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Preparation, characterization and performance evaluation of fly ash-based composite geopolymer membranes for methylene blue dye removal

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ABSTRACT

Utilization of fly ash-based geopolymer membranes for the removal of environmentally hazardous materials has become an attractive route due to its cheaper processing cost and durability. This paper reported the preparation of geopolymer composite membranes and its performance for the removal of methylene blue contaminant. The geopolymer membrane was prepared by dissolving fly ash in alkaline activator of sodium hydroxide solution. Various proportions of foaming agents made from a mixture of hydrogen peroxide and egg white was added to investigate their influence on the membrane pore structures and morphology. The morphology, pore structure and functional groups of geopolymeric composite membranes were characterized using field emission scanning electron microscopy (FESEM), Brunauer-Emmett-Teller (BET) analysis and Fourier transform infrared spectroscopy (FTIR), respectively. The performance of the membranes, which were denoted as GE0 (0% w/w of egg white), GE1 (1% w/w of egg white), GE2.5 (2.5% w/w of egg white) and GE5 (5% w/w of egg white), was evaluated for the removal of methylene blue from aqueous solution via membrane filtration system. The highest rejection of 96% and the best permeation of 15 L/m^2.h were obtained for GE5 membrane. This finding is supported by bigger pore size (19.6 nm) with better uniformity as revealed by BET and FESEM analysis.

Keywords: geopolymer membrane; methylene blue; egg white; porosity

1. INTRODUCTION

Several industries produce and utilize various types of synthetic dyes for their end products, resulting in a major environmental concern due to the release of highly colored effluents (Zaghbani et al., 2007). Not only colors, but their degradation products of the dyes are also toxic and harmful to human and living organisms, raising attention to the world on the importance of removing the dyes from the wastewater (Aluigi et al., 2014). The regions where the

water resources are vital for keeping the maintenance of the ecosystem must be protected and freed from the harmful effects of the dye effluents. Various methods have been made available to remove the pollutants from water, which, for example, biodegradation (Saravanan et al., 2013), flocculation-coagulation (Norkhairunnisa and Fariz, 2014), oxidation (Kumar and Kumar, 2011) and adsorption (Bai and Colombo, 2017; Siyal et al., 2018; Siyal et al., 2021). Recently, membrane separation techniques using polymeric materials have been introduced for dye removal, but their major disadvantages such as declining in permeate flux (Zaghbani et al., 2007) and low resistance of organic solvent, have opened the door for the inorganic membranes to be an alternative. However, inorganic membranes suffer from high fabrication costs primarily due to the expensive powder processing and need to be sintered at high temperature. Therefore, another possible way to overcome this issue is using low-cost material and mild preparation conditions of the inorganic membrane. Thus, this paper proposed the use of fly ash as the material for the geopolymer membrane as it is environmentally friendly material and is available abundantly.

Geopolymers, by definition, are chains of networks of mineral molecules linked with covalent bonds and have basic characteristics such as non-crystalline (amorphous) networks. The synthesis of a geopolymer involves raw material and an alkaline activator. Previous studies reported that geopolymer has a faster dissolution and gelation, and also cure rapidly as it can gain high strength of geopolymer as early as 24 h (Wang et al., 2017). The excellent properties such as high strength, alkali resistance, easy synthesis and free-sintering of the geopolymer make it suitable for membrane filtration application (Alanazi et al., 2017). Other than aforementioned properties, fly ash-based geopolymer is known to be porous in its pristine form. Although being porous, speficic sizes and types of pores play important roles in determining the success of a membrane filtration (Wang and Lu, 2020). Unfortunately, geopolymer's pores are low and small in sizes. A series of studies have been done on geopolymer cements (Li et al., 2019; Ortega et al., 2017) and geopolymer concretes (Nath and Sarker, 2013), but only few studies have been done on porous geopolymers.

