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# Cellulose Acetate – Based Mixed Matrix Membrane for Methylene Blue Wastewater Treatment

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**Abstract.** The industrial advancement in the textile industry has caused negative environmental impacts because of the large amount of dye wastewater effluent. The treatment of dye wastewater can be conducted using adsorption, biological treatment, and flocculation-coagulation processes. However, all of these methods involve chemical additions, which may increase the operational cost of the wastewater treatment process. To overcome this problem, membrane separation technique has been widely used to treat textile dye wastewater. Cellulose acetate-based membranes in particular are attractive because they have a relatively high percentage for dye rejection. However, the flux still needs to be improved as the volumetric flow rate from textile industries is very high. In recent years, the incorporation of inorganic fillers inside polymer matrix inside mixed matrix membranes (MMMs) attracts many researchers to investigate their potential for gas and water treatment. This research studied the synthesis of cellulose acetate-based MMMs to treat methylene blue, a cationic dye commonly found in textile wastewater. ZIF-8 and TiO<sub>2</sub> were investigated as inorganic fillers for the MMMs. From the experiments, it was found that ZIF-8 exhibited better dispersion and improved dye rejection than TiO<sub>2</sub> particles in the cellulose matrix. However, hydrophilic TiO<sub>2</sub>-containing MMMs could produce flux as high as 260 LMH, which was higher than pure polymeric membranes and ZIF-8-based MMMs.

**Keywords:** Cellulose acetate, ZIF-8, TiO<sub>2</sub>, methylene blue, membrane

## INTRODUCTION

Industrial activities drive the economic development in every country including Indonesia. Such industries that provide a great contribution to Indonesian development and economy are textile and fashion industries. In addition to their contribution, textile and fashion industries also create water and wastewater problems in their process production. Dye wastewater is considered as the largest challenge for these industries. Dye wastewater consists of organic dye molecules, such as methylene blue, that might potentially harm the environment. [1]. Hence, the treatment of methylene blue is very important to protect the nature. One of the potential methods is by using polymeric membrane due to its simplicity and compact nature. However, polymeric membranes used for wastewater treatment experience fouling phenomenon thus lower their product output [2]. Fouling is a complex phenomenon; hence, many researchers try to obtain novel membrane materials that can reduce the fouling problem in membranes. In recent years, mixed matrix membranes (MMMs) attract attention to be used in water and wastewater treatment [3,4]. This is because MMMs combine inorganic filler and polymeric material that can exploit the advantages of

each material [5]. In addition, the presence of fillers can alter the polymer matrix, hence change the fouling phenomenon experienced by the membranes. MMMs have been commonly used for gas separation processes. However, there are still limited numbers of MMMs applied for wastewater treatment compared to MMMs for gas separation application [5].

In a study conducted by Sitter et al. (2012), titanium dioxide particles were incorporated into polyether sulfone matrix to produce MMMs for wastewater treatment. It was concluded that the inclusion of inorganic fillers could enhance the ability of the membrane to reduce the fouling propensity of the membranes [6]. Titanium dioxide particles were also incorporated inside polyvinylidene fluoride or PVDF polymer matrix and the membranes were applied for methylene blue separation from pure water. An increase in permeate flux and a decrease in membrane rejection efficiency were observed after particle insertion. This was mainly attributed to the hydrophilic nature and pure inorganic nature of titanium dioxide particles that create incompatibility with the organic nature of polymer matrix [7]. Hence, in recent years, researchers are conducting research to find suitable materials to be combined with polymer matrix that can produce MMMs with high permeate flux and rejection efficiency. Metal Organic Frameworks (MOFs) particles have been considered as a suitable material for the synthesis of MMMs due to their nature having the organic-inorganic combination of materials [8,9]. The organic part of MOFs is believed as an advantageous effect that reduces the incompatibility issue with the polymer matrix. There are hundreds of MOFs that can be synthesized in the laboratory scale, hence it is very important to find suitable MOFs for MMMs fabrication. Among hundreds of MOFs available until recently, Zeolitic Imidazolate Framework (ZIF) particles are few MOFs that are already available commercially [10–12]. ZIF particles are relatively easy to synthesize and are available in a wide range of pore sizes. One particular ZIF that has been combined inside MMMs is ZIF-8. ZIF-8 has a pore size around 0.34 nm and several studies have reported that the real pore size of ZIF-8 can be larger than 0.34 nm [13]. ZIF-8 is very easy to synthesize at room temperature [14]. However, the application of ZIF-8 as MMMs for water or wastewater treatment is still limited as in a recent study ZIF-8 was reported as not quite stable inside aqueous environments [15]. The application of ZIF-8 based MMMs is mainly in gas separation applications. ZIF-8 has been incorporated inside copolymer Pebax-1657 [16,17], polysulfone and cellulose acetate [18,19]. All studies indicated an improvement in gas permeance through MMMs with a slight reduction in gas selectivity. In addition, ZIF-8 showed a relatively better dispersion than other pure inorganic particles. Hence, it is very interesting to study the incorporation of ZIF-8 inside polymer matrix for MMMs used in wastewater treatment.

