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Preparation of kaolin-based low-cost porous ceramic supports using different amounts of carbonates

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ABSTRACT

Flat ceramic membrane supports were prepared using kaolin as the major constituent with varying amounts of carbonates and sintered at 900˚C. The prepared supports were subjected to SEM, XRD, and porosity tests. The supports prepared without using carbonates had the largest mean pore size with the lowest porosity. The porosity of membranes increased by increasing the amount of calcium carbonate. The supports prepared using calcium carbonate had wider pore size distribution on the surface than those prepared using sodium carbonate. Small amount (10%) of sodium carbonate acts as a pore modifier resulting in smaller mean pore size, while large amount (>20%) of sodium carbonate blocks the pores by forming a sodium silicate layer and results in nonporous support. Therefore, calcium carbonate should be preferred over sodium carbonate for preparing highly porous ceramic membranes.

Keywords: Ceramic membrane; Sintering; Kaolin; Microfiltration; Porosity; Pore size distribution

1. Introduction

Porous ceramic membranes (also called inorganic membranes) possess superior chemical, thermal, and mechanical stability with longer life span than polymeric membranes [1–3]. Their high cost and large pore size limit the extensive use of ceramic membranes. However, these membranes are used in high-temperature applications such as filtration of hot oily streams, high-pressure applications such as nanofiltration and reverse osmosis, and handling of corrosive fluids as well as detergent-containing wastewaters [4,5]. Early research on the preparation of ceramic membranes involved the utilization of α-alumina as the building material [6], which is expensive and requires high sintering temperature (>1,300˚C). To overcome these issues, current research is emphasized toward the use of cheaper alternative materials such as ball clay, fly ash, dolomite, and kaolin for membrane fabrication [7–9]. Among these materials, kaolin is widely used as a chief constituent in the preparation of microfiltration range ceramic membranes [7,10–12].

Low-cost ceramic membranes prepared using kaolin have been found to be applicable in different purification processes such as fruit juice clarification, separation of catalyst particles in heterogeneous reactions, removal of *E. coli*, separation of coal from organic solvents, treatment of oily wastewaters,

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micellar-enhanced microfiltration of heavy metal ions and treatment of industrial effluents [10–15]. In addition, they are used as supports for preparing composite membranes such as polymer/ceramic ultrafiltration membranes and metal/ceramic composite membranes [16–19].

The flat ceramic membranes are generally prepared by either paste method or dry powder compaction method. In comparison with dry compaction, the paste method has been found to produce highly porous ceramic membranes [20]. Therefore, the paste method was followed in this work for membrane preparation.

Earlier research on the development of ceramic membranes has been focused on the optimization of sintering temperature and selection of alternative low-cost raw materials [21,22]. However, the role of carbonates as pore-forming agents in the fabrication of porous membranes cannot be ignored. No study has been reported so far on the effect of amount of different carbonates such as Na_2CO_3 and $CaCO_3$. Therefore, it is important to systematically study the effect of these carbonates on the permeation properties of inorganic membranes.

This work uniquely describes the effect of different carbonates on the thermal and phase transformations, porosity, surface pore size distribution, and mean pore size of ceramic membrane supports. A comparative assessment of support properties has been carried out for the membranes prepared using calcium carbonate, sodium carbonate, and mixed carbonates. This work will be helpful to ascertain the role of the two different carbonates in the pore modification during the preparation of low-cost inorganic microfiltration membranes.

2. Materials and methods

2.1. Raw materials

All the chemicals, namely kaolin, sodium carbonate, calcium carbonate, sodium metasilicate, and boric acid, graded with at least 99.5% purity, were procured from CDH India Ltd. and were used without any pre-treatment. These five different constituents impart different properties to the ceramic supports. Kaolin not only serves as a cheaper raw material but also provides low plasticity and better refractory characteristics. Boric acid (a white powder that dissolves in water) brings homogeneity to the membrane structure and increases the mechanical strength, by forming metallic metaborates at high temperatures [7]. Sodium metasilicate is used as a binder as it forms silicate bonds among the particles and induces high mechanical strength to the membranes. Calcium carbonate and sodium carbonate provide porous texture to the membranes. Calcium carbonate decomposes at temperatures above 650°C into calcium oxide and carbon dioxide [10,11]. On the other hand, sodium carbonate melts at temperatures above 800°C and reacts with silica of kaolin to form sodium silicate and carbon dioxide [12,23]. The released carbon dioxide gas bubbles pass through the membrane, causing rearrangement of solid grains and thereby, imparting porous texture to it.