Studies to enhance pores in geopolymer have been done by a few methods such as by heat treatment (Cilla et al., 2014), addition of sodium silicate (Alghamdi and Neithalath, 2018), varying alkaline activator concentration (Rocha et al., 2018), addition of pore foaming agents (Bai and Colombo, 2017; Kaliappan et al., 2019) and many more. However, only little information can be obtained on how to produce higher porosity geopolymer with homogeneous distribution. Combining pore forming agents in 2-stage geopolymerization could further improve pore sizes and its distribution, but porosimetry data from literature review is still lacking. Based on a study reported by Bai and Colombo (2017), the combination of peroxide route with organic additives were able to produce a total of 80% porosity. Therefore, the aim of this research was to produce a geopolymer membrane by combining two pore foaming agents that could enhance pore sizes and homogeneity of geopolymer matrices. Pores enhancement is important because previous studies on porous geopolymer reported that no interconnecting pores present on the cell walls, thereby limiting the permeability of the liquids or gases of the component (Cilla et al., 2014).

2. MATERIALS AND METHODS

2.1 Materials

Methylene blue (MB) powder was purchased from R&M Chemicals, Subang, Malaysia. Fly ash was collected from a coal-fired power plant located at the suburb of Manjung, Perak, Malaysia. Analytical grade sodium hydroxide pellets (NaOH; Merck, Sigma-Aldrich (M), Petaling Jaya, Malaysia), hydrogen peroxide (H_2O_2, R&M Chemicals, Subang, Malaysia; 30% concentration), and distilled water was used to prepare an alkaline solution. Egg white was used as a pore-forming agent in the preparation of geopolymer composite material.

The chosen fly ash raw material was first characterized using X-ray fluorescence (XRF, Bruker, S8 Tiger series, Bruker (Asia) Subang Jaya, Malaysia), and determined to be class F type fly ash due to its high silica (SiO_2) and alumina (Al_2O_3) contents as shown in Table 1. The high silica and alumina contents are a prime prerequisite for the development of geopolymeric material. The chemical compositions present in the fly ash that led to the determination of class F fly ash are tabulated as follows.

Table 1. Chemical composition of class F fly ash, Jana Manjung power plant, Malaysia

Compound	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	C	K_2O	SO_3	Others
Composition (% w/w)	44.52	22.48	11.58	9.84	3.92	2.54	1.73	1.35	2.05

2.2 Preparation of composite geopolymer

The schematic diagram for geopolymer synthesis is shown in Figure 1. The standard formulation of a geopolymer paste was prepared at the ratio of 3:1 of fly ash to the alkaline sodium hydroxide solution activator at room temperature. The ratio of fly ash to the alkaline activator, as well as the sodium hydroxide concentration (10 M), were maintained throughout the experiments. The formulation procedure was initiated by mixing sodium hydroxide solution with the fly ash, forming a mixed slurry solution, and the slurry solution was then stirred mechanically for 9 min using an overhead mixer (IKA RW20, Sigma-Aldrich (M), Petaling Jaya, Malaysia) with the speed of 600 rpm to achieve a uniform mixture.

Egg white as a pore-forming agent was added to the mixture at various percentage weights of 0, 1.0, 2.5, and 5% w/w based on the total weight of the slurry followed by the addition of 0.6% w/w of hydrogen peroxide. The composite slurry mixture was then mixed at 1,000 rpm for about 10 min until it forms a geopolymer paste (Bai and Colombo, 2017). The geopolymer paste was poured into a mold (5 cm diameter and 2 mm thickness) for the preparation and fabrication of geopolymeric membrane. The cast paste was then cured at 60°C for 24 h. Finally, the cured samples were demolded, characterized, and tested for their performance. The parameters used for the development of the composite geopolymer membrane are shown in Table 2.