This research combined two different types of material, i.e. Titanium Dioxide and Zeolitic Imidazolate Framework -8 (ZIF-8), with Cellulose Acetate (CA) polymer to be synthesized as a new MMMs for dye wastewater treatment. ZIF-8 is a class of Metal Organic Frameworks (MOFs) that has organic component, which will improve the ZIF's dispersibility inside the polymer matrix. Better or good dispersibility of filler inside the polymer matrix will ensure better separation performances through MMMs. In this research, MMMs were synthesized using a combination of dispersion-phase inversion techniques where the ZIF-8 and CA were mixed in different proportions and were tested for their flux and rejection during methylene blue filtration. The synthesized MMMs were characterized for their morphology and separation performances using synthetic methylene blue solution. For comparison, Titanium dioxide-based MMMs will be also synthesized in this research.

## **MATERIALS AND METHODS**

### **Materials**

Cellulose acetate (CA) used for polymer matrix for MMMs was kindly provided by Sigma Aldrich. Formamide and acetone were kindly provided by Sigma Aldrich. These two chemicals were applied as the solvents for cellulose acetate. Zinc nitrate hexahydrate and 2-methylimidazole for the ZIF-8 particle synthesis were supplied by Sigma-Aldrich. For comparison, as a pure inorganic filler, Titanium dioxide from Sigma Aldrich was used for MMMs synthesis. Methylene blue as a typical wastewater component was provided by Sigma Aldrich. All materials were applied with no further purification.

## Methods

### The fabrication of ZIF-8

Room temperature synthesis using water as the solvent technique for ZIF-8 was adopted from [20]. In a typical procedure, 2-methylimidazole solution was synthesized by mixing 3.7 g of particles with 32.5 ml of pure water. The solution was combined with 32.5 ml of zinc nitrate hexahydrate solution that contained 0.18 g of particles. The solution was then mixed for one hour. The product was treated using a bench-scale centrifuge for around 10 minutes at 10,000 rpm. The particles that were collected at the bottom of the centrifuge tube were dried overnight inside an oven at 85 °C.

### The fabrication of mixed matrix membranes

Phase inversion process was adopted in pure polymeric membrane and MMMs synthesis process. In a typical procedure, cellulose acetate powder was dissolved in acetone as solvent. Then, the addition of formamide was conducted. For MMMs synthesis, inorganic filler (either ZIF-8 or TiO<sub>2</sub>) was incorporated into the polymer solution. The particle concentration was varied from 0, 3, 5, to 10 wt.% based on the weight of polymer. The suspension was stirred for 24 hours. Then, the solution or suspension was casted on a flat glass. The film was then occurred followed by solvent evaporation. Then the film was soaked in a water bath for 24 hours and the solid film started to form. The membranes were then dried and kept wet before analysis and permeation test.

### Dye wastewater preparation

Methylene blue, as a synthetic challenge solution, was synthesized by dissolving 0.025 g of methylene blue powder into 500 ml of pure water for 50 ppm concentration and dissolving 0.1 g of solids into 500 mL of water for 200 ppm concentration. The 200-ppm sample was used to investigate the rejection behavior at high wastewater concentration.

