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The role of TiO₂ nanoparticles to enhance membrane antifouling properties: A short review

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ABSTRACT

The incorporation of nanoparticles (NPs) to membrane has developed the membrane separation to overcome fouling and to enhance antibacterial properties of membrane. To date, Titanium Oxide (TiO₂) NPs are utilized in various industrial applications and recently the development in membrane technology, owing to their superb antifouling, antibacterial, thermal and mechanical stability. This study aims to evaluate the TiO₂ NPs roles to enhance the membrane quality in terms of antifouling, antibacterial properties and the membrane performance. The contribution of this study is to bring the researcher's attention for the TiO₂ NPs importance to modify the membrane characteristics to overcome the fouling issues.

1. Introduction

Nanoparticles (NPs) have potential to be used in various fields such as food recovery by membranes [1], in pharmaceutical to enhance the surface charge of miRNA [2]. In recent years, membrane separation advances have been achieved by introducing the nano-materials in membranes. There are variety of nano-materials such as nanoparticles (NPs), nanotubes, nano-sheets and nano-spheres, which serve as excellent nano-fillers within the membrane matrices. Hence, the combination of membrane and nanotechnology could lead to new breakthrough in the membrane development industry due to its versatile and attractive advantages to enhance the membranes physical properties and overall performances. NPs are essentially particles that exist in crystal or powder form which span the range between 1 nm to 100 nm. NPs are the most fundamental component in the formation of a nanostructure and they yield a high surface-to-volume ratio. NPs have gained intense attention in recent years, owing to their unique properties in terms of photoemission, antimicrobial activity, optical properties and catalytic activity. All of these essential NPs characteristics enhance their potential application in various fields such as desalination, biomedical, communications, electronics and other industries [3]. To

date, the establishment of NPs-embedded membranes is attaining tremendous interest due to its ability to enhance properties of membranes. The metallic NPs would normally yield different characteristics from bulk metals, in which the nano-size particulates would exhibit greater physical and chemical properties due to the enhanced surface area. There are a number of studies reporting the applications of NPs in membrane applications such as water and wastewater treatment. Numerous types of metal or metal oxide NPs have been reportedly used in membrane applications, such as Silver (Ag), Iron Oxide (Fe₂O₃, Fe₃O₄), Silica (SiO₂), Aluminum Oxide (Al₂O₃), Titanium Dioxide (TiO₂), Magnesium Oxide (MgO), and Zirconium Dioxide (ZrO₂) [4]. Some of these metal oxide NPs are quite expensive and thus efforts have been focused to find a low cost metal oxide. One of the popular low-cost metal oxide is titanium oxide (TiO₂), which has been used as a new antimicrobial agent.

2. Additives to Enhance Membrane Antifouling Properties

Many researchers have been investigated the effect of different types of organic and inorganic additives on membrane performance and morphology of polymer blend membranes. The role of organic and inorganic additives is to create a spongy like membrane structure

by interruption of macrovoid formation, enhance pore formation, improve pore interconnectivity and/or introduce Hydrophilicity.

Nanoparticles (NPs) have received much attention recently due to their unique properties in terms of antifouling, antimicrobial properties and mechanical strength improvement. The contribution of NPs in the membrane technology has gained considerable attention in the recent decades for various purposes. Incorporation of various types of NPs in membrane fabrication is a new trend in producing excellent membrane quality in terms of permeability, rejection capability and also fouling propensity in addition to excellent physical properties such as mechanical and thermal stabilities. To date, the NPs which are commonly reported in membrane fabrication are TiO₂, SiO₂, Silver, Polyaniline (PANI), and Zinc Oxide (ZnO).

The permeation study of xylitol mixture was carried out at 4 bar for 1 h to evaluate the membrane separation. The results show the increasing flux on the modified membrane where the solution flux of pure PES membrane was observed at 4 L/m².h, while PES/TiO₂ membrane's flux increased to 7 L/m².h [5].