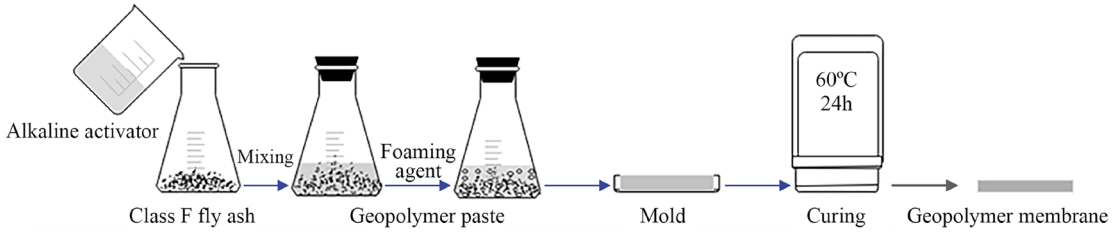

Figure 1. Schematic diagram for the geopolymer membrane sample preparation

Table 2. Geopolymer composite membranes preparation parameters

Samples	Egg white (% w/w)	Hydrogen peroxide (% w/w)	Sodium Hydroxide Concentration (M)	Si/Al Ratio
GE0	0	0.6	10	3:1
GE1	1.0	0.6	10	3:1
GE2.5	2.5	0.6	10	3:1
GE5	5.0	0.6	10	3:1

2.3 Characterization

2.3.1 FESEM analysis

The morphological properties of starting materials and geopolymer composite membranes were analyzed using field-emission scanning electron microscope (FESEM, model: Zeiss Supra 55 VP, Carl Zeiss Sdn Bhd, Petaling Jaya, Malaysia). FESEM was preferred because of its higher resolution, brightness and small sized probe size. Before analysis, sample was mounted to the aluminium stub with a carbon tape and the sample was then analyzed using secondary electron imaging technique at different magnifications (Hamidi et al., 2016).

2.3.2 BET analysis

The pore diameter distribution was measured by Brunauer-Emmett-Teller (BET) method using the surface area analyzer and porosimetry system (BET-Micromeritics ASAP 2020, G.A.T. Scientific Sdn Bhd, Petaling Jaya, Malaysia). Prior to analysis, samples were placed in a tube and subjected to degassing in a vacuum chamber at 100°C for 10 hours. The measurement of its specific surface area is performed by using BET method on N_2 adsorption-desorption isotherm (Ge et al., 2015).

2.3.3 FTIR analysis

The composite geopolymeric membranes were characterized using Fourier Transform Infra-red analysis (Spectrum one, FTIR, Perkin Elmer (M), Petaling Jaya, Malaysia) to identify the new chemical compounds introduced in the membrane matrix. The geopolymer samples were prepared in a pellet form using standard KBr pellet. The sample for analysis was ground and casted into pellet. Then, the sample was examined at a scanning rate of 24 scans/sample and a frequency range of 4000-400cm^{-1} (Rasouli et al., 2015).

2.4 Performance evaluation

The prepared composite geopolymer membrane performance was evaluated for the removal of methylene blue from aqueous solution with initial concentrations of 300, 400 and 500 ppm.

The methylene blue rejection, flux and membrane backflushing were analyzed using a fabricated membrane filtration device as shown in Figure 2. The removal performance was conducted using a Shimadzu UV-1800 spectrophotometer (Shimadzu Malaysia Sdn Bhd, Petaling Jaya, Malaysia) (Zaghbani et al., 2007).

The filtration efficiency of dye removal from feed solution was evaluated through dye rejection test, which was calculated using the classical rejection coefficient (Bai and Colombo, 2017; Siyal et al., 2021):

$$\text{Removal efficiency, } R\ (\%) = \left(1 - \frac{C_P}{C_o}\right) \times 100 \quad (1)$$

where;
C_o = Initial concentration of the dye in the feed
C_p = Dye concentration in the permeate

The fabricated membranes were tested for the flux by using Equation 2 with an effective membrane area of 0.0007014 m^2.

$$\text{Flux, } J = \frac{dQ}{A.dt} \quad (2)$$

where;
J = Permeate flux (L/m^2h)
Q = Quantity of the permeation water filtered (L)
A = Effective surface area of the membrane (m^2)
t = Time interval (h)

3. RESULTS AND DISCUSSION

3.1 Characterization of fly ash-based geopolymer membrane

3.1.1 FESEM analysis

The FESEM images of the cross-sectional part of the samples GE0, GE1, GE2.5 and GE5 are shown in Figure 3. The differences of the pore structures between GE0, GE1, GE2.5 and GE5 were the resulted form the different amounts of egg white added into the geopolymer solution. Obvious pore structures could be seen through the inner part of geopolymer membranes, as shown in Figure 3c and d, which serve as good passages for methylene blue dye permeation. For sample GE0 (Figure 3a), the image shows very little existence of pores with high amount of unreacted fly ash in the sample. Meanwhile, from Figure 3b, the pores were formed and appeared to be uniform and bigger in sizes, but with some unreacted fly ash due to incomplete chemical reaction during geopolymerization process in GE1 (Hamidi et al., 2016). The morphology of sample GE2.5 was

almost similar to GE1, but with more distributed and interconnected pores.