### Analysis and characterization of pristine polymeric membrane and MMMs

Membrane characterization was conducted to study the morphology of produced membranes using scanning electron microscopy (SEM). SEM was conducted. The surface of the flat membrane was coated with Pd/Au before being analyzed under Evo MA 10 Carl Zeiss SEM.

### Liquid permeation test

The liquid permeation test was performed using a dead-end filtration mode. Before the permeation test, the thickness of the membrane was determined using a micrometer. The effective membrane area for filtration was 31.2 cm<sup>2</sup>. As a preliminary step, the compaction test was done by using 150 ml of water that passed through the membrane. The pressure for performance and compaction test was maintained at 101 kPa by compressed air. The liquid permeation test determined water flux, wastewater flux, and rejection percentage. Each sample run used 150 ml of sample. Permeate was collected and measured to determine the flux ( $J$ ) with the time interval of 5 min using Eq. (1) below:

$$J = \frac{V}{A \times t} \quad (1)$$

where  $V$  is the volume of permeate (L),  $A$  is the area of membrane (m<sup>2</sup>), and  $t$  is the time (hour).

The rejection efficiency of the membranes during methylene blue separation was analyzed using UV-VIS Spectrophotometer. Samples were collected and analyzed using a wave number of 640 nm. Meanwhile, the rejection percentage (% $R$ ) was calculated according to Eq. (2) below:

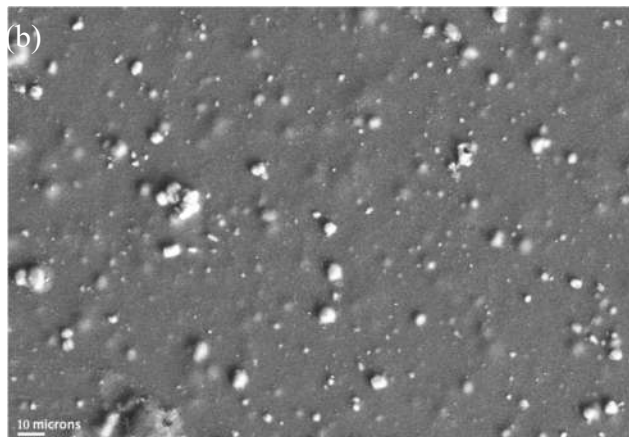
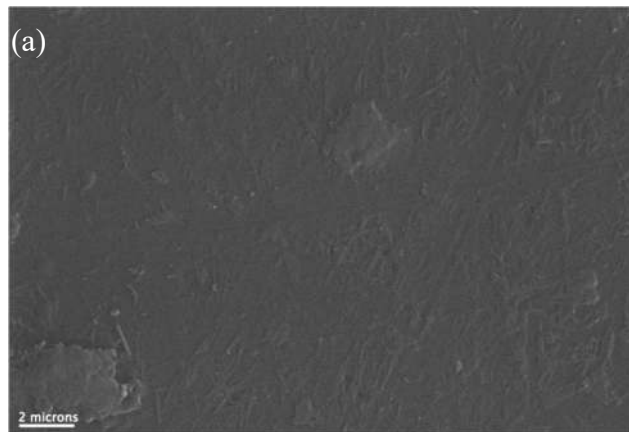
$$\%R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (2)$$

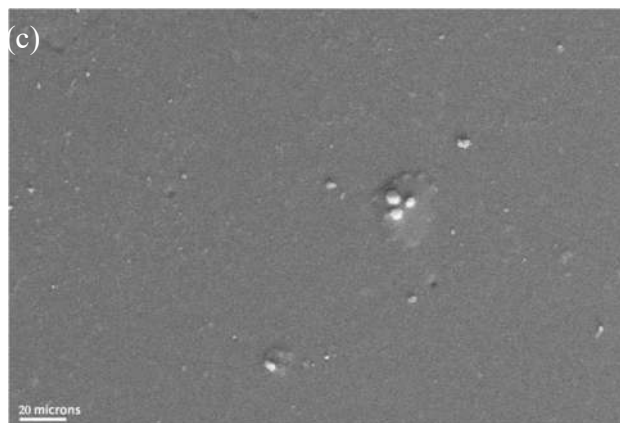
where  $C_p$  and  $C_f$  are the concentration of permeate (ppm) and feed respectively. In this work, each run was taking minimal 3 times of samples.

