Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2022.28788

270 (2022) 60–69
September

Effect of particle-size distribution of fly ash on compressive strength, pore size and porosity of geopolymeric membrane for car wash water treatment

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Received 19 April 2021; Accepted 26 July 2022

ABSTRACT

Particle-size distribution plays an important role in synthesis cost and overall structural parameters of geopolymeric membrane. In this research work, the effect of particle-size distribution on the pore size, porosity, and compressive strength was investigated in the synthesis of geopolymeric membranes. The average particle-size distribution of 4.30, 23.30, 46.20, 65.32, 86.24, and 98.53 μm of fly ash was investigated separately based on pore size, porosity, and compressive strength of prepared geopolymeric membrane using sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) as a chemical activator and hydrogen peroxide (H_2O_2) (10 v/v %) as a pore-forming agent. Compressive strength of 13 MPa with 48% porosity and more uniform pore size has been obtained at source material with a particle-size distribution of 23.30 μm. No significant change in the compressive strength of the prepared membrane has been observed for source material particle sizes below 23.30 μm. The results show that there is no need for energy and cost-intensive very fine grinding below 23.30 μm as the particles were completely dissolved in an alkaline solution. A permeate flux of 40.53 L/m^2 h and 93% rejection of total solids were reported for the membrane prepared from 23.30 μm particle size.

Keywords: Geopolymerization; Particle-size distribution; Fly ash; Membrane; Compressive strength

1. Introduction

Membrane separation is a sustainable and preferred unit operation to other conventional technologies due to its being cost-effective and eco-friendly [1]. Membrane separation is cost-effective due to its minimum utility, and less energy-intensive than other conventional techniques of separation such as evaporation and distillation. Both inorganic and polymeric types of membrane materials play a vital role in the treatment of wastewater, gas separation, food, petrochemical industries, and other various

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applications [2]. Based on its applications, the inorganic membrane has great potential over the polymeric membrane due to its long-term stability, compatibility, and resistance to the harsh environment. The easy cleaning at high temperature and sterilization gives added advantage to ceramic membranes over polymeric membranes. On the other hand, the synthesis of inorganic membranes is more expensive than polymeric due to raw material powder preparation, suspension formation, calcination, consolidation, and sintering steps and therefore, cannot process such as liquid state [3]. Furthermore, the fabrication of inorganic membranes involves expensive raw materials such as mullite, titania, and zirconia at high calcination and sintering temperatures which increase the cost of membranes [4]. In ceramic membranes, compressive strength, pore size, and porosity are controlled through particle-size distribution of precursor through heating techniques which make the synthesis less economical. Therefore, much attention is paid to reducing the synthesis cost and variation of physical properties of the inorganic membrane. Siddiqui et al. [5] found that the permeability-selectivity of ultra and microfiltration membrane is dependent on the pore size and shape which is a tradeoff due to particle-size distribution of raw materials. The heat-intensive manufacturing steps and expensive raw materials for inorganic membrane synthesis were replaced by a chemical reaction using fly ash as source material through geopolymerization technique.

Geopolymer is an inorganic polymeric material; developed by Joseph Davidovits in the 1970s for construction purposes and has attracted the attention of researchers due to its low carbon footprint and low energy consumption process [2,6]. Synthesis of geopolymerization involves, alkali activation of SiO_4 and AlO_4 to yield geopolymer gel followed by hydrolysis where the bond breaking of SiO_4 and AlO_4 tetrahedral units by water molecules occurred. The geopolymer gel solidified and formed a three-dimensional porous aluminosilicate network. Abdullah et al. [7] reported that the strength of microstructure geopolymeric materials is highly dependent on the physical properties such as particle-size distribution of fly ash. Particle-size distribution is the quantification of particles based on their average diameters which can affect the reactivity of chemical reactions. Yan et al. [8] concluded that particle size of fly ashes strongly affects the pore dimensions and strength of the resulting product of ceramic materials due to fast chemical reaction which produced strong compact ceramic materials with lower porosity. Bai et al. [9] reported that geopolymerization is an innovative technique for regulating pore size and porosity through a strong bond between solid aluminosilicate oxide and a high alkaline silicate solution. As in geopolymerization, the reactions for bonding start after dissolution, therefore, the source materials particles do not significantly affect the porosity. He et al. [4] regulated the porosity and pore size by varying the hydrothermal temperature in the range of 60°C–180°C for 24 h for the use of pervaporation. Amir et al. [10] achieved porosity in the range of 66%–83% with the compressive strength between 0.3 to 9.4 MPa of geopolymeric membrane using hydrogen peroxide which evaporated during hydrothermal treatment and left a microporous surface. Recently, our group compared hydrogen peroxide and starch (10 v/v %) as foaming agents in geopolymeric paste for enhancing the porosity of geopolymeric membranes from 29% to 37.33% and 51.09%, respectively. Incomplete dissolution of source materials in the alkaline activator may result in an incomplete reaction which may cause increased tortuosity affecting the permeate flux. Synthesis of geopolymeric membrane contains leaching of alumina and silica oxides in fly ash through chemical activators, so it makes sense that a greater surface area will have a greater amount of silica and alumina oxide which will enhance the rate of geopolymerization and form better gel formation [11]. Further, the finer the particle's size distributions, the higher will be the compressive strength of the prepared membrane. The reason for this study was to investigate the physical properties of the resulting geopolymeric membrane prepared from different particle-size distribution materials because geopolymerization in a chemical reaction and dependent on the surface area of fly ashes.