Peroxide routes enable the expansion of gas bubbles (Bai and Colombo, 2017) during the curing process while the egg white retains the bubbles in time that the expansion will not rupture the pores formed. Meanwhile, sample GE5 showed the desired morphology with homogenous porosity, and almost all of the pores were interconnected. This showed that addition of egg white facilitated the pore formation as well as the homogeneity of pores, which is very important for membrane filtration study.

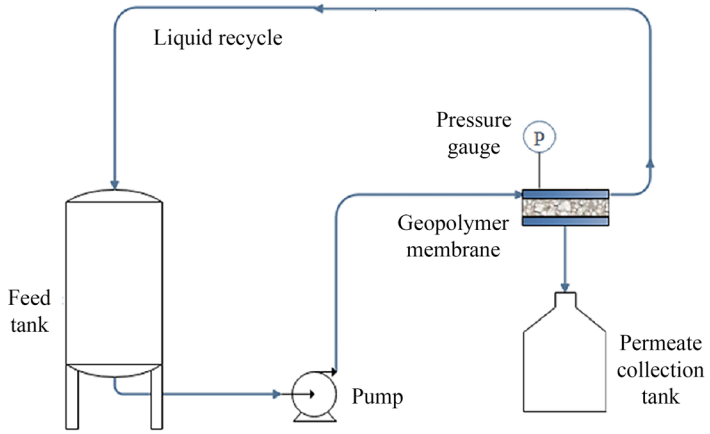

Figure 2. Schematic diagram of the membrane water filtration unit

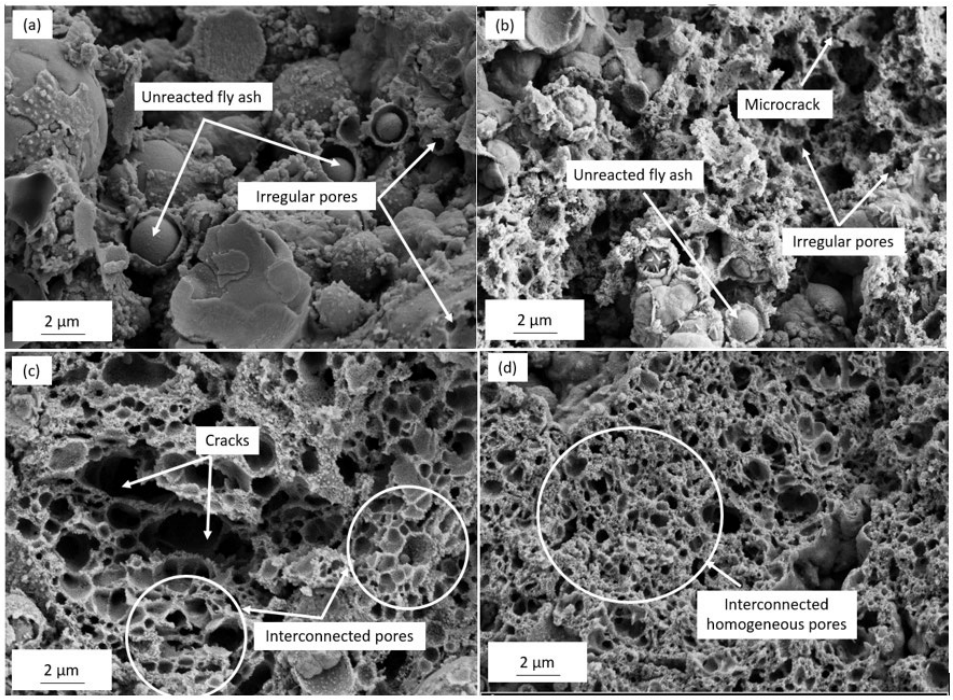

Figure 3. FESEM images of (a) GE0 (unmodified geopolymer), (b) GE1 (1% w/w of egg white, 0.6% w/w of H_2O_2), (c) GE2.5 (2.5% w/w of egg white, 0.6% w/w of H_2O_2), and (d) GE5 (5.0% w/w of egg white, 0.6% w/w of H_2O_2)