## RESULTS AND DISCUSSION

### The morphology of pristine polymeric membrane and MMMs

The morphology of produced membranes in this study was studied by analyzing membrane surface using SEM. For SEM analysis, three membranes, i.e. pristine cellulose acetate membrane (CA), membrane with 5 wt.% of  $\text{TiO}_2$ , and membrane with 5 wt.% of ZIF-8, were analyzed.





**FIGURE 1.** SEM images of the membrane surfaces (a) pure cellulose acetate membrane; (b) TiO<sub>2</sub>-Cellulose acetate based MMMs; and (c) ZIF-8-Cellulose acetate based MMMs

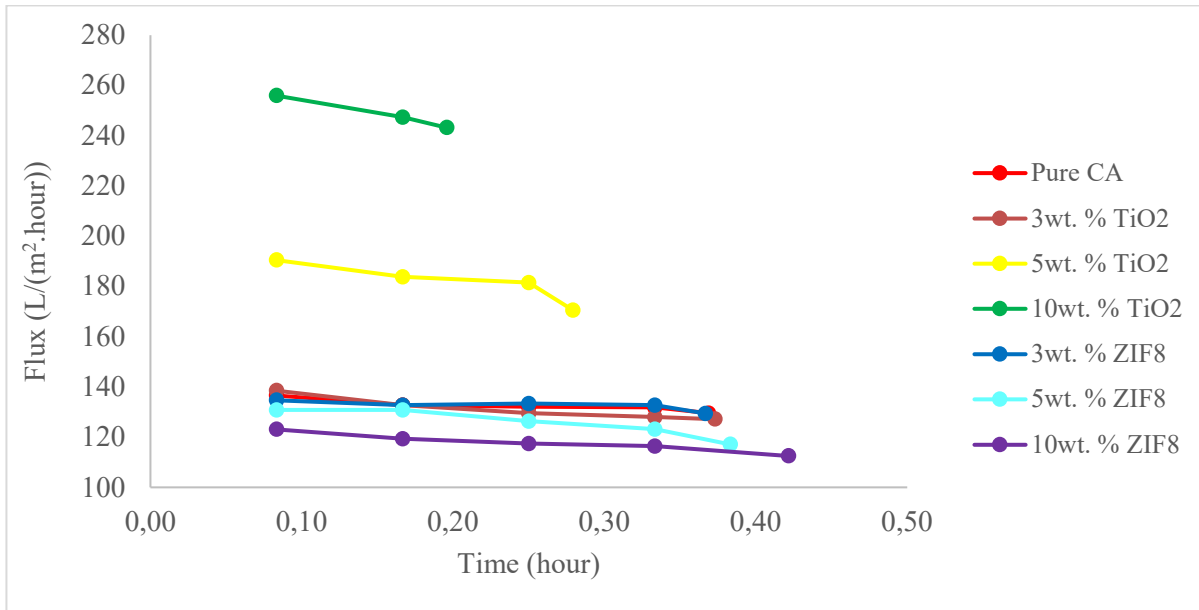
Fig. 1 depicts the SEM analysis results of the surface of three different membranes. Pure cellulose acetate membranes in Fig. 1a showed a relatively smooth surface indicating the presence of no particles inside the polymer matrix. The surface of the membrane was relatively smooth due to the absence of particles inside the membrane. Different observation was observed with TiO<sub>2</sub>-based membrane, where the presence of particles inside the polymer matrix was clearly observed in Fig. 1b. The position of the particles was quite dispersed, however, there were several agglomerates observed inside the polymer matrix. The tendency of the particles to form agglomerates will affect the formation of defect-free membranes. In general, the voids and defects inside or on the surface of the membranes will decrease the rejection capacity of the membrane and will also increase the permeate flux. Further investigations on this issue will be resented in the next section on the permeation test of the membranes.

In contrast to TiO<sub>2</sub>-based MMMs, ZIF-8-based membranes showed a relatively smoother surface than TiO<sub>2</sub>-based membranes as depicted in Fig. 1c. The presence of inorganic part in ZIF-8 structure helps to increase the compatibility between the particles and polymer matrix. This will bring an advantage to reduce the possibility of defects formation. However, from Fig. 1c, it is also detected a small agglomerate on the membrane surface indicating a tendency of the ZIF-8 particles to agglomerates, even though the agglomerates were not as severe as in TiO<sub>2</sub>-based membranes.

