

Effect of processing parameters on mullite bonded SiC membrane for turbid water filtration

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Abstract. A water-filtration membrane made of SiC possesses some excellent properties, but its application is limited by high fabrication cost. In this study, two sets of mullite bonded porous SiC ceramics membranes were prepared at reduced temperature by oxidation bonding method using different processing conditions. Dead-end filtration mode was utilized for the determination of permeability and their efficiency towards removal of turbidity. It was found that all the membranes prepared using different composition, pore formers (graphite, PVC and PMMA) and sintering temperature exhibited high turbidity removal efficiency (> 99%). This study provides an efficient method to prepare porous SiC ceramics with excellent permeability and turbidity removal efficiency, which will be helpful for the design of low cost SiC ceramic filters for water treatment.

Keywords: membrane; microfiltration; SiC ceramic; turbidity

1. Introduction

In the rainy season, high-turbidity water has been reported as a challenge for water treatment facilities and turbidity is the most problematic factor in water treatments (Kan *et al.* 2002, Mann *et al.* 2007, Bilotta and Brazier 2008). The higher turbidity level in drinking water causes risk to human health due to the suspended solids contaminated with viruses, bacteria etc. (Mann *et al.* 2007, Bilotta and Brazier 2008). Ceramic membranes are mainly used to remove particles contained in drinking water; however, they have recently been gradually expanded to other water treatments (Chang and Liao 2003, Ciora and Liu 2003, Cha *et al.* 2019, Jang and Lee 2018). Coagulation/flocculation pre-treatments, combined with ceramic MF membrane filtration, are nowadays seen as an alternative, less energy-consuming membrane process for microbial, natural organic matter and turbidity removal (Li *et al.* 2010, Meyn and Leiknes 2010, Maddah and Chogle 2015). Membranes used in MF/UF operations are very similar to conventional filters, with a rigid, highly porous structure of randomly distributed and interconnected voids. However, these pores differ from those in a conventional filter by being extremely small, in the order of 0.01–10 µm in diameter (Baker 2012, Gitis and Rothenberg 2016). According to the pore size distribution of the membrane, all particles larger than the largest pores are completely rejected by the membrane and particles smaller than the largest pores but larger than the smallest pores are partially rejected (Baker 2012). Ceramic membranes are mainly

made from inorganic ceramic materials (zirconia, alumina, titania, SiC etc.) and consist of several layers of one or more expensive ceramic material with direct bearing on the commercial ceramic membrane price (Li 2007, Mulatu 2014, LiqTech International 2014). Therefore development of low cost ceramic membrane is highly demanding in separation and purification technologies. SiC based ceramic filters promote new opportunities as they show possibilities of wastewater treatment in many advanced chemical and oil industries but again are limited by high fabrication cost. To reduce high sintering temperature and costly inert atmosphere, oxide-bonding technique is accepted as a simple technique for fabrication of macro porous SiC membrane (Kim *et al.* 2017, Bukhari *et al.* 2017, Das *et al.* 2020a, b). In the oxide bonding technique, powder mixtures of SiC, with and without oxide additives (Al₂O₃, MgO, etc.) and pore former are heated in air instead of an inert atmosphere. At the heating stage, the oxidation derived SiO₂ glass from surface oxidation SiC particles crystallize to cristobalite and simultaneously reacts with oxide additive to form secondary oxide bond phases like, mullite, cordierite, etc., depending on the types of additive used.

The detailed studies on applicability of oxide bonded SiC ceramic membrane for treatment of waste water with high turbidity are very few. The aim of the present work is to explore the applicability of porous SiC ceramic membrane in removal of turbidity from drinking water. The membranes used in this study were prepared using two different sintering aids, e.g., (a) small amount of clay and (b) fine industrial waste fly ash. The effect of organic (PMMA and PVC) and inorganic (graphite) pore formers on the pore structures of ceramics and efficiency of turbidity removal are evaluated.