2.1. Titanium Dioxide (TiO₂) Nanoparticles

TiO₂ is a special nano-materials with excellent photocatalytic effect and hydrophilic properties. In recent years, TiO₂ had received high attention in membrane applications. A brief review on the membrane preparation and characterization with the incorporation of TiO₂ was performed. Early development of PES/TiO₂ composite membranes was investigated with the employment of Rutile TiO₂ (30 nm) in PES membranes was able to enhance the membrane fouling resistance, hydrophilicity, mechanical and thermal properties [6]. There was a study reporting the PVA/PES thin film nano-composites membranes deposited with TiO₂ also exhibited significant membrane performance in terms of water permeability, NaCl rejection and swelling degree [7]. Membrane hydrophilicity enhancement after addition of TiO₂ nanoparticles have resulted in water flux improvement [8]. Furthermore, when 1% – 2 % of TiO₂ is incorporated with the pure membrane, the membrane porosity is increased, while when the ratio is increased to 3 %, the agglomeration of NPs occurred [9]. Generally, the incorporation of NPs into the membrane influences the membrane characteristics significantly. The trade-off between permeability and selectivity of polymeric membranes could be solved by incorporating NPs. The presence of NPs has been found to alter the characteristics of the membranes' top layer, pore size, thickness, hydrophilicity and charge potential, and parameters related to the membrane structure such as the porosity and macrovoid morphology of the asymmetric support [10]. Hence, the selection of TiO₂ NPs in this research was based on the advantages listed as following:

- i. Cheap in cost
- ii. Non-toxic and bio-safe material
- iii. Antifouling properties
- iv. TiO₂ has a good antibacterial properties

TiO₂ NPs were anchored on the PA-TFC RO membrane surface to enhance the antifouling properties by the way of self-assembly through the interaction between the positively charged colloidal TiO₂ and the carboxylic

groups on the membrane surface [11, 12]. TiO₂ nanoparticles distributed on the membrane surface can re-lease the strong oxidative species, such as Hydroxyl radicals (OH•), super-oxides (O₂•-), and hydroperoxyl radicals (HO₂•-), under the UV light irradiation, which can effectively kill most microorganisms, inhibiting the formation of biofilms and subsequently enhancing the antibiofouling properties of the membrane. However, this strategy needs the additional facility, such as the UV light irradiator, which will increase the cost inevitably. Thus, to improve the antifouling properties of the membrane by combining it with TiO₂ nanoparticles has seen comparatively little re- search in recent years.

3. Antifouling Factors affecting TiO₂ Membranes

Fouling mitigation has attained tremendous attention and concern in the development of membrane applications to improve the membrane process durability, efficiency and reliability [13]. Recent studies show that the incorporation of nano-TiO₂ is a vital approach to reduce the fouling susceptibility of membranes.

Therefore, the potential of TiO₂ NPs in the membrane antifouling mechanisms could be explained based on several factors as listed below:-

i. Hydrophilicity

The enhancement of membrane antifouling performance might be attributed to the hydrophilic nature of nano-TiO₂ which could increase the membrane surface hydrophilicity [14]. This is an encouraging phenomenon due to the higher affinity of membrane towards water molecules which helps in preventing the foulants deposition [15].

ii. Surface roughness

Generally, the membrane surface roughness correlates well with fouling tendency. It was observed that membranes with smoother surface are less prone to fouling due to the reduced foulants adsorption or deposition on the valleys or peaks presented on the membrane surface [16].

iii. Pore size

Membrane with smaller pore size would reduce the penetration of foulants solutes into membrane matrices. This is essential due to the prevention of pore blockage with less foulants accumulation onto the membrane surface, which indirectly promotes the membrane fouling resistance [17].

iv. Adhesion forces

The investigation on foulant-membrane and foulant-foulant adhesion forces is important in fouling mitigation. The employment of hydrophilic TiO₂ would weaken the van der Waals forces and electrostatic interaction of the foulants, resulting in higher fouling resistance [18].