Therefore, the effect of particle-size distribution on the pore size, porosity, and compressive strength has been investigated during the synthesis of geopolymeric membranes using fly ash as source material. Novelty in this work, which is a comparative analysis of all the membranes prepared from different particle-size distribution precursors, was carried out in terms of rejection and flux using wastewater from the car wash center through dead-end filtration.

2. Materials and methods

Geopolymerization is a sintering-free chemical reaction technique of inorganic membranes synthesis using waste ashes, alkaline activators, water, and pore-forming agents as raw material [12]. Geopolymeric paste was obtained by a separate mixing method of sodium hydroxide (NaOH) solutions with different particle-size distribution materials and sodium silicate solutions (Na_2SiO_3) at 120 rpm for 24 h as shown in Fig. 1. In separate mixing, the sodium hydroxide solution was first mixed with fly ash for the first 10 min and subsequent addition of sodium silicate solution to the mixture at 120 rpm for 24 h [13]. Quality parameters such as total dissolved solids, turbidity, phosphate, sulfates, settleable solids, hardness, oil, and greases were investigated as shown in Fig. 9. Wastewater from the carwash was tested for flux and rejection through dead-end filtration.

2.1. Selection of source materials

Geopolymer is the class of amorphous aluminosilicate porous materials that are manufactured from aluminosilicate raw materials such as kaolin, fume, blast slag and fly ash which contains silica and alumina more than 70% as main constituents. In the present study, fly ashes of Lakhra power plant, Sindh, Pakistan were used as source materials for membrane synthesis through geopolymerization [14]. After removing moisture content at 105°C for 2 h, the color of fly ash changed from black into white grey. X-ray fluorescence (XRF) (Model: XRF-1800, manufacturer: Shimadzu) was used to measure the composition of the source material. X-ray diffraction (XRD) (machine model: JDX3532, X-rays:

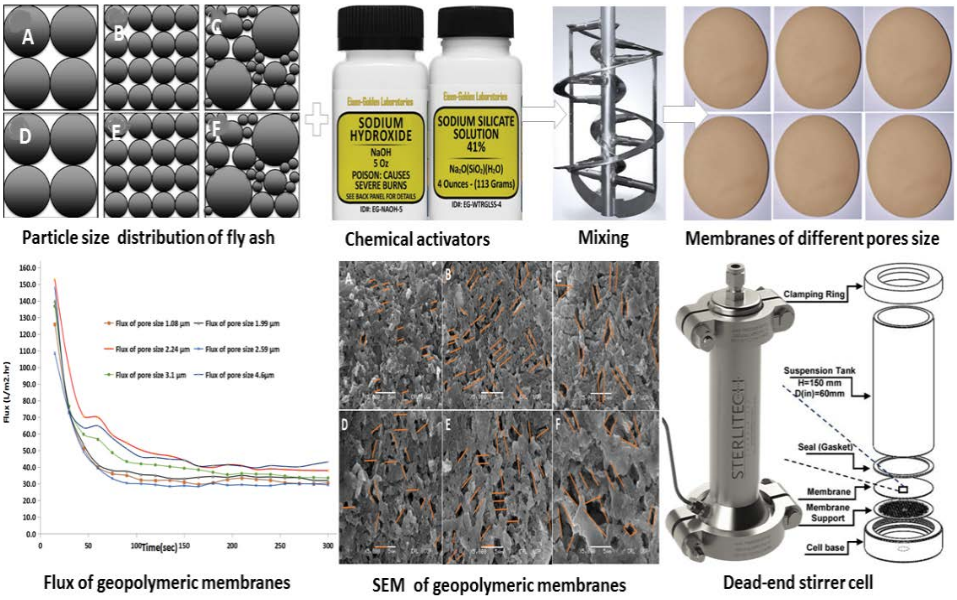


Fig. 1. Synthesis of geopolymeric membranes for various flux.

CuKa ($\lambda = 1.54$ Å)) was used for fingerprints to analyze the elements of fly ash and Fourier-transform infrared (FTIR) spectroscopy (model: FTIR-4000, manufacturer: JASCO) was used to find the functional group stretching. The ratio of silica and alumina oxides is the prime factor for selecting source material which must be in the range of 2.5–3. Many researchers have reported that fly ash has been chosen as the best source material due to its optimum silica and alumina ratio (Si/Al = 3.0), high dissolution toward chemical activators, free-calcine nature, and pulverized powder [15]. It is observed that the fly ash of the thermal power plant used in this study has a ratio of SiO_2 to Al_2O_3 of 2.8 which seems to be optimum for the geopolymer synthesis.

2.2. Chemical activators

Chemical activators are the starting binder materials for activating fly ashes. Dissolution of silica and alumina starts when fly ash is mixed into the alkaline solutions as shown in Eq. (1). The most used alkaline activators are the hydroxide and silicate of sodium or potassium. However, in this study, the ratios of chemical activators of sodium hydroxide and sodium silicate (Na_2SiO_3/NaOH = 2.5, 15 M) solutions were kept constant during the formation of geopolymeric paste. The sodium silicate solution contains 14.7% Na_2O, 29.4% SiO_2 and 55.9% water, with specific gravity of 1.53 g/cm^3 and viscosity of 400 cP at 20°C. Rattanasak and Chindaprasirt [16] recommended separate mixing of 16 M NaOH with sodium silicate in the ratio of 2.0 for 24 h. Furthermore, other researchers found that increasing the molarity of NaOH increases the compressive strength and decreases the porosity of the prepared geopolymer. Abdullah et al. [17] found that applications of resulting products are intrinsically interlinked with the particle-size distribution of fly ash.

$$\left(\mathrm{Si_2O_5}, \mathrm{Al_2O_2}\right)_n + n\,\mathrm{H_2O} \rightarrow n\,\left(\mathrm{OH}\right)_3 - \mathrm{Si} - \mathrm{O} - \mathrm{Al}\left(\mathrm{OH}\right)_3 \quad (1)$$

The proposed mechanism for the formation of hydrated aluminosilicate contains the breakdown of Si–O–Si and Al–O–Al bond; after mixing, the Al atom penetrates the Si–O–Si and forms a three-dimensional network of Si–O–Al–O along with the formation of secondary water.