3.1.2 Pore structure analysis using BET

The effect of different amounts of egg white on pore volume distribution of geopolymer samples was described (Figure 4). The pore volume increased as the pore diameter of the samples increased. Some fluctuations were observed, due to the possible pore ruptured and inconsistent of pore distributions in the geopolymer samples (Bai and Colombo, 2017). The pore volumes for all of the samples GE0, GE1, GE2.5, and GE5 were all accumulated in 0-50 nm of the pore diameters. In sample GE0, the pore volume was smaller, compared to samples with the addition of egg white, i.e., GE1, GE2.5, and GE5. This was strongly related to the effect of high viscosity of the slurry of a geopolymer when more egg white was added. The higher amount of foam agent enables the geopolymer samples to retain the gas bubbles as it expands during the curing process (Xu et al., 2019).

The type of membrane studied was categorized as ultrafiltration membrane as the pore size was between 2 and 50 nm (Padaki et al., 2015) and made it suitable to remove methylene blue with the approximate dimensions

of the monomer molecule of 1.25 - 1.60 nm long by 0.57 - 0.84 nm wide with a thickness of about 0.5 nm (Grattan-Bellew, 2001).

3.1.3 FTIR analysis

The unmodified fly ash-based geopolymer (GE0) and modified geopolymer with the addition of egg white (GE1 and GE2.5) spectra were obtained to analyze the surface, functional groups. The FTIR results of the samples are illustrated in Figure 5. All spectra exhibited almost similar in terms of their functional groups. However, there were some shifts and additional peaks for GE1 and GE2.5, where it indicated changes of surface chemistry after modification. The bands at 3432-3441 cm^{-1} were OH groups of Si-OH and adsorbed water moleculed on the surface of fly ash-based geopolymer (Alehyen et al., 2017). The C-H stretching at 2923 cm^{-1} and 2853 cm^{-1} indicating C-H stretching, bending of H-O-H at 1645 cm^{-1}, stretching of O-C-O (carbonate groups) at 1450 cm^{-1}, Si-O-Si and Al-O-Si asymmetric stretching at 997-1075 cm^{-1} and Al-O bending vibration at 611-795 cm^{-1} were observed. The main feature of GE0 was the band at 1000 cm^{-1}, which was attributed to the Si-O-Si and Al-O-Si asymmetric stretching. This band was shifted to lower wavenumber in GE1 and GE2.5 (990 cm^{-1}), demonstrating the change in microstructure (Alehyen et al., 2017). On top of that, the shifting towards higher transmittance and appearances of peaks at 2923 cm^{-1} and 2853 cm^{-1} of GE1 and GE2.5 indicated the egg white in geopolymer. There were also peaks at 1500 cm^{-1} from samples GE1 and GE2.5, which denoted the stretching of C-N of amide bond II, representing the presence of protein in geopolymer (Mahobia et al., 2016).

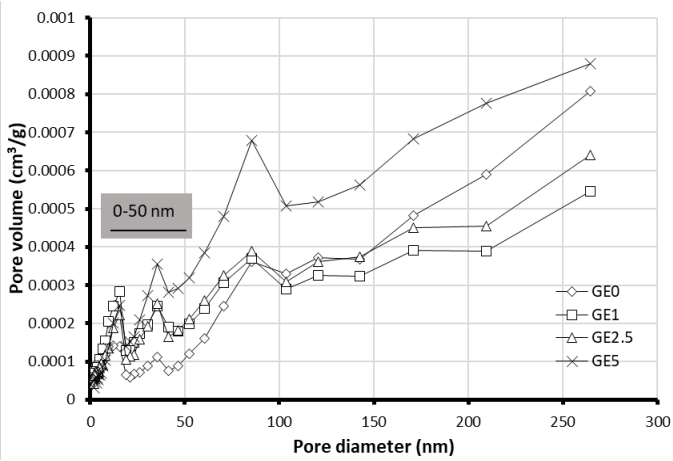

Figure 4. Pore volume distribution of the membrane samples with addition of different amounts of egg white