### **Methylene blue separation performances of pure polymeric membrane and MMMs**

Permeability and membrane selectivity had been examined in this work using the dead-end filtration cell. Flux and rejection data were investigated from 150 ml of samples with 50 ppm methylene blue concentration.

### Flux vs time profile

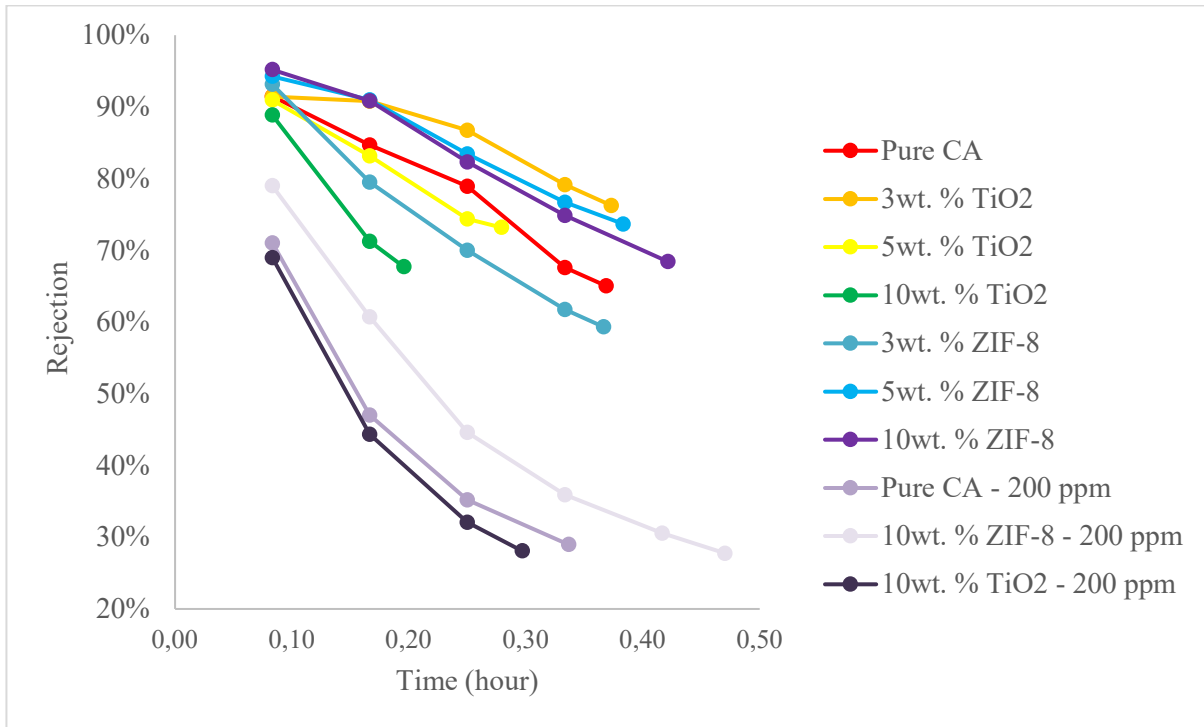


**FIGURE 2.** Flux vs time profile of all membranes

Flux vs time profile behavior of all membranes is shown in Fig. 2. The fluxes were observed every 5 minutes. From this figure, all membrane fluxes decreased with filtration time. The shortest profile was obtained for CA-TiO<sub>2</sub> with 10wt. % of particles membrane, whereas CA-ZIF-8 with 10wt. % of particle membrane was the longest. These behaviors corresponded to the hydrophilicity and hydrophobicity properties of the membrane after the addition of fillers. The hydrophilicity of TiO<sub>2</sub> particles facilitated the permeate to pass the membrane easily. In addition, high concentration of TiO<sub>2</sub> particles in the membrane might cause the formation of many interfacial voids. These voids are improving the fluxes. In contrast, ZIF-8-based MMMs with 10 wt. % of ZIF-8 showed hydrophobicity property. Hence, the permeate needs more time to pass the membrane. In addition, the possibility of the polymer matrix rigidification after particle incorporation might also affects the separation performances of the MMMs. The rigidified polymer layer around ZIF-8 particles might improve membrane selectivity. This rigidification allows the reduction of pore size, therefore the flux of membrane decreased.