4. Membrane Surface Modification

Despite the advance in membrane development nowadays, there are still some limitations to achieve greater membrane characteristics. Common challenges or issues are the production of membrane with enhanced antifouling properties and simultaneously high permeability rate as well as good physical and chemical properties. A variety of membrane modifications methods are introduced for this purpose, including blending of nano-materials [19]. The enhancement of

membrane structural properties could be done by performing modifications such as the employment of additives like organic materials which includes hydrophilic or amphiphilic polymers and inorganic nanomaterials [20]. The drawbacks in the application of membranes including fouling, scaling and bio-fouling which can cause high maintenance cost, low membrane selectivity. These issues can be avoided by surface modification of the pure membranes; there are various approaches to modify the membrane surface such as grafting, coating and blending [21]. The modification procedures allow finding a compromise between hydrophobicity and hydrophilicity, localize the hydrophilic material specifically in the membrane pores, where they have a positive effect on flux and fouling reduction [22]. A variety of researches has been performed to overcome the fouling issues. Several membrane modification methods have been practiced widely to improve the membrane performance, i.e. development of composite membranes via blending [23, 24], interfacial polymerization [25], UV-initiated grafting [26], or plasma treatment [27]. Among these techniques, blending of inorganic hydrophilic nanoparticles or hydrophilic copolymer into the polymer has attracted the most considerable attention due to its ability to directly improve the hydrophilicity and water permeability [28] as well as controlling the tradeoff between the selectivity and flux of the membrane. Therefore, the membrane surface modification by blending is reviewed briefly in this section including the blending methods and the phase inversion technique to ensure more progressive membrane production with excellent performances.

4.1. Blending Methods

Blending is the simplest and most widely used method to modify the pure membranes both for flat-sheet and hollow fiber membranes, as illustrated in Fig 1.

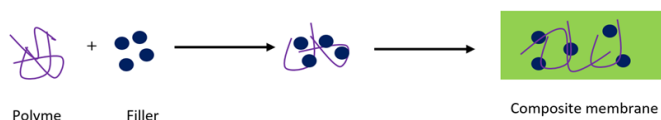


Figure 1. Blending method for membrane fabrication.

Blending is a pre-fabrication method that can be employed to modify membrane properties. It is one of the most important membrane modification methods as it can easily alter membrane structures and surface properties. Blending has been successfully adopted for membranes using hydrophilic polymers, such as cellulose diacetate, polyaniline (PANI) nanofibers and sulfonated poly (ether ether ketone). Blending of these polymers can improve membrane behaviors like membrane permeability, flux decline and retention capability [29, 30].

Incorporating nanomaterials in a blending solution is another effective way that has been adopted to minimize bio-fouling problems by integrating nanoparticles such as TiO₂, ZnO, ZrO₂ and biogenic silver into membrane matrices [4]. This method is reportedly effective in enhancing membrane properties, such as reduce the fouling tendency, increase hydrophilicity, and increase water flux and solute rejection [31-33]. Blending such

materials directly into the membrane solution, however, induces several challenges with agglomeration, nanoparticle leaching from the membrane matrices and complications with direct mixing when high mixing ratios of 10-30% are involved [34].

5. Membrane Characterization

Membrane characterization can be categorized into three categories: physical, chemical and thermal. These examinations will help to understand the relationship between the membrane structure and its separation behavior. Several considerations such as simplicity, availability, accuracy and equipment consistency should be considered for better analyses and characterizations. The effects of the additives (NPs) on the membrane structure, stability and morphology have to be characterized in various aspects such as performance testing (water flux and rejection), membrane morphology (FESEM, EDX and AFM), physical properties (contact angle, thermal stability and mechanical strength) and finally membrane fouling resistances.

5.1. Contact Angle

The contact angle value of the membrane shows the material's hydrophilicity. As the hydrophobicity increases, the contact angle of the droplets with the surface increases. Surfaces with contact angles greater than 90° are labeled as hydrophobic [35]. The contact angle is measured to investigate the pure membrane and the TiO₂ modified membrane's hydrophilicity. Both membranes were dried for 72 h prior to measurement. The Rame-Hart Model 200 standard contact angle goniometer (Fig 2) was used with DROP image Standard Software with an accuracy of 60.10° is one possible instrument that may use for this purpose. The medium used to measure the contact angle was deionized water and air at ambient temperature (27°C).

