 cm^{-1} are enhanced, indicating that more OH bonds are formed by adding silver and gold nanoparticles to PVDF membranes. It can also be observed, that the intensity of C-F stretching peak and bending peak exists at 1150 cm^{-1}, the peaks at 1380 cm^{-1} are attributed to the stretching vibrations of CH, but the peaks of gold PVDF and silver PVDF composite membranes at 1380 cm^{-1} are stronger than the PVDF membrane. This result is caused by hydrogen bonds formed between OH groups and C-F further indicating the compatibility between silver and PVDF through H bond which ensures silver composite membranes posses better capabilities even after fouling occurred.

3.2.5 Anti-Microbial Activity of Membrane

The antimicrobial activity of silver and gold nanoparticles coated membrane was evaluated using disc diffusion. According to several studies on the microbicidal properties of silver and gold nanoparticles [27], they interact with the cell membrane, penetrate into the cell, act with the DNA materials, and thus inhibiting the cell to adhere on the membrane. The results shown in Table 2 and Figure 9 indicated that the silver coated membrane had strong inhibition towards *E. coli* and similarly Table 3 and Figure 10 indicated gold coated membrane also showed significant inhibition towards *Staphylococcus aureus* and *E. coli.* Therefore, the results indicated that silver and gold nano particles coated membrane appeared to be an effective resistant towards the growth of micro organisms.

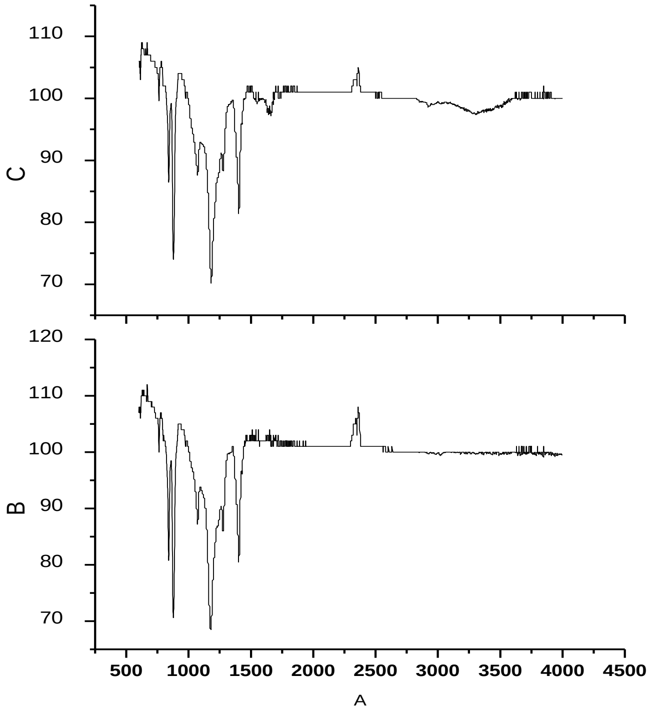

Figure 8 (a) FTIR Spectra of neat PVDF membrane before and after fouling

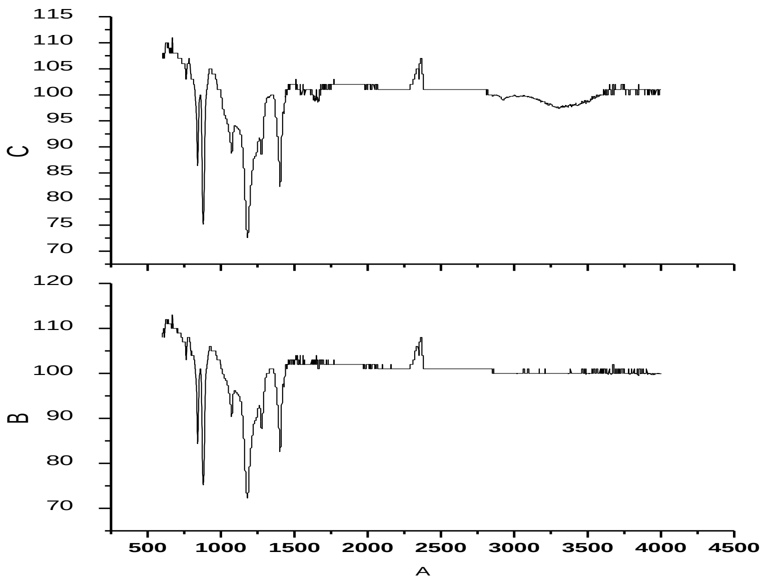

Figure 8 (b) FTIR Spectra of PVDF/AgNPs incorporated membrane before and after fouling