Flux vs time profile of pure cellulose acetate membrane is also identified from Fig. 2. The flux was lower than CA-TiO<sub>2</sub> membranes of 5 and 10 wt. % particle concentrations, and also higher than CA-ZIF-8 membranes of 5 and 10wt. % particle concentrations. However, the flux was nearly the same as CA-TiO<sub>2</sub> and CA-ZIF-8 with 3wt. % particle concentration. This is an indication that the addition of 3wt. % of both inorganic particles did not affect the flux of the membrane. At the larger concentration, compared to the ZIF-8 particles, TiO<sub>2</sub> particles increased the flux of the membrane. Meanwhile, the addition of ZIF-8 particles decreased the flux of the membrane.

### Rejection vs time profile



**FIGURE 3.** Rejection vs time profile of all membranes

Rejection profiles of all membranes tested in this research are shown in Fig. 3. The reduction in the percentage of rejection of methylene blue with time were observed for all membranes. This phenomenon indicates the ability of membranes to separate a dye solution were decreased. The reduced rejections were attributed to the concentration polarization phenomenon on the membrane surface and the decrease in active surface of particles for the adsorption of methylene blue. Concentration polarization phenomenon can be initiated by the increase of solute concentration near the membrane surface. Then, the concentration build-up generates a diffusive flow back to the bulk of the feed. Therefore, the concentration of permeate also increases, and rejection decreases. In addition, the mechanism of separation inside MMMs is also determined by the adsorption capacity of the filler, hence the decrease of rejection efficiency of MMMs might be affected by the decrease in active surface available for methylene blue adsorption through the pores of fillers.

In order to ensure the rejection profile at higher concentration, methylene blue with 200 ppm concentration had been tested on the membranes. From Fig. 3, the results show, the rejections at high concentration had the same trend to 50 ppm concentration. Therefore, this confirms that fouling of membrane contributes to reduced rejection. Another factor that causes the reduced rejection was the dimension of dye molecules. Methylene blue molecule has very small size, that is  $17 \text{ \AA} \times 7.6 \text{ \AA} \times 3.3 \text{ \AA}$ . The size is smaller than  $\text{TiO}_2$  particles that ranging from 10-40 nm, therefore it enables the dye molecules to pass through pores and particles in membrane. Conversely, the ZIF-8 particle has smaller size than dye molecule's dimension that is 3-12  $\text{ \AA}$ . However, the dye molecules were still able to pass the membrane. This is occurred because of the ZIF-8 particles undergo the gate-opening effect. This is the state of particle in which pore size opens and gets bigger, consequently, the pore size is bigger than the dye molecule.



### Correlation between flux and particle concentrations

**TABLE 1.** Membrane fluxes of various particle concentrations

| Membrane                    | Particle concentrations (%wt.) | Flux (J) (L/m <sup>2</sup> .hr) |
|-----------------------------|--------------------------------|---------------------------------|
| Pure cellulose acetate (CA) | 0                              | 136.604                         |
|                             | 3                              | 138.528                         |
| CA + TiO <sub>2</sub>       | 5                              | 190.476                         |
|                             | 10                             | 255.892                         |
|                             | 3                              | 134.68                          |
| CA + ZIF-8                  | 5                              | 130.832                         |
|                             | 10                             | 123.136                         |

The impacts of particle concentration with respect to cellulose acetate membrane are shown in Table 1. The flux of pristine cellulose acetate membrane was lower than CA-TiO<sub>2</sub> membranes and higher than CA-ZIF-8 membranes. Higher flux of CA-TiO<sub>2</sub> membrane was caused by TiO<sub>2</sub> particles, consequently, created incompatibility between TiO<sub>2</sub> and the polymer. These different properties of particles cause void formation on the membrane. Therefore, the voids yield empty space for the permeate to pass the membrane easily. In addition, TiO<sub>2</sub> particles have hydrophilic properties which attract water, consequently, the flux increases as the water pass the membrane easily. Thus, the flux increased as the TiO<sub>2</sub> concentration increased.