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2. Experimental procedure

The detailed preparation and characterization of the membranes used in this study are described in our earlier publications (Das *et al.* 2020a, b). High purity α -SiC powder (Grindwell Norton, India; SiC 98.20%, $d_{50} = 22.5 \mu\text{m}$), Al_2O_3 (Indian Aluminium Co. Ltd., India, HIM-30 Grade, α - Al_2O_3 ; purity > 98%, $d_{50} = 0.35 \mu\text{m}$), Kaolin clay (Loba Chemie, purity 99.99%, $d_{50} = 4.32 \mu\text{m}$), ground sieved fly ash (CESC Budge Budge, Kolkata, India, $d_{50} = 16 \mu\text{m}$), and Molybdenum Oxide (MoO_3 , Loba Chemie, India) were used as raw materials for preparation of SiC membranes. Graphite powder (Kanodia Minerals & Chemical Co., Howrah, India; $d_{50} = 10 \mu\text{m}$), polymethyl methacrylate (PMMA, Sigma Aldrich, $d_{50} = 8 \mu\text{m}$) and poly vinyl chloride (PVC, Loba Chemie, Mumbai, India, grinded and sieved to $d_{50} = 10 \mu\text{m}$) were used as pore formers. The powder mixture was homogeneously mixed with 10% poly vinyl alcohol solution (PVA, Loba Chemie, India) and uniaxially pressed at 23MPa in a stainless steel die into circular disc with diameter of 40 mm and thickness of 8 mm. Membranes prepared using Batch A composition (90 g SiC, 10 g Al_2O_3 , 1 g clay and 10 g pore former) at 1400°C for 1 h were named as SC, SC-G, SC-PC and SC-P respectively. Membranes prepared using Batch B composition (80 g SiC, 20 g fly ash, 5 g MoO_3 and 10 g pore former) at 1000°C for 1 h were designated as SF, SF-G, SF-PC and SF-P respectively. The mechanical and water permeability data of the membranes are summarized in Table 1.

The turbidity removal of drinking water test was carried out in steady-state regime at room conditions using a laboratory-made apparatus as illustrated in our earlier publication. The typical evaluation of the retention ratio (R) of MF membranes is carried out under constant transmembrane pressure (TMP), where the concentration of the pollutant in the feed (C_f) is kept constant and the pollutant concentration (C_p) and the flux (J) in the permeate are monitored along time. In this work, an alternative transient method was applied to enable faster comparison of samples. The turbid water was prepared by adding 200 g of kaolinite (powder) in 8 L of chlorine-free water under 300 rpm agitation for 2 h. The mixture was settled by 48 h under room conditions and the supernatant water was collected for the experiments, with a turbidity level in the range 104-166 NTU. For each membrane sample, two tests were run: first with distilled water to acquire the permeation profile and then with the turbid water containing kaolinite particles to verify the retention ratio (R) and fouling of the membrane. The sample under test (diameter of 40-41 mm and thickness of 9-11 mm) was sealed with annular rubber rings within a cylindrical sample holder that provided a useful medium diameter of 2.4 cm a circular flow area (A_{flow}) of 4.52 cm^2 . During the test, a volume of distilled water ($V_{w,i}$) of 200 mL was fed in the reservoir above the sample of total volume (V_t) of 448 mL. Therefore, an initial volume of air ($V_{\text{air},i}$) of 248 mL was kept above the water level. The air pressure (P) in the reservoir was raised to provide an initial transmembrane pressure ($\text{TMP}_i = P_{f,i} - P_{\text{atm}}$) of $\approx 900 \text{ mbar}$ (90 kPa) relative to the atmosphere. The valve between the

Table 1 Features of the prepared membranes used for turbidity removal

Sample code	Material property			Water permeability		
	Pore former	Porosity (vol. %)	Flexural strength (MPa)	k_1 (10^{-13} m^2)	k_2 (10^{-8} m)	Sp. water permeability $\text{Lm}^{-2}\text{h}^{-1}\text{bar}^{-1}$
Membranes prepared with Batch A composition						
SC	----	38.22 \pm 0.06	38.47 \pm 0.03	0.92	1.79	4755
SC-G	Graphite	48.56 \pm 0.20	31.86 \pm 0.13	2.62	1.85	12344
SC-PC	PVC	49.90 \pm 0.10	28.53 \pm 0.17	2.34	2.63	10992
SC-P	PMMA	48.02 \pm 0.44	31.53 \pm 0.19	3.16	4.32	14079
Membranes prepared with Batch B composition						
SF	----	36.36 \pm 0.14	38.40 \pm 0.09	0.36	0.10	1532
SF-G	Graphite	42.03 \pm 0.11	34.31 \pm 0.05	0.83	0.80	3574
SF-PC	PVC	44.7 \pm 0.13	28.60 \pm 0.15	1.13	3.51	5261
SF-P	PMMA	45.96 \pm 0.15	27.92 \pm 0.18	1.51	2.66	6113