The major raw material, kaolin was analyzed by SEM (shown in Fig. 1(a)) and EDAX (presented in Table 1). The results indicated that the kaolin sample was pure and K_2O was present in trace amounts. The particle size distribution of raw materials was analyzed by a laser particle size analyzer (Malvern, Mastersizer 2000) and is shown in Fig. 1(c). The mean particle sizes of these materials (raw kaolin, $CaCO_3$, Na_2CO_3, and finely ground mixture) were 4.6, 7.9, 29.4, and 1.1 μm, respectively. It can be observed from this figure that the raw materials mixture after grinding in a ball mill had a narrow particle size distribution as compared to fresh raw materials. Sodium metasilicate was in the form of colorless crystals (of size 0.1–1.2 mm) that are readily soluble in water.

2.2. Preparation of porous ceramic supports

Ceramic supports were prepared by paste method [8] using twelve different compositions presented in Table 2 with varying amounts (0–40 wt.%) of calcium carbonate and sodium carbonate. Firstly, the raw materials were accurately weighed according to the chosen composition and were ground to a fine powder using a ball mill (SEM picture is shown in Fig. 1(b)). Then, a predetermined amount of distilled water (25–35 wt.%) was added to make a thick and uniform paste. The quantity of water required for paste-making depends on $CaCO_3$ content, ambient temperature, and humidity. The raw materials paste was casted in a circular mold of inner diameter of 50 mm and height 5 mm. The prepared ceramic supports were kept under a uniform load of 2 kg for 12 h, to prevent deformation, followed by ambient drying for another 12 h before sintering. Sintering induces hard, rigid, and porous texture to the membranes due to thermal and phase transformations. The supports were placed in a muffle furnace for sintering, and the temperature of the furnace was increased slowly to 250°C at a heating rate of 50°C/h and raised up to the sintering temperature (900°C) at a heating rate of 100°C/h. This temperature (900°C) was maintained for about four hours to complete the sintering process. The sintered

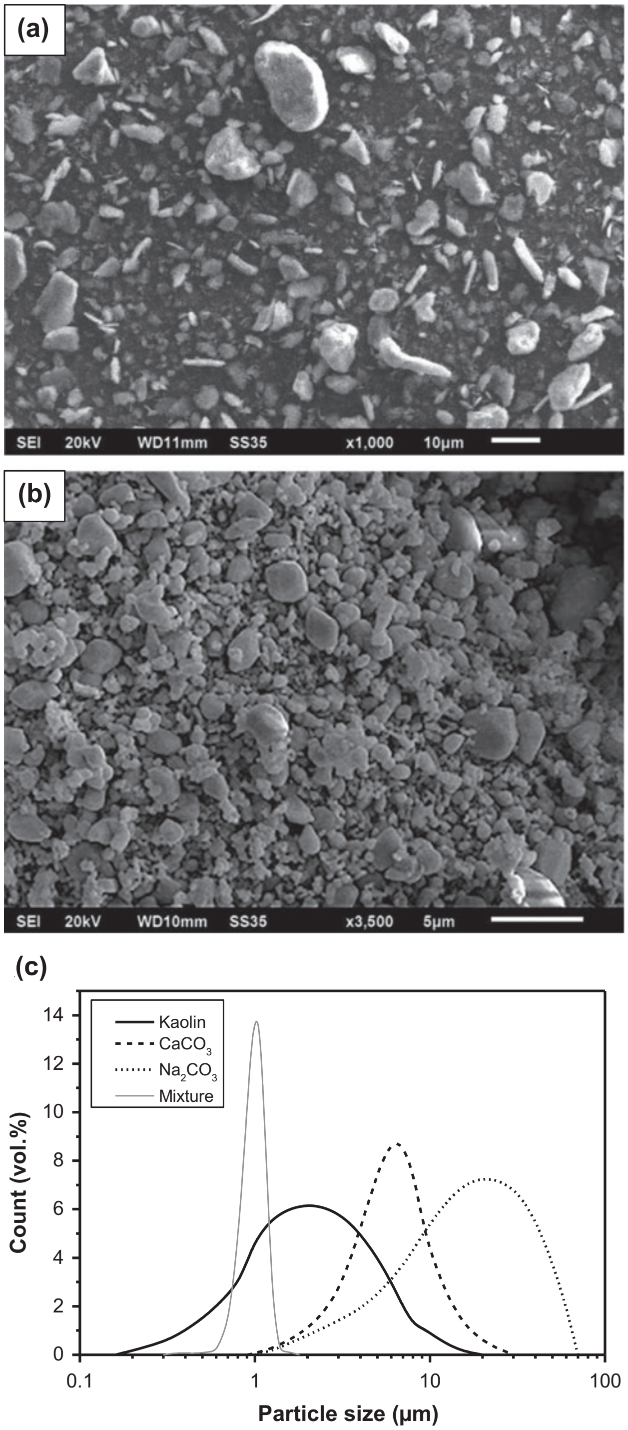

Fig. 1. (a) SEM image of kaolin; (b) SEM image of finely ground mixture; and (c) particle size distribution of raw material powders.

ceramic supports were polished on SiC abrasive papers (C-100 and C-220) and cleaned in an ultrasonic bath to remove loose particles (formed during the polishing step).