2.3. Size reduction of fly ashes

The reduced size of fly ash shows a high dissolution rate and reactivity in the chemical activators due to its large surface area hence resulting in complete dissolution [18–20]. Size reduction of fly ash particles was performed through a ball mill for a total grinding time of 24 h and rotational speed of 160 rpm using different grinding media. Particle-size distribution was measured by colter-counter working on the principle of particles pulled through an orifice, concurrent with, producing a variation that is proportional to the volume of the particle traversing the orifice. The suspended fly ash particles in the control volume electrolyte create short-term changes in the impedance across the aperture during its flow and displayed a graph between volume% and particle size. The main objective of mechanical activation was to investigate the effect of particle size on pore size, porosity, and compressive strength rather than a change in mineralogical content.

2.4. Mixing of source materials and chemical activators

Geopolymeric slurry was obtained by mixing sodium silicate and sodium hydroxide solutions purchased from Nobel Chemical Limited, Pakistan with fly ashes (fly ashes/chemical activators ratio of 2.5) for 24 h at 120 rpm. The ratios of chemical activators of sodium hydroxide and sodium silicate (Na_2SiO_3/NaOH = 2.5, 15 M) solutions were kept constant during the formation of geopolymeric paste as shown in Table 1. Rattanasak and Chindaprasirt [16] reported that separate mixing produced a strong

Table 1
Weight analysis of raw materials

S. No	Mixture of geopolymer	Purity (%)	Mass (g)
1	Mass of fly ash	70.45	4,344.5
2	Mass of NaOH	91.43	599.250
3	Mass of Na_2SiO_3	82.09	1,498.10
4	Mass of water for NaOH dilution	99.92	1,000.00
5	Extra water (DI)	99.92	500.000
6	Hydrogen peroxide (v/v %)	99.99	494.375

geopolymeric material in which NaOH solution is first mixed with fly ash and then Na_2SiO_3 solution is added subsequently, while in normal mixing fly ash, NaOH and Na_2SiO_3 are mixed at the same time. Separate mixing was done and the geopolymeric paste was molded in MS ring molds. Prepared membranes were placed in a closed oven at a temperature of 60°C for curing.

2.5. Pore size, porosity and compressive strength

The compressive strength of geopolymeric membranes was tested using Universal Testing Machine (UTM model: 100–500 KN, Testometric Inc.) while the porosity was measured through water absorption technique. All the prepared samples of different particle sizes were placed in a universal testing machine and subjected to a specific load until the sample was broken down. This procedure was applied to all the prepared membranes and their respective breakage points were recorded as reported earlier. Similarly, the porosity (ε) of the prepared geopolymeric membranes was determined by the gravimetric method through Eq. (2) using distilled water [21]. The porosity of membrane is the volume of void spaces over the total volume of membrane which offers permeation to permeate. Through the gravimetric method, pore volume and total volume relies on the ability to fill the pores of the sample with water and to find out an accurate determination of void spaces volume.

$$\varepsilon = \frac{m_1 - m_2}{A \times D_m \times d_w} \tag{2}$$

where ε is the porosity of coated geopolymeric membrane, m_1 is the weight of wet membrane, m_2 is the weight of dry membrane, A is the effective surface area of membrane, D_m is the cross-sectional thickness of the membrane, d_w is the density of distilled water (0.98 g/cm^3). Silva et al. used the same methodology to find the porosity of laminar MoS_2 nanosheet for forward osmosis applications [22]. The scanning electron microscope (SEM) was used to analyze the average pore size of prepared geopolymeric membranes. The pore size of membranes was measured by plotting the standard scale on the pore size and the average was taken on the different dimensions of pores.

2.6. Testing method for separation properties

Synthesized fly ash-based geopolymeric membrane was tested in a stirrer cell of 2 L for the treatment of produced water. Initially, the membrane was washed in the sonicator for 10 min to remove loose particles from the membrane and dried at 120°C. Wastewater from the carwash was treated through the prepared membrane in a dead-end stirred cell of 2 L under the constant pressure of 2 bar. The intrinsic properties such as permeation channels and tortuosity of porous geopolymeric membranes were investigated through cross-sectional SEM as shown in Fig. 7. Naveed et al. [23] used geopolymeric membrane of a pore size of 1.77 and has a compressive strength of 14.09 MPa with 51.09% porosity for household wastewater treatment through dead-end filtration.

3. Results and discussion

3.1. XRF analysis of fly ash

X-ray fluorescence (XRF) analysis shows that fly ashes collected from Lakhra coal power plant contain silica and alumina oxides in amount and proportion which is suitable for geopolymerization. The aggregate percentage of silica and alumina oxide in fly ashes is 85% which is considered an optimum ratio for the synthesis of geopolymeric membrane. XRF analysis shows that fly ash collected from thermal power plants contains SiO_2 and Al_2O_3 at 63% and 22%, respectively with a silica to alumina ratio of 2.86 and needs no additives for adjustment of silica to alumina ratio.