reservoir and the sample was then open and the water was allowed to permeate downward through the sample, being collected in a test tube for analysis. The air pressure inside the reservoir continuously decreased due to the reduction in the water level caused by the permeation through the membrane. The TMP data was collected in a datalogger at each 1 s and a mass balance inside the reservoir allowed to estimate the cumulated volume of water permeated through the membrane (V_p) as a function of time by:

$$V_p = V_{\text{air},i} \left(\frac{\text{TMP}_i - \text{TMP}}{\text{TMP} + P_{\text{atm}}} \right) \quad (1)$$

The decay in the transmembrane pressure along time (PD) was obtained by:

$$\text{PD} = \left(\frac{\text{TMP}_i - \text{TMP}}{\text{TMP}} \right) 100\% \quad (2)$$

The same procedure was carried out with the same membrane, but this time with replacement of clean water by turbid water (diluted kaolinite suspension). The retention ratio (R) was evaluated after 300 s of test by measuring the turbidity of water in the feed and in the permeate using a portable turbidimeter (HACH, model 2100 p, Loveland, CO, U.S.A.). The atmospheric pressure at the laboratory conditions (P_{atm}) was in the range 935-942 mbar (93.5-94.2 kPa) and temperature between 21 and 24°C . The reduction of flow caused by fouling was obtained by comparing the volumes of permeate of clean and turbid water after 300 s of test:

$$\text{FR} = \left(\frac{V_{p,\text{clean water}} - V_{p,\text{turbid water}}}{V_{p,\text{clean water}}} \right) 100\% \quad (3)$$

3. Results and discussion

The porosity, average pore diameter and flexural strength varied in the range of 38-50 vol%, 3.7 - $6.5 \mu\text{m}$ and 28-38