The incorporation of ZIF-8 particles into the cellulose acetate membrane was decreased the flux. These particles had organic linkers that could interact with the membrane polymer, therefore, the ZIF-8 particles were distributed uniformly in the membrane. This leads to the rigidification of polymer in the vicinity of particles, then, the pore size of the membrane becomes smaller. In addition, the hydrophobicity of ZIF-8 makes the water become harder to penetrate. Consequently, the flux decreased as the ZIF-8 concentration increased.

### Correlation between rejection and particle concentrations

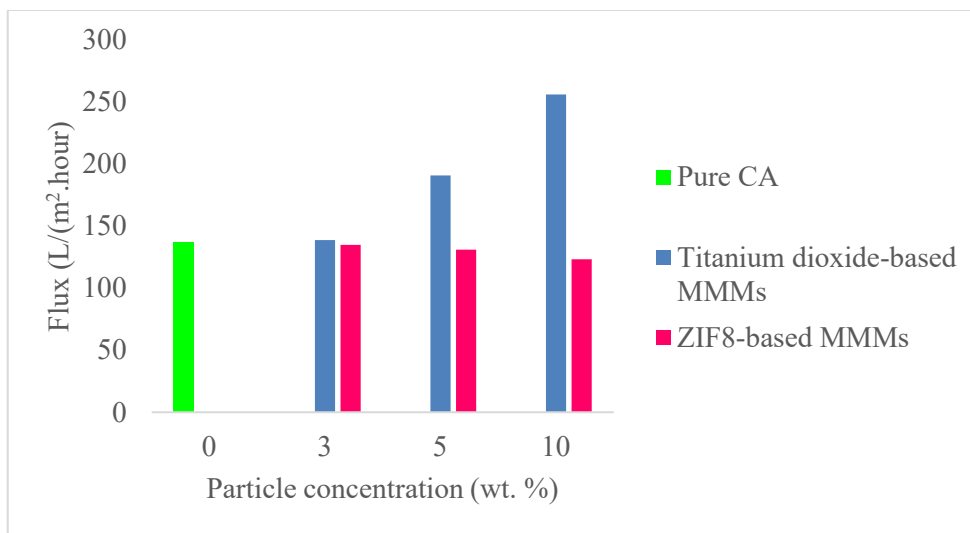
**TABLE 2.** Membrane rejection percentages of various particle concentrations

| Membrane                    | Particle concentrations (%wt.) | %R     |
|-----------------------------|--------------------------------|--------|
| Pure Cellulose Acetate (CA) | 0                              | 91.455 |
|                             | 3                              | 91.421 |
| CA + TiO <sub>2</sub>       | 5                              | 90.931 |
|                             | 10                             | 88.844 |
|                             | 3                              | 93.13  |
| CA + ZIF-8                  | 5                              | 94.231 |
|                             | 10                             | 95.176 |

The concentration of inorganic fillers also affects the rejection of the membrane. The results are presented in Table 2. According to the table, the rejection of pure cellulose acetate membrane was higher than CA-TiO<sub>2</sub> membranes but lower than CA-ZIF-8 membranes. The incompatibility of TiO<sub>2</sub> particles leads to defect formation. Consequently, the dye molecule passes the membrane easier, then, the CA-TiO<sub>2</sub> permeate concentration becomes higher than the pure polymeric membrane. This makes the rejection of the CA-TiO<sub>2</sub> membrane become lower than the polymeric membrane as the TiO<sub>2</sub> concentration increases.

In contrast, the ZIF-8 particles have organic linkers that enhance compatibility with polymer matrix. However, still, there is a possibility for the rigidification of polymer matrix after the incorporation of particles. Therefore, the rigidification of polymer in the vicinity of particle surfaces makes the pores become smaller. Thus, the membrane becomes more difficult to be passed by dye molecules, and conversely, the rejection increases.