3.2. FTIR analysis of fly ash and geopolymeric membrane

In the present study, FTIR spectroscopy was used to find the stretching of Si–O–Al, Si–O–Si and H–O–H in the fly ash as shown in Fig. 2. Spectral analysis of fly ash was obtained in the range of 4,000–450 cm^{-1} at a resolution of 1 cm^{-1} and the central band at 1,445 and 846 cm^{-1} caused the asymmetric stretching mode which shows high reactivity toward chemical activators. Furthermore, the results show that relative intensities of Si–O–Si and Si–O–Al for geopolymerization reaction are found in the range of 1,500–650 cm^{-1}. FTIR spectrum of fly ash shows a minor quantity of hydroxyl groups at 3,451 cm^{-1} which is due to the presence of bounded moisture contents. Functional groups (–OH) and particle-size distribution of fly ash predict the rate of geopolymerization reaction and effecting the resulting physical properties such as compressive strength, pore size and porosity of geopolymeric materials.

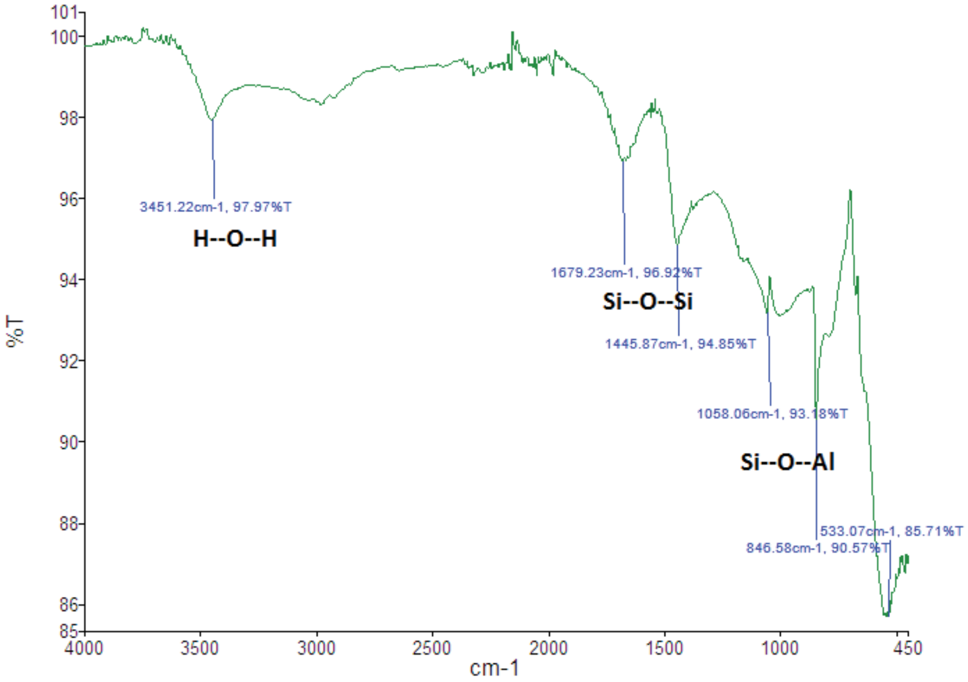


Fig. 2. FTIR pattern of fly ash used as a source material.

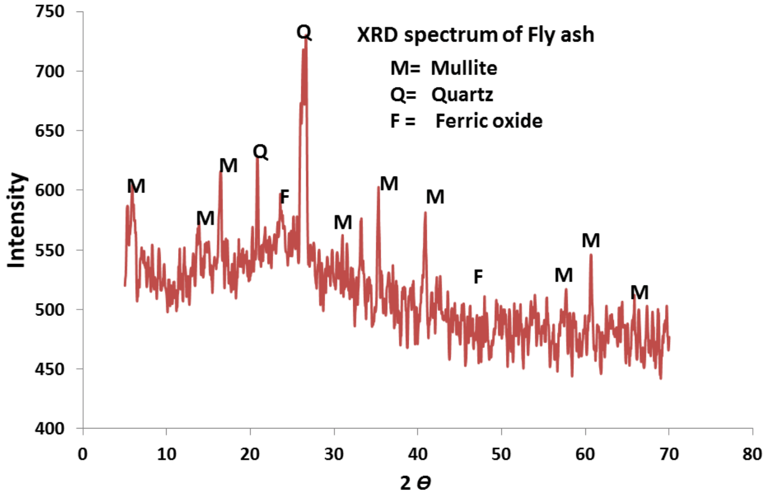


Fig. 3. XRD spectrum of fly ash used as a source material for membrane synthesis.

3.3. XRD analysis of fly ash

X-ray diffraction techniques were used to investigate the diffraction pattern of fly ash The fingerprints of fly ash collected from Lakhara power plant contain ferric oxide, quartz and mullite. XRD pattern shows that the fly ash was mostly an amorphous phase; however, some semi-crystalline peaks at 2θ value between 20°–27° were observed as shown in Fig. 3. Furthermore, the fly ash collected from power plant has a very similar mineralogical composition to red mud, metakaolin and rice husk. It was observed that mechanical activation of fly ash reduced the crystalline nature of fly ash and increase amorphous levels due to chemical activation and slight changes in the composition.

3.4. Particle-size distribution analysis of fly ashes

To investigate the effect of particle-size distribution on the reactivity of fly ash with chemical activators in geopolymerization, the particle size of fly ash was reduced to different ranges as shown in Fig. 4. Particle-size distribution of six different fly ash samples was recorded, that is, 4.30, 23.30, 46.20, 65.32, 86.24, and 98.53 μm through coulter counter. Fig. 4 shows that narrow particle-size distribution at low particle sizes compared to the wide particle-size distribution at larger particle sizes. Narrow particle-size distribution with small size particles typically reduces the time of dissolution and tends to completely dissolve in a short time.