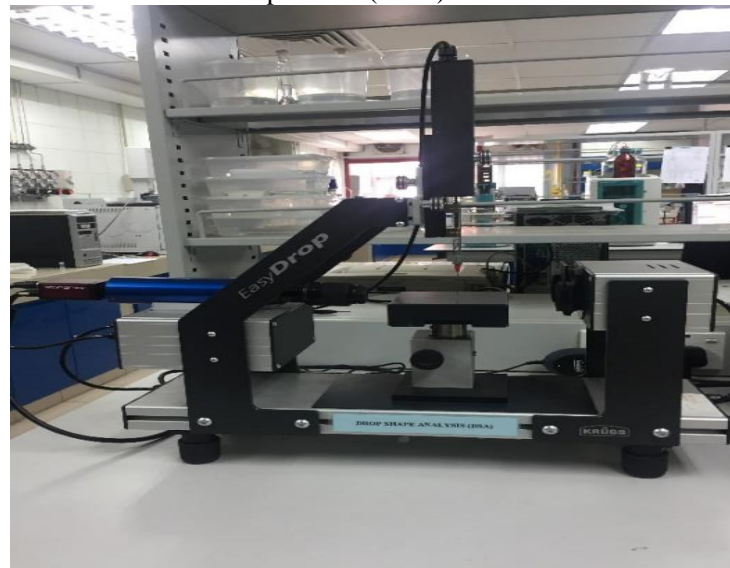


Figure 2. Contact Angle device.

5.2. Energy-Dispersive X-ray Spectroscopy (EDX)

The line scan of the spectrum of energy dispersion of X-ray (EDX) has been used to investigate the nanoparticles presence on the surface of the synthesized membrane.

5.3. Atomic Force Microscopy (AFM)

Surface morphology of original and modified membranes is investigated to obtain the membranes surface roughness by surface image analysis. The membrane roughness can be obtained from the root mean square roughness (RMS). The membrane roughness is proportional to membrane pore size; changes in roughness can be due to the changes in pore size [36, 37]. The higher fouling rate was attributed to surface roughness, the high surface roughness leads to high membrane fouling [38]. The advantage of this technique is that no pretreatment is required and the measurement can be carried out under atmospheric conditions. A disadvantage is that high surface roughness may result in the images which are difficult to be interpreted. Moreover, high forces may damage the polymeric structure.

5.4. Membrane Performance (Water Flux – Rejection)

The pure water flux and solution rejection of the pure and PES/TiO₂ membranes are carried out by a dead-end filtration in a pressure filtration unit as illustrated in Fig 3. Initially, both membranes are compacted at pressure higher than operating pressure until steady water flux is achieved (more than 1h) using DI water prior to filtration experiments. The pure water flux (PWF) was measured by applying Equation (1).

$$J_w = \frac{V}{A \cdot t} \quad (1)$$

where J_w is the water flux (L/m².h), V is the permeate volume (L), A is the membrane area (0.00146 m²), t is the filtration time (h).

While, the rejection (R) was calculated by using Equation (2):

$$R (\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (2)$$

where C_p and C_f are concentrations of permeate and feed respectively.

Also, the fouling process can be analysed by determining several equations in order to describe the membrane antifouling property (R_t), which defined as the degree of total flux loss caused by total fouling as stated in Eq(3). High R_t value indicates to a large drop in flux.

$$R_t = 1 - \frac{J_p}{J_w} \quad (3)$$

where R_t is the antifouling property, J_p and J_w are the permeate flux and water flux respectively.

Reversible fouling (R_r) is the degree of the reversible flux loss caused by reversible fouling while irreversible fouling (R_{ir}) is the degree of irreversible flux loss caused by irreversible fouling. R_r caused by mixture solution adsorption or desorption on the membrane surface can be washed by hydraulic cleaning but the R_{ir} is the other way round and require extensive hydraulic cleaning. To distinguish between R_r and R_{ir} , these two values were calculated using Eq(4) and (5), respectively.

$$R_r = \frac{(J_{w2} - J_p)}{J_{w1}} \quad (4)$$

where J_{w1} , J_p and J_{w2} are initial water flux, the permeate flux and the final water flux respectively.

$$R_{ir} = \frac{(J_{w1} - J_{w2})}{J_{w1}} \quad (5)$$

Next, the flux recovery ratio (FRR) was evaluated in order to evaluate the antifouling property (R_t) of the membranes and their efficiencies after refreshing by DI water. The water flux recovery was defined from Eq(6):

$$\text{Flux recovery ratio} = \frac{J_{w2}}{J_{w1}} \times 100 \quad (6)$$

The higher FRR value obtained, the better antifouling ability and higher washing efficiency are possessed by the membrane.