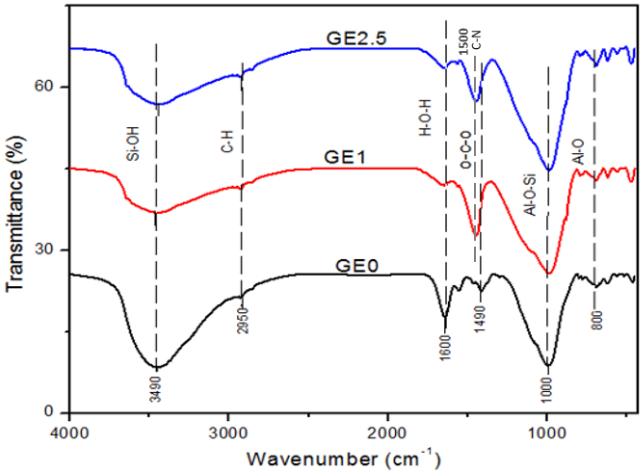

Figure 5. FTIR spectra of unmodified geopolymer (GE0) and modified geopolymer (GE1 and GE2.5)

3.2 Permeability of fly ash-based geopolymer membrane

Figure 6 shows the effect of filtration time on the water flux of the fly ash-based geopolymer membrane. The two membranes, GE2.5 and GE5, exhibited similar patterns of flux decline along with the permeation time. The porous structure of the inorganic membrane and the attenuation, along with the infiltration time are believed to be the main causes of the flux decline (Ge et al., 2015). As shown in Figure 6, the initial flux of the GE2.5 and GE5 were 9.57 $L/m^2.h$ and 15.41 $L/m^2.h$, and then the final fluxes reduced to 0.6 $L/m^2.h$ and 1.6 $L/m^2.h$ respectively. The flux values of the membrane with 5% w/w of egg white (GE5) were comparatively higher than that of 2.5% w/w of egg white (GE2.5). This is due to the larger pore sizes depicted from the membrane GE5. Higher content of egg white

subsequently led to an increase in porosity of membrane. This could be explained by the increase in foam agent. The viscosity of the slurry also increased, which affected the expansion of the gas bubbles during the curing process. Besides, the high viscosity of the slurry makes it well suited to retain the gas bubbles as it expands during the curing process (Xu et al., 2019).

Figure 7 shows the decrease of flux throughout the permeation time for 70 min. The first permeation initially showed high permeability with 11 L/m^2.h but decreased to 5 L/m^2.h after 70 minutes of separation. For the backflush investigation, the membrane was placed in the opposite direction. Water of 250 mL was used to clean it. The value of the initial flux decreased to 8.5 L/m^2.h, and the final flux decreased to 2 L/m^2.h after first backflushing (Figure 7). The second backflush exhibited a similar decline pattern, that is, initial flux decreased from 6 L/m^2.h to 0.8 L/m^2.h. The decrement pattern of the fluxes was due to the membrane fouling that restricted the continuous flow of the membrane separation process. Based on the result, the geopolymer membrane has a high potential to be reused and regenerated if the properties or methods are improved.