3.5. Compressive strength and porosity of membranes

Fig. 5 shows the compressive strength and porosity against the source material particle size. No significant change in compressive strength and porosity can be observed below the source material particle size of 23.30 µm. This is attributed to the fact that most of the silica and alumina were completely dissolved in the alkaline solution during the specified mixing time resulting in the complete geopolymerization reaction. The compressive strength of 13 MPa obtained at 23.30 µm source material particle size is typically reasonable for microfiltration and ultrafiltration applications. As the fine size reduction is an energy-intensive operation, therefore, there is no need to reduce the source material particle size to a very fine range instead a little more time of mixing is preferable. This will significantly reduce the synthesis cost geopolymeric membrane.

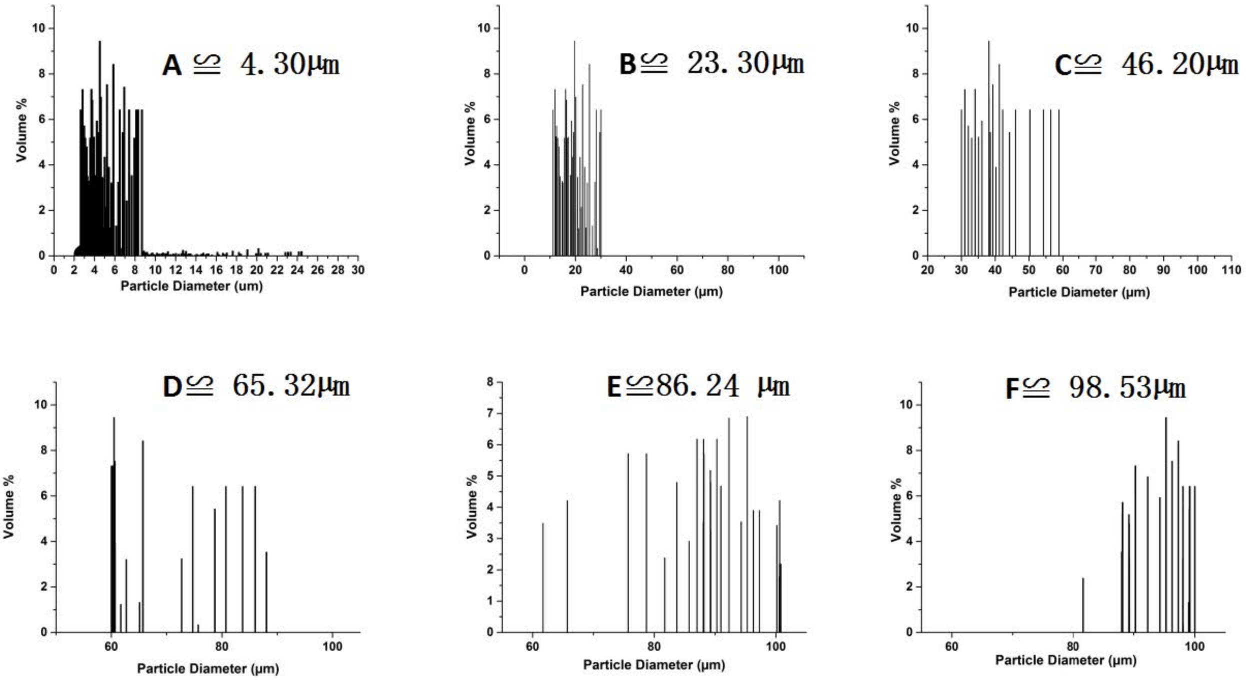
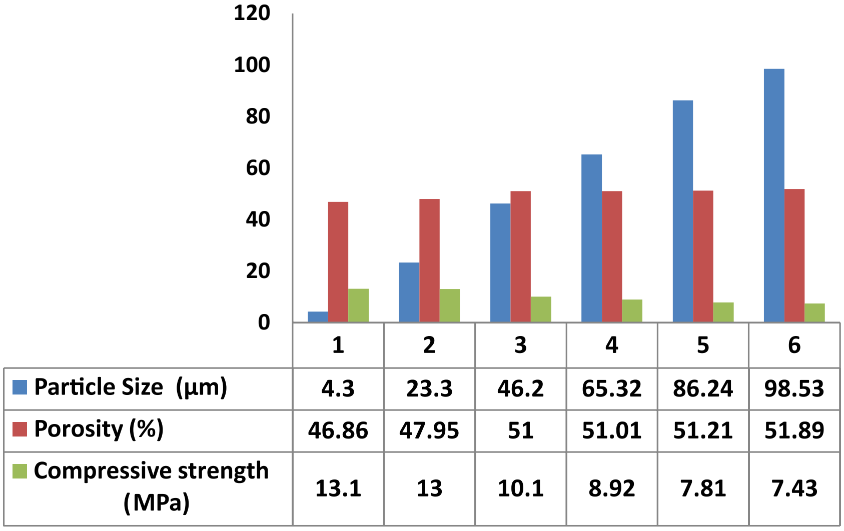


Fig. 4. Particle-size distribution of fly ash of different samples.



	1	2	3	4	5	6
🟦 **Particle Size (µm)**	**4.3**	**23.3**	**46.2**	**65.32**	**86.24**	**98.53**
🟥 **Porosity (%)**	**46.86**	**47.95**	**51**	**51.01**	**51.21**	**51.89**
🟩 **Compressive strength (MPa)**	**13.1**	**13**	**10.1**	**8.92**	**7.81**	**7.43**

Fig. 5. Influence of particle-size distribution of fly ash on the porosity and compressive strength of geopolymeric membrane (2 mm thickness; 50 mm diameter; 10 d).