Figure 3. The dead-end permeation cell.

5.5. Field Emission Scanning Electron Microscopy (FESEM)

FESEM was used to examine the cross-sectional structures of the fabricated membranes. Liquid nitrogen is used to fracture the membranes and all the membranes are coated with platinum before scanning. FESEM is a very convenient and simple method for characterizing and investigating the porous structure and substructure of microfiltration and other asymmetrical membranes, respectively. A clear and concise picture of the membrane can be obtained in terms of top layer, cross-section and bottom layer. In addition, the porosity, pore size distribution and geometry of the pores can be estimated from the photographs. A narrow beam of electrons with kinetic energies in the order of 1-25 kV hits the membrane sample. The incident electrons are called primary (high-energy) electrons, and those reflected are called secondary (low-energy) electrons. Secondary (low-energy) electrons mainly determine the imaging. Burning of membrane can be avoid by coating the sample with a conducting layer, often a thin gold layer, to prevent charging up the surface.

5.6. Fourier Transform Infrared (FTIR)

FTIR spectrometer within the range of 4000-500 cm⁻¹ is used in order to obtain the infrared vibrational absorption spectrum of the membrane material. Interpretation of the spectrum allows functional groups of the membrane

material to be identified and the determination of the membrane polymer using reference spectra.

5.7. Membrane Porosity

The porosity of the pure/blended membranes is identified by their water retention capacity.

The porosity (ϵ) of the membranes is measured after wiping away the excessive water with filter papers prior to drying the membranes in a vacuum oven at 60 °C for 6 h before weighing it.

The porosity of the pure/blended membranes was identified by their water retention capacity, which was measured by a gravimetric technique using Equation 7:

$$\epsilon = \frac{W_1 - W_2}{(\rho_w)(A)(l)} \quad (7)$$

where W_1 and W_2 are the weights of the wet and dry membranes; ρ_w is the water density (998 g/m³); A is the effective area of the membrane (0.00146 m²), and l is the membrane thickness (0.0002 m).

The mean pore radius (r_m) of the membranes was determined on the basis of the pure water flux and porosity values. The mean pore radius (r_m) was measured using the GuerouteElford-Ferry Equation (refer to (Feng et al., 2004)) as given in Equation 8:

$$r_m = \sqrt{\frac{(2.9 - 1.75\epsilon) \times 8\eta l Q}{\epsilon \cdot A \cdot \Delta P}} \quad (8)$$

where η is water viscosity (8.9 x 10⁻⁴ Pa s); l is the membrane thickness (0.0002m); ΔP is the operation pressure (0.4 MPa).

5.8. Inductively Coupled Plasma Mass Spectrometer (ICP-MS)

The major obstacle that facing NPs stability on and inside the membrane matrix is the NPs leaching out during filtration. The ICP-MS is used to investigate the release of NPs from the membrane during filtration process.

6. Conclusion

For membrane applications where low cost would be highly desirable, the nanoparticles are the ideal choice for membrane fouling mitigation. Thus, it is worthwhile to investigate the performance of polymeric membrane with TiO₂ NPs as one way of modification. This paper discussed the incorporation of TiO₂ NPs with the polymeric membranes which were fabricated by other researchers and their performances were tested and reported.

As discussed, the TiO₂ NPs are suitable to be used in membrane separation processes. However, the performance of pure polymeric materials as membranes may not be ideal for industrial use because of fouling. Thus, membrane modification by adding nanoparticles to the polymeric materials is reported to have more advantages over pure polymeric materials alone in most cases. Polymeric membranes modified by adding TiO₂ NPs might increase in permeability, lowering the fouling, higher tensile strength, higher selectivity of certain components, better performance in wider temperature and pH range, and higher diffusion rate.

The most challenges that face the NPs incorporation with membrane were the agglomeration and leaching out of the NPs during phase inversion and filtration. Despite these challenges, nanoparticles have been showed great

promise for the development of membrane antifouling system.

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