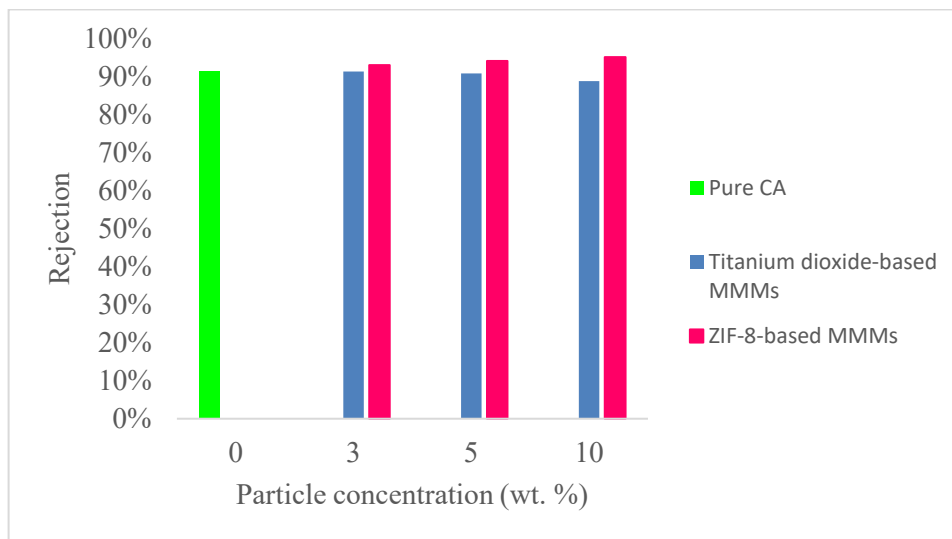
#### Correlation between flux and particles types



**FIGURE 4.** Correlation between membrane fluxes and particle types

In order to understand the correlation of particle types and flux, we correlate the flux with respect to particles types in Fig. 4. In this figure, all concentrations of CA-TiO<sub>2</sub> membranes are higher than CA-ZIF-8 membranes. The different properties of these particles, hydrophilicity, and hydrophobicity, lead to the different results of fluxes. The hydrophilic membrane is easier than the hydrophobic membrane to pass the permeate. Therefore, CA-TiO<sub>2</sub> membrane fluxes were higher than CA-ZIF-8 membrane.

### Correlation between rejection and particle types



**FIGURE 5.** Correlation between membrane rejection percentages and particle types

Lastly, the correlation between particle types and rejection are is shown in Fig. 5. Compared to ZIF-8 particles, TiO<sub>2</sub> particles' rejection were lower. As explained in the latter section, the hydrophobicity of ZIF-8 contributes to the high fluxes of ZIF-8 particles, meanwhile, the hydrophilicity of TiO<sub>2</sub> makes the fluxes low.

### CONCLUSION

Pure cellulose acetate and cellulose acetate-based MMMs have been synthesized and tested for their separation performance. The results from this study provide a relatively good understanding of the interaction between the polymer matrix and inorganic particles and the relation between the incorporation of particles with membrane performances. The addition of TiO<sub>2</sub> into the cellulose acetate matrix improved the flux of the permeate, accompanied by slightly decrease in the rejection. The fluxes of each TiO<sub>2</sub> concentration (3%, 5%, 10%), were 138.528 L/m<sup>2</sup>.hr, 190.476 L/m<sup>2</sup>.hr, and 255.892 L/m<sup>2</sup>.hr, respectively. Furthermore, the characterization of CA-TiO<sub>2</sub> membrane results indicated the formation of particle agglomerates, which defects the membrane and increases the flux. Besides, incorporation of ZIF-8 membrane into the cellulose acetate improved the rejection of permeate, meanwhile, the flux reduced as the concentration was higher. Compared to TiO<sub>2</sub> particles, the fluxes of each ZIF-8 concentration (3%, 5%, 10%) were 134.68 L/m<sup>2</sup>.hr, 130.832 L/m<sup>2</sup>.hr, and 123.136 L/m<sup>2</sup>.hr, respectively. The presence of organic linker inside ZIF-8 structure improved the ZIF-8 interaction with the polymer matrix. Hence, this study reveals the better dispersion of ZIF-8 particles compared to titania particles. Better dispersion enhanced the separation performances of the membranes.

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