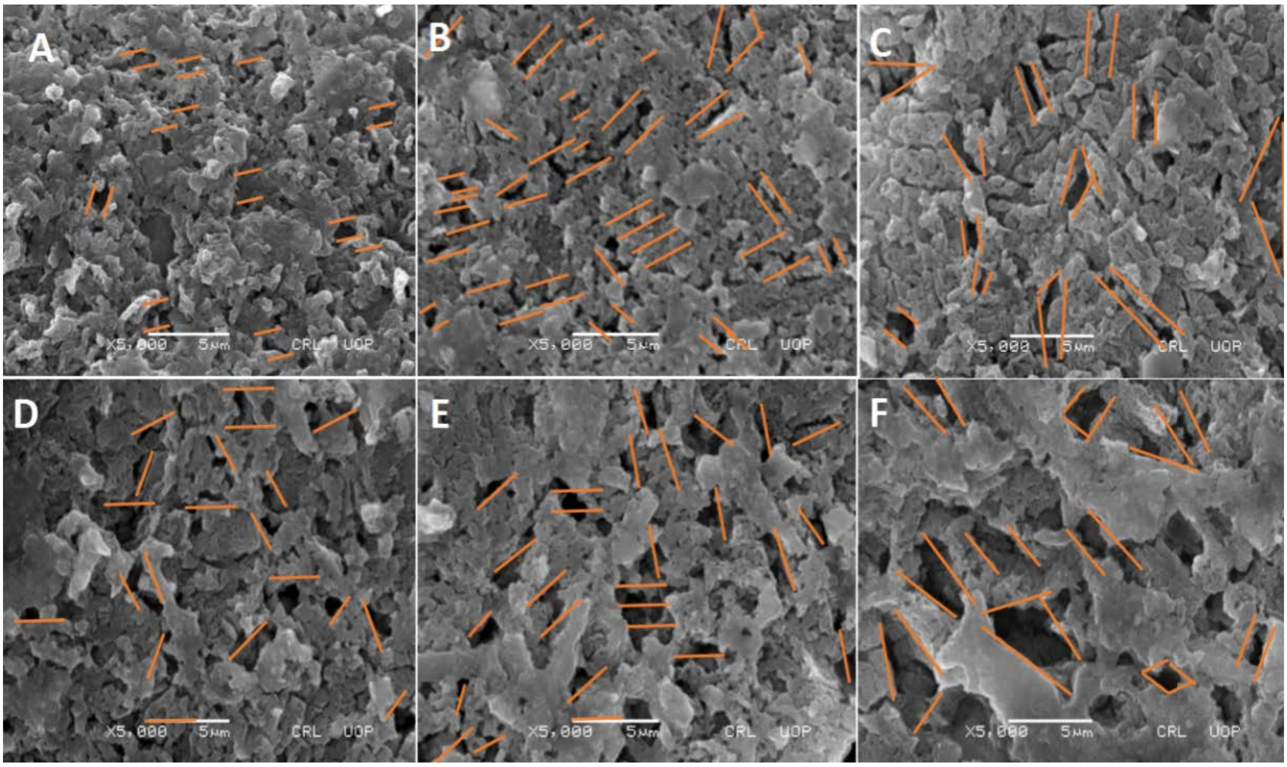


Fig. 6. Surface SEM micrograph of geopolymeric membrane showing different pore sizes. (A) 1.08 μm, (B) 1.99 μm, (C) 2.24 μm, (D) 2.59 μm, (E) 3.1 μm, and (F) 4.6 μm.