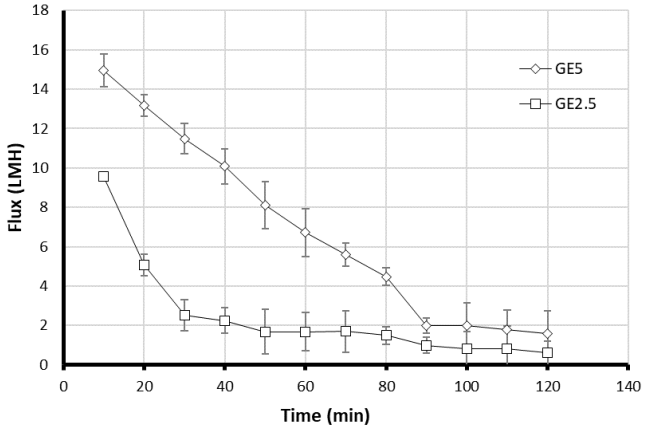

Figure 6. Flux relationship with time for membranes GE2.5 and GE5

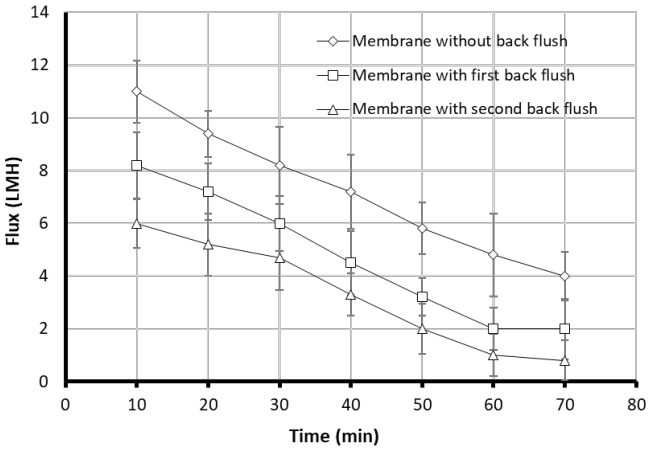

Figure 7. Flux attenuation effect during the separation process of methylene blue solution

3.3 Removal efficiency of methylene blue dye solution

The efficiency of methylene blue removal was investigated on the membrane GE5 exhibited the desired morphology with homogenous porosity, and interconnected pores. As shown in Figure 8, the removal of methylene blue was the highest (96%) when 500 ppm of methylene blue was filtered. When 400 ppm and 300 ppm of methylene blue were filtered, the removal efficiency decreased to 95% and 90%, respectively, at 70 min. This might be due to the phenomenon called concentration polarization (Sablani et al., 2001), which the solute precipitation may form, thus causes a gel layer formation that restricts more solutes to be filtered through the membrane. However, this result proved that the geopolymer membrane has successfully removed more than 90% of methylene blue, which met the requirement for discharge limit of dye effluents into the water streams.

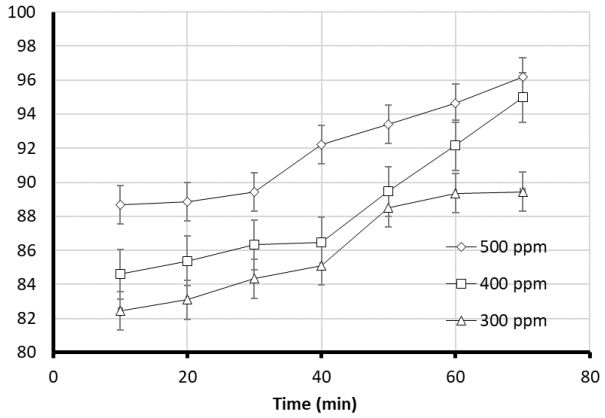

Figure 8. Effect of initial concentration of methylene blue on removal efficiency

4. CONCLUSION

The different amounts of egg white added did influence the porous structure of the geopolymer membrane. The higher the concentration of egg white, the larger the pore sizes of the membrane. As the membrane possessed larger pore sizes, the permeation of the methylene blue also increased. The membrane showed a high value of 15.41 L/m^2.h from the filtration of GE5. However, there were flux declines in which the attenuation and concentration polarization could be the main effects. This phenomenon, however, could be controlled by decreasing solute adsorption by, for example, changing the operating parameter of the membrane or chemically modifying it (Sablani et al., 2001). The removal efficiency of the geopolymer membrane above 90% complied to the discharge limit requirement. Lastly, this geopolymer membrane has proved to be a promising membrane material not only for its high flux and high removal efficiency, but also in terms of the low cost (Adesanya et al., 2018), convenience (Masindi and Gitari, 2016), and low energy consumption during preparation.

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