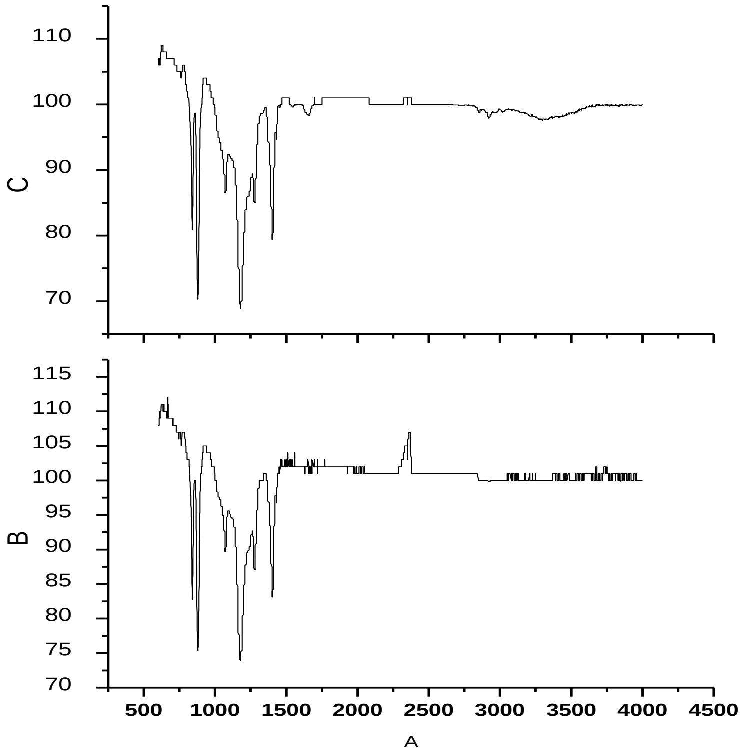

Figure 8 (c) FTIR Spectra of PVDF/AuNPs incorporated membrane before and after fouling

4.0 CONCLUSION

The PVDF UF membranes containing silver and gold nanoparticles have been prepared by phase inversion method. The silver and gold nanoparticles were uniformly distributed in PVDF matrix as observed by SEM and the diameter of silver and gold nanoparticles was 5-15 nm. During water and waste water treatment, these UF membranes would be useful for biofouling prevention. Fouling cannot be eradicated completely, but reducing fouling of membranes will make membrane separation process easier to maintain, more economical and more sustainable. Experimental results have shown the feasibility of blend membranes of PVDF enables the ultra-filtration of culture. The performance of the synthesized nanoparticles impregnated to the membranes was investigated and the results revealed that enhanced performance of PVDF can be obtained by the change in PVDF, PVDF/AuNPs and PVDF/AgNPs ratio in casting solutions. Characterization analysis of modified PVDF membranes has given adequate results pertaining to surface properties of nanoparticles impregnated membranes. Membrane UF employing such newly tailored membranes helped to achieve desired permeability without much affecting the rejection performance. Also, it can be concluded that the biologically derived AgNPs are proven to be effective additives for polymeric membrane preparation that can be used for industrial purposes. Membrane fouling could reduce the permeation efficiency and restrict the wide application of ultra-filtration membrane.

Table 2 Antimicrobial activity of pathogenic organisms against silver coated membrane

Organisms	Zone of inhibition (Diameter in mm)
Pseudomonas aeroginosa	8
Staphylococcus aureus	7
Escherichia coli	9
Bacillus subtilus	7

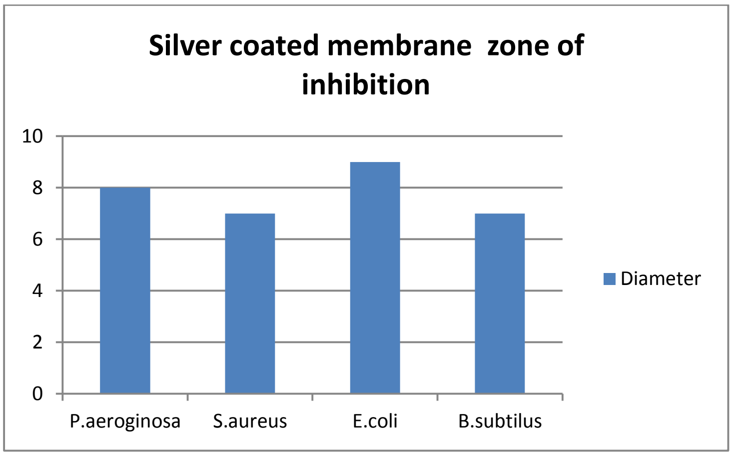

Figure 9 Zone of inhibition of pathogenic organisms against silver coated membrane

Table 3 Antimicrobial activity of pathogenic organisms against gold coated membrane

Organisms	Zone of inhibition (Diameter in mm)
Pseudomonas aeroginosa	7
Staphylococcus aureus	8
Escherichia coli	8
Bacillus subtilus	7

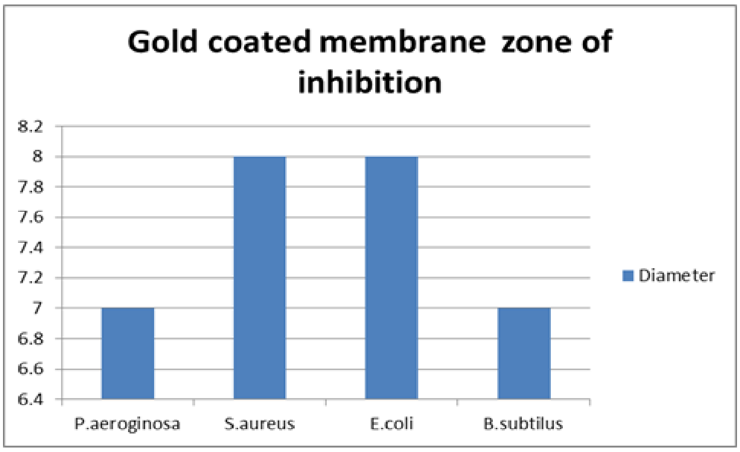

Figure 10 Zone of inhibition of pathogenic organisms against gold coated membrane

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