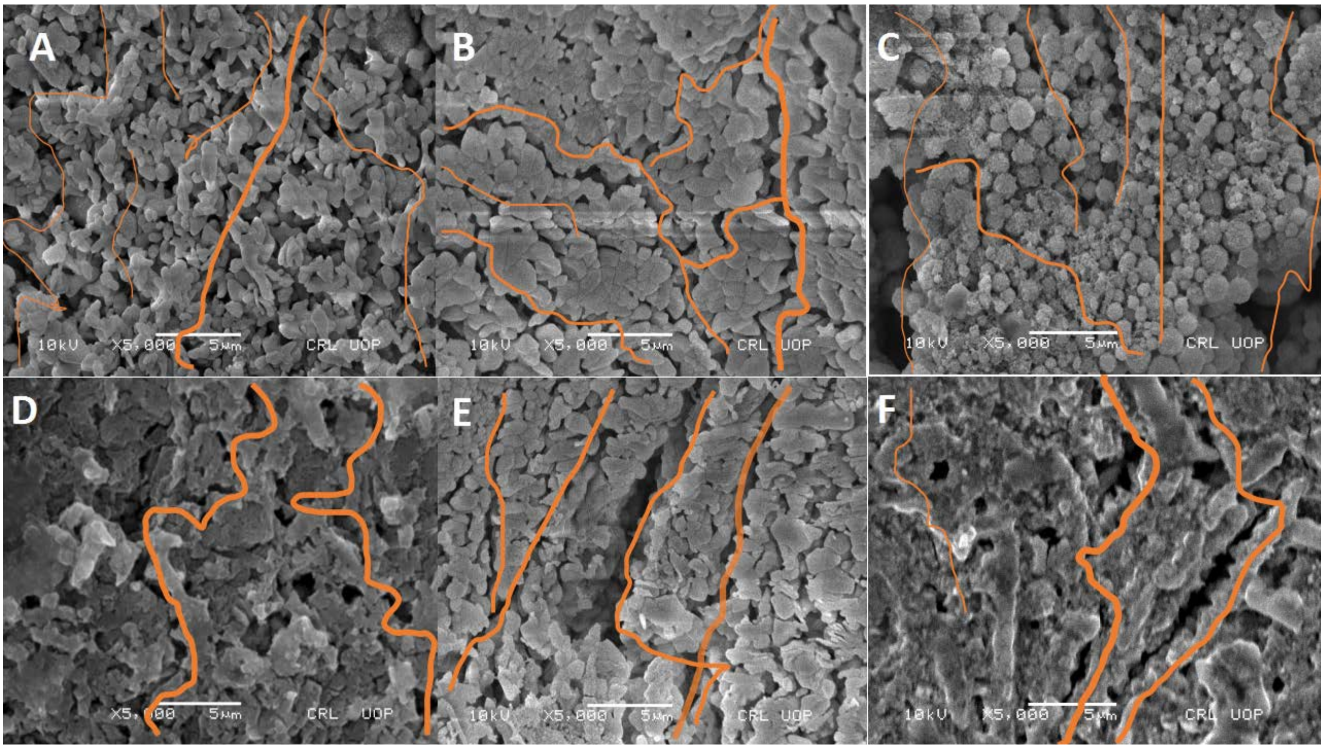


Fig. 7. Cross-sectional SEM micrograph of geopolymeric membrane showing different flow channels.

3.6. SEM of geopolymeric membrane

Fig. 6 shows the surface SEM of membranes prepared from six different source material particle sizes. No cracks have been observed in all prepared membranes. The average pore size of all the membranes of 4.30, 23.30, 46.20, 65.32, 86.24 and 98.53 µm particle sizes was found to be 1.08, 1.99, 2.24, 2.59 3.1 and 4.6 µm, respectively through the same procedure as reported in our previous study. More uniform pore sizes are observed as the particle size of the source material decreases. This could be attributed to the high dissolution of source material resulting in more mature polymerization. Irregular pore sizes at large particle sizes are due to incomplete dissolution and agglomeration of particles during geopolymerization which also resulted in low compressive strength. The intrinsic properties such as permeation channels and tortuosity of porous geopolymeric membranes were investigated through cross-sectional SEM as shown in Fig. 7. Fig. 7A and B show that permeation channels' widths of 4.30 and 23.30 µm of particle size membrane is narrow which are responsible for better rejection. It was observed that prepared membranes of large particle sizes such as 46.20, 65.32, 86.24 and 98.53 µm have scattered wide permeation channels with more tortuosity due to incomplete geopolymerization reaction.

3.7. Permeation test of different prepared membranes

A locally fabricated dead-end stirred cell having a capacity of 1 L was used for measurement of permeation and