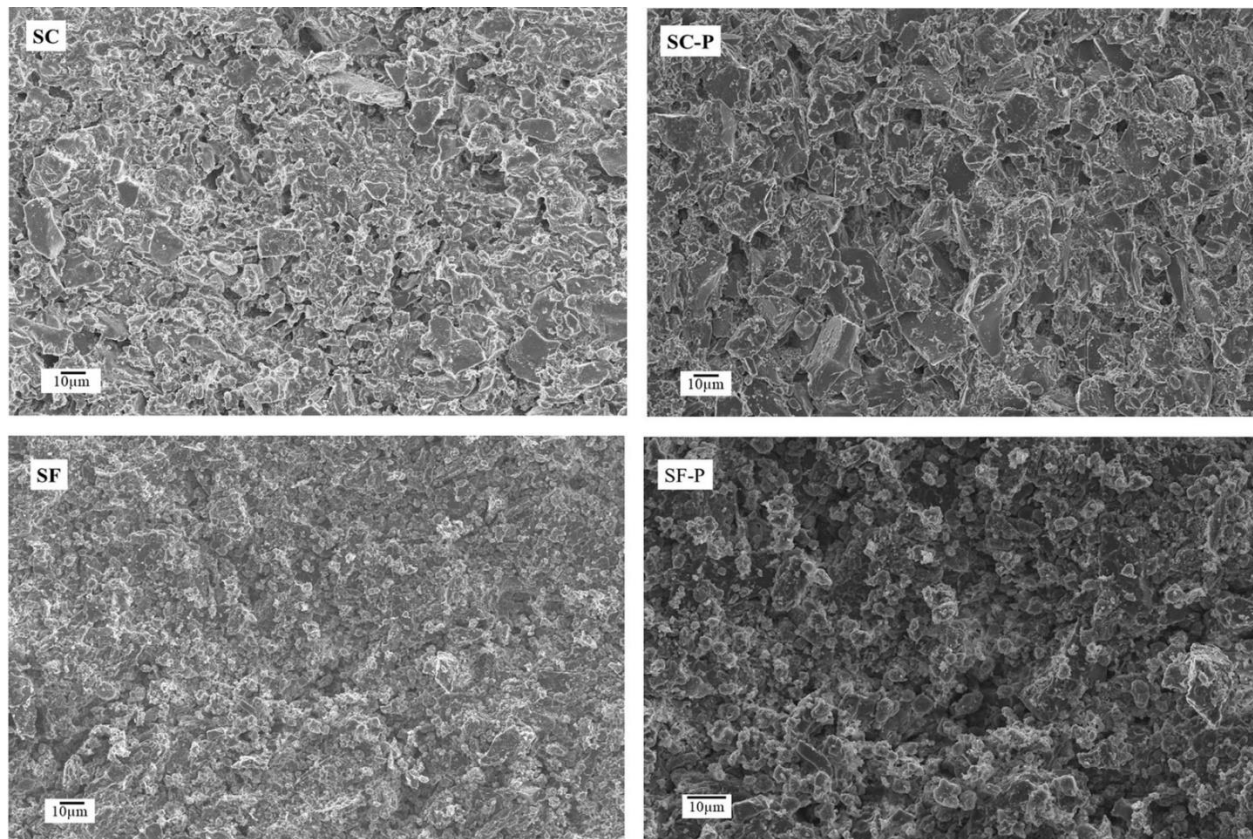


Fig.1 SEM micrographs of porous SiC ceramic membranes prepared (L) without and (R) with pore formers

MPa respectively for samples prepared with batch A composition. The samples prepared using industrial waste fly ash (batch B) showed a variation of porosity, pore size and flexural strength in the range of 36-46 vol%, 2.9-4.0 μm and 28-38 MPa respectively. The porosity of the sintered sample for both the batches were increased significantly with the addition of pore formers. The variation of porosity in final ceramics prepared with different pore formers was found to be negligible. Slightly higher porosities were obtained for the sample prepared with PMMA over graphite and PVC may be due to comparatively lower density of PMMA (Boaro *et al.* 2003). The microstructures of SiC microfiltration membranes prepared with and without pore former of each batch are shown in Fig. 1. Number of pores and pore sizes were found to increase with addition of pore former as evident in the microstructure which lead to higher porosity in sample. The specific permeability (SP) of clean distilled water of the membrane prepared using batch B composition decreased from 6113 to 1532 $\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$ with decrease of porosity from 46 to 36 vol%. Similarly, the SP of membrane prepared using batch A composition also exhibited decrease in SP from 14079 to 4755 with decrease of porosity from 50 to 38 vol%. A higher SP value obtained for the sample prepared with PMMA in both batches probably due to better dispersion of PMMA and formation of spherical pores coming from the regular size of PMMA particles compared to irregular size and agglomerated particles of graphite and PVC. Elomari *et al.* (2017) prepared clay based microfiltration membrane of varying

porosity using 0-20 wt% corn starch as pore former and reported a highest clean water SP value of 1300 $\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$ for the sample prepared with 20% corn starch having porosity 48.2 vol%. Aloulou *et al.* (2017) reported SP of 1228 $\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$ for the porous sand based microfiltration membrane with porosity > 40 vol% prepared using starch as a pore former.

The turbidity removal efficiency of the membranes prepared using different sintering aids and sintering temperatures were also compared. In the dead-end filtration, the complete feed flow was forced perpendicularly through the membrane and the retained matter is accumulated as a fouling layer (or cake) on the surface of the membrane, while the permeate (or filtrate) is collected as it passes through the membrane. Pictures of membranes after the filtration tests are given in Fig. 2. The thickness of this cake therefore increases with time and the permeation rate correspondingly decreases. The plots of water flux (J) as a function of time of all samples are presented in Fig. 3.