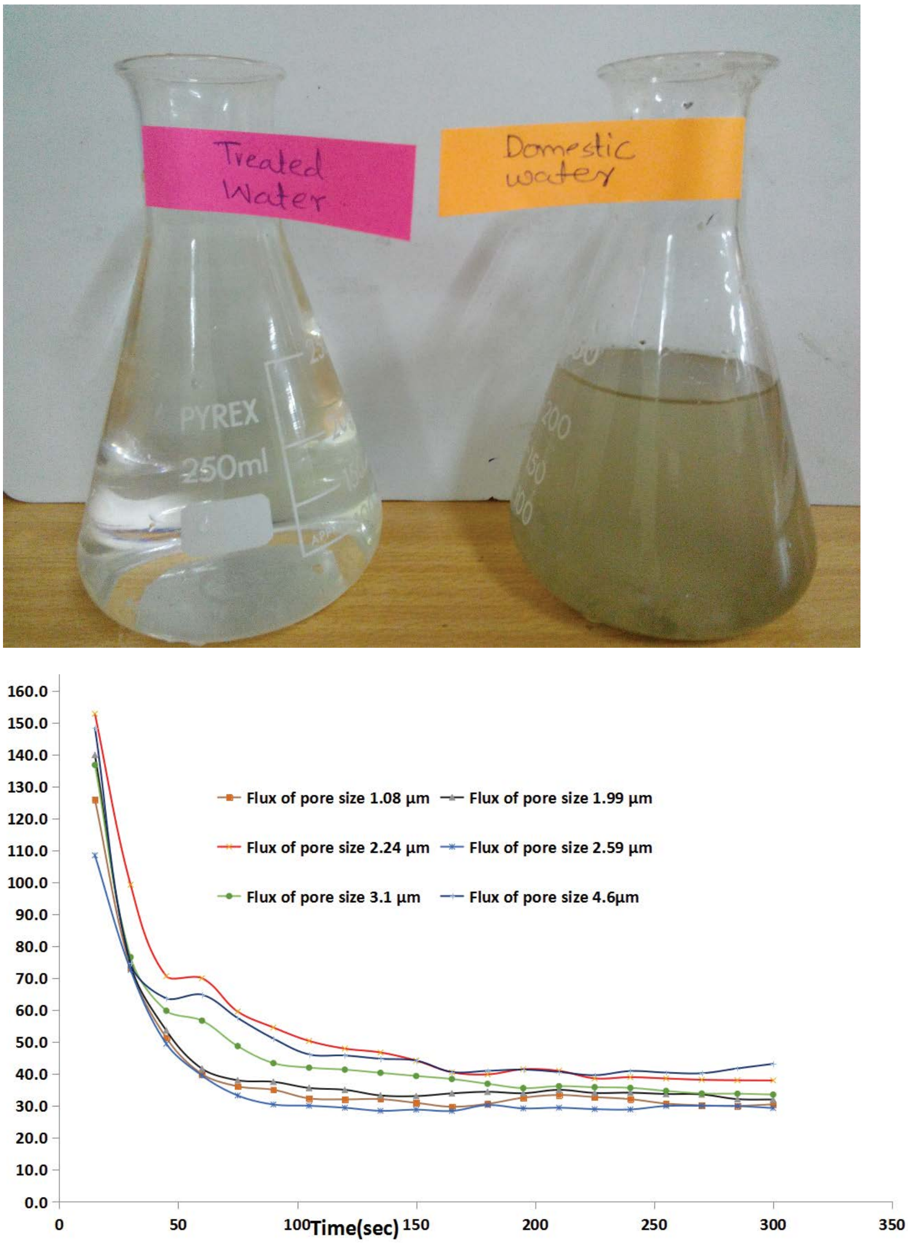


Fig. 8. Permeate flux of prepared geopolymeric membrane of different pore sizes before and after treatment.

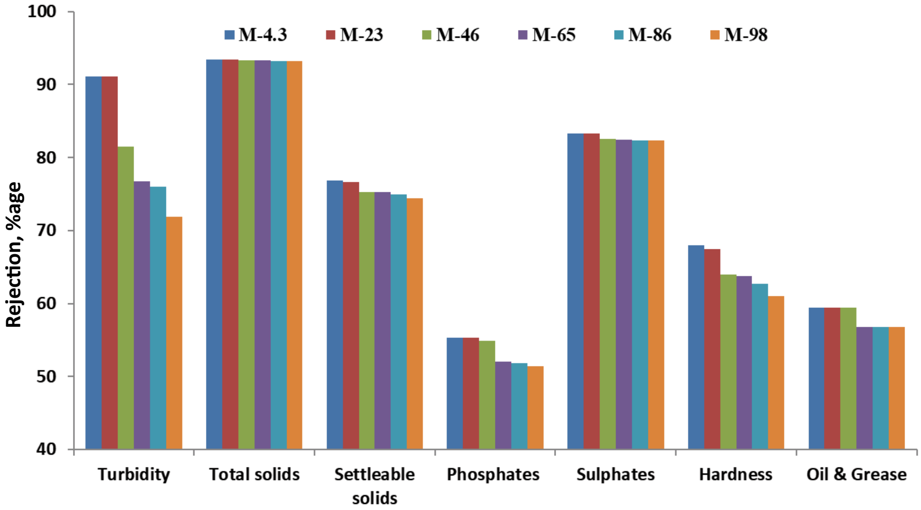


Fig. 9. Rejection and separation performance of geopolymeric membranes of different pore sizes.

rejection. Permeation tests were performed at 2 bar using geopolymeric membranes with pore sizes of 1.08, 1.99, 2.24, 2.59, and 3.1, 4.6 µm separately. The permeate flux (J) is computed using Eq. (3) and rejection through Eq. (4):

$$J_w = \frac{Q}{\left(\Delta t \times A_{\text{mem}}\right)} \tag{3}$$

where Q is the weight of filtrate in kg, A is the effective area in m^2 and Δt is the time in hours. Rejection of all the geopolymeric membranes has been calculated using Eq. (4):

$$R\% = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \tag{4}$$

where $R\%$ is the rejection of solute, C_p is the concentration of permeates and C_f is the concentration of the feed. The result shows that with increasing pore size, the flux increases slightly but rejection decreases significantly due to small change in porosity but large variation in pore sizes. Flux decline of 150–50 L/m^2 h can be observed at the very start due to the formation of the cake layer on the membrane surface as shown in Fig. 8. However, the membrane prepared with a pore size of 2.24 um showed a higher flux than other membranes. This might be due to less cake formation in the membrane at optimum pore size. Subsequently, the membrane prepared with a pore size of 4.6 um showed the lowest flux among all prepared membranes. The decline in the membrane flux could be due to the cake formation on the membrane surface and impurities in the wastewater which is affecting the overall performance of the membranes. After a decrease in flux for some time, a stable permeate flux was observed in all prepared membranes. The stability of the membrane is directly related to the impurities in the feed concentration. Initially, small impurities might reach a secondary layer of the membranes by diffusion and settle on the pores of the membrane to agglomerate in the cake form, however, the membrane pore size doesn't allow them to permeate through the membranes. Therefore, the flux at the start of the membrane was very high and high rejection rate but with time the membranes showed a stable performance after 150 s of each experiment. Experimental results show that geopolymeric membranes prepared from 23.30 µm are suitable for the treatment of wastewater from car wash center to separate total solid, settle-able solid, phosphates, sulfates, and greases contents as shown in Fig. 9.

4. Conclusion

In this study, we reported the effect of particle size of source material on the compressive strength, pore size and porosity of synthesized membrane due to dissolution of particles. No significant change was observed below 23.30 µm in compressive strength, pore size and porosity due to complete dissolution of fly ash in chemical activators and completion of geopolymerization reaction. Uniform pore sizes and porosity has been observed at low particle size of the source material due to mature geopolymerization reaction. This study suggests that the very fine particle size reduction is to be avoided as the fine size reduction is a very energy-intensive operation instead more mixing time is to be provided for the particles to be completely dissolved in the alkaline solution. Mixing time should be enhanced instead of energy and time on particle size reduction. Uniform pore sizes and porosity has been observed at a low particle size of the source material due to mature geopolymerization reaction. This study suggests that the very fine particle size reduction is to be avoided as the fine size reduction is a very energy-intensive operation instead more mixing time is to be provided for the particles to be completely dissolved in the alkaline solution.

Acknowledgment

The present research work was fully funded by the Pakistan Science Foundation (PSF/NSFC-II/Eng/KP-UET-09).

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