The flux reduced to 47.34%, 77.8%, 80.01% and 77.87% respectively for SC, SC-G, SC-PC and SC-P membranes after 150 s of filtration. Similarly, the flux declined to 1.78%, 42.62%, 66.38% and 75.23% respectively for SF, SF-G, SF-PC and SF-P. The turbidity level of water before and after filtration ($t = 300$ s) for all membranes is given in Fig. 4. More than 99% of turbidity was removed by all the ceramic-membranes used in this study tested under same conditions. In this study the test is done with high turbid water. At the beginning of the filtration, complete blocking, intermediate blocking, and

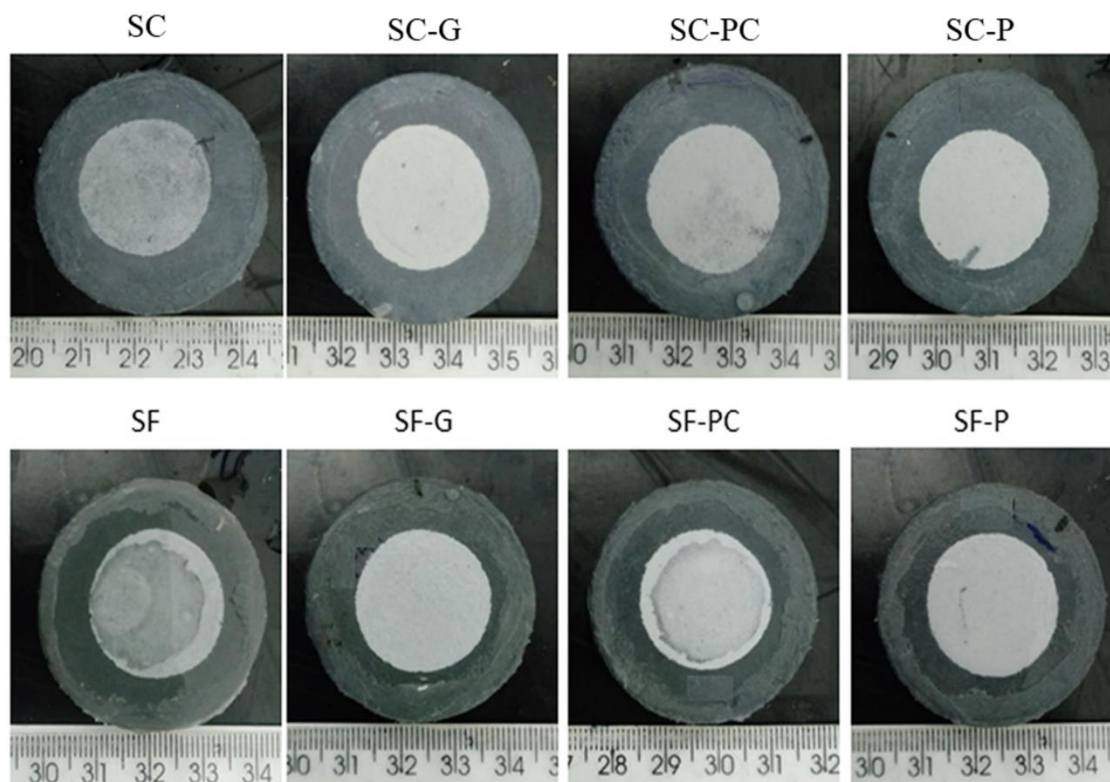


Fig. 2 Picture of membranes after the turbidity removal test

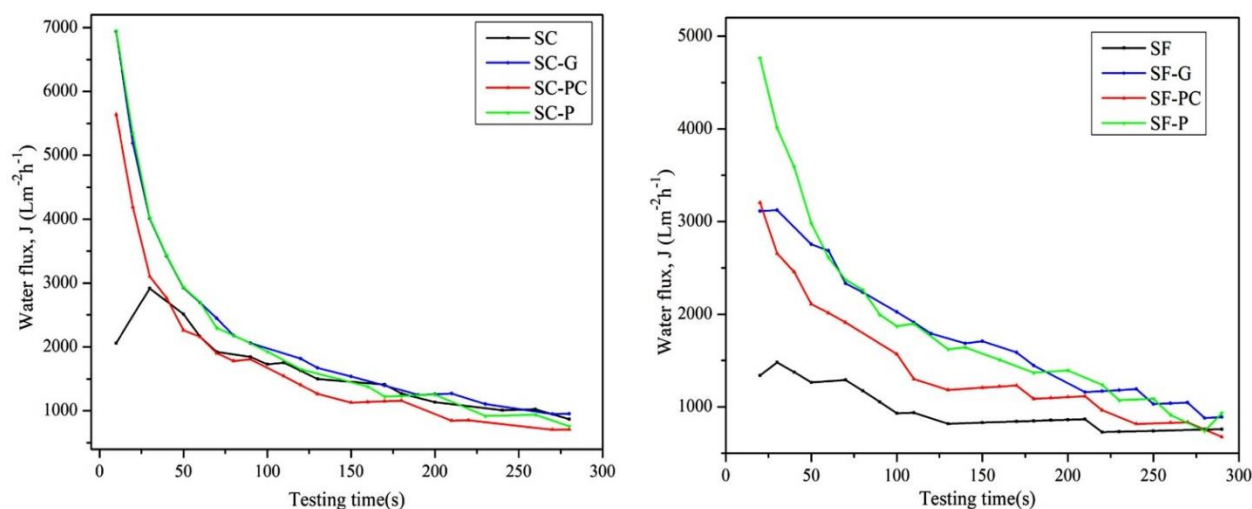


Fig. 3 Water flux curves for tested membranes during turbid water flow

cake filtration are the major fouling mechanisms, as the blocking lowered drastically, and cake filtration became the main fouling mechanism. It is well established that as the turbidity in the feed water increased, the irreversible fouling increased (Park *et al.* 2020). The turbidity and formation of cake layer played the major influential factor in filtration through ceramic membrane. Therefore, in the present study all membranes showed ~ 99% removal efficiency irrespective of their porosity.

The small variation in flux rate was due to the variation in microstructure, porosity and pore diameter of final ceramics arisen from variation in green formulation, pore former and processing temperature. Commercial ceramic

tubes with porosity > 70% and pore size > 0.2 μm showed more than 95% turbidity removal from water containing turbidity of 9.7 NTU (Hofs *et al.* 2011). Park *et al.* (2020) reported removal of > 99% turbidity from water containing turbidity of 60 NTU by $\text{ZrO}_2\text{-TiO}_2$ hollow tube with pore size > 0.2 μm . During microfiltration of textile effluent water containing initial turbidity of 167 NTU at operating pressure of 0.12 bar by clay based membrane, it was reported a decrease in turbidity removal efficiency from 96 to 80% after 60 min of filtration with increase in porosity of the membrane from 30.8 to 48.2 vol% (Elomari *et al.* 2017).

The percentage variation on turbidity removal and flow reduction (FR%) due to fouling after 300 s with variation of

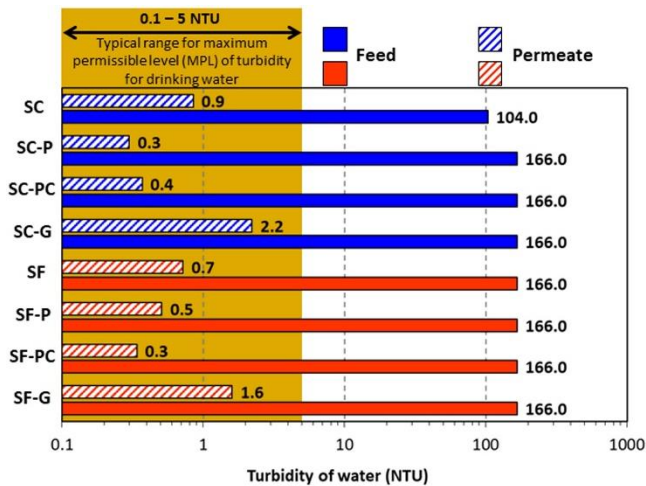


Fig. 1 Schematic representation of the experimental procedure

Table 2 Performance parameters of prepared SiC membranes for turbidity removal

Sample	Porosity (vol%)	Turbidity removal (%)	Flow reduction due to fouling after 300s (%)	Retention ratio (turbidity removal) after 300 s of filtration	Sp. water permeability for turbid water ($\text{Lm}^{-2}\text{h}^{-1}\text{bar}^{-1}$)
Batch A composition					
SC	38.2	99.2	19.2	99.2	3422
SC-G	48.5	98.7	61.1	98.7	8264
SC-PC	49.9	99.8	67.5	99.8	6617
SC-P	48.0	99.8	62.1	99.8	8255
Batch B composition					
SF	36.4	99.6	29.0	99.6	926
SF-G	42.0	99.0	33.3	99.0	2470
SF-PC	44.7	99.8	53.3	99.8	3981
SF-P	45.9	99.7	48.7	99.7	5412

membranes types are summarized in Table 2. It can be seen that flow reduction increases with increase in porosity and pore size of the membranes. For batch-A membranes, with increases in porosity from 38 to 50 vol%, FR increases from 19.2 to 67.5%. For the membranes prepared using batch-B composition the FR increases from 29 to 53.3% with increase in porosity from 36 to 46 vol%. Decline of flow mainly caused by two factors, one is pore blocking and another is concentration polarization (Chakrabarty *et al.* 2008). During microfiltration of turbid water, membrane with high porosity and pore size experiences higher pore blocking phenomenon in early stages compared to membrane with lower porosity, caused enhanced flux decline and as the microfiltration proceed concentration polarization also takes place (Vasanth *et al.* 2013). After some time of microfiltration pore blocking phenomenon become insignificant and the flux is reduced due to concentration polarization and this type of fouling is reversible in nature. It has been reported that the ceramic membrane can be easily cleaned by simple backwashing

with water and can be reuse for another filtration purpose. Vasanth *et al.* (2013) also reported highest flux decline of 83.2% for clay membrane with porosity 30 vol% and pore size $1.30\text{ }\mu\text{m}$ while for the membrane with porosity 23 vol% and pore size $0.45\text{ }\mu\text{m}$, it showed 52.2% flux decline during oil-water filtration. According to Rajanna *et al.* (2019) a highest decline of flux $\sim 83.8\%$ was obtained after 24 h filtration of industrial waste water using porous SiC ceramic membrane with pore size $1.5\text{ }\mu\text{m}$. They also reported simple backwashing increased the recovery of flux value significantly and the membrane was found to be reusable. A high flux reduction of 95% were obtained after 36 min during microfiltration of synthetic high turbid water with initial turbidity of 60 NTU using commercially available $\text{ZrO}_2\text{-TiO}_2$ (Sterlitech Co., WA, U.S.A.) based ceramic membrane with average pore size of $0.20\text{ }\mu\text{m}$ (Park *et al.* 2020). The reduction of flux was explained by fouling due to formation of cake by the particles present in the turbid water during microfiltration. The permeability, turbidity removal and flow reduction results of the membrane prepared in the present work are comparable with the reported membrane in the literature.

4. Conclusions

The efficiency of SiC membranes were evaluated to remove the turbidity from water containing initial turbidity 104–166 NTU. All the membranes showed 99% removal efficiency irrespective of the porosity and composition. Depending on the porosity of the membranes specific permeability of turbid water varied from 926 to 8264 $\text{Lm}^{-2}\text{h}^{-1}\text{bar}^{-1}$. The membrane prepared without pore former showed best result in respect of the reduction of flow (FR%) due to fouling. On the other hand, the membrane prepared with PMMA as pore former showed best water flux. The research findings of this work clearly demonstrate the need of the low cost SiC ceramic membranes prepared by utilizing waste industrial fly ash or clay and alumina as sintering additives in comparison with the value added porous SiC ceramics prepared at high temperature. The membranes prepared in this study with batch B composition may be recommended as the best as it is prepared at comparatively low temperature (1000°C) using waste coal fly ash which not only reduce environmental pollution but also produce high-valued SiC-membranes with strong application possibilities in various fields. The excellent permeability and turbidity removal efficiency results of the porous SiC membrane prepared in this study will be helpful for the design low cost ceramic filter for water treatment.

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