2.3. Characterization of ceramic supports

The inorganic mixture (raw materials paste) was subjected to TGA (EXSTAR TG/DTA 6300) by heating it from room temperature to 1,000˚C at a heating rate of 10˚C/min to identify various thermal transformations of the materials during the sintering process. XRD analysis of the ceramic supports was conducted on a diffractometer (X'Pert PRO, Panalytical) using Cu-K_α radiation at a wavelength of 1.54 Å to assess the extent of phase transformations.

SEM analysis (JSM-6610LV, JEOL) of sintered supports at various magnifications (500–10,000×) was carried out to study the surface morphology, identify the presence of possible defects, and evaluate the surface pore size distribution. The surface SEM images were taken at random locations of the ceramic supports, and the pores present in various SEM images of all supports were analyzed using the scientific image analysis program, ImageJ (version 1.46), to depict the surface pore size distribution [8]. For this purpose, more than 600 pores per sample were selected from images taken at different locations. The surface average pore size ($d_{\mathrm{p,s}}$) was calculated by area averaging of all pores as follows:

$$d_{\mathrm{p,s}} = \left(\frac{\sum n_i d_i^2}{\sum n_i}\right)^{0.5} \tag{1}$$

where d_i is the diameter of i^{th} pore and n_i is the number of pores of size d_i.

The porosity of the ceramic supports was determined by pycnometric method (also called gravimetric method) using water as the wetting medium [7] under ultrasonic conditions. It has been proven that porosity determination by this method under ultrasonic conditions [23] is as accurate as gas permeation (with little/negligible error). The difference between wet and dry weights of a membrane corresponds to its pore volume. The porosity of the ceramic supports was evaluated using the following formula (Eq. (2)).

$$\text{Porosity (\%)} = \frac{\text{Volume of pores}}{\text{Total volume}} \times 100 \tag{2}$$

All experiments were repeated for at least three times, and the standard deviation was found to be within ±5%.

3. Results and discussion

3.1. Physical observations

Preparation of defect-free ceramic supports using different compositions presented in Table 2 was

Table 1
EDAX of kaolin powder

Element	Weight (%)	Atomic (%)	Compound (%)	Formula
C (K)	0.35	0.58	1.27	CO_2
Al (K)	14.91	11.07	28.16	Al_2O_3
Si (K)	32.95	23.51	70.49	SiO_2
K (K)	0.07	0.03	0.08	K_2O
O	51.73	64.8	–	–

Table 2
Different raw material formulations used in the preparation of ceramic supports along with mean pore size obtained from surface SEM analysis

S. no.	Membrane label	Kaolin (wt.%)	$CaCO_3$ (wt.%)	Na_2CO_3 (wt.%)	Boric acid (wt.%)	Sodium metasilicate (wt.%)	Mean pore size (μm)
1	B0	95	–	–	2.5	2.5	0.916
2	B1	75	10	10	2.5	2.5	0.541
3	B2	65	20	10	2.5	2.5	0.606
4	B3	55	30	10	2.5	2.5	0.712
5	N1	85	–	10	2.5	2.5	0.529
6	N2	75	–	20	2.5	2.5	0.473
7	N3	65	–	30	2.5	2.5	–
8	N4	55	–	40	2.5	2.5	–
9	C1	85	10	–	2.5	2.5	0.597
10	C2	75	20	–	2.5	2.5	0.618
11	C3	65	30	–	2.5	2.5	0.765
12	C4	55	40	–	2.5	2.5	0.924

targeted in this work. All the ceramic supports prepared using different amounts of $CaCO_3$ were found to be defect-free. However, two of the compositions (N3 and N4) containing Na_2CO_3 resulted in nonporous (dense) membranes. It was physically observed that excess amount of sodium carbonate results in the formation of a dense glossy layer on the surface of the membranes, which could not be removed by polishing with the SiC abrasive papers. This is because of the formation of sodium silicate (also known as liquid glass) on the membrane surface, when molten sodium carbonate or sodium oxide reacts with silica. N3 membranes were found to stick loosely to the surface of the brick on which they were placed for sintering, while N4 membranes fused strongly to the brick surface. This might be due to the lower melting point of sodium carbonate (<850˚C) compared with that of calcium carbonate (1,340˚C). In addition, the membranes prepared using these two compositions were found to shrink in size by more than 20% v/v (approx.) and remained impermeable to air and water (tested using a laboratory permeation cell). Consequently, they (N3 and N4) can no longer serve as membranes due to their nonporous nature. Therefore, it is not recommended to use sodium carbonate in excess amounts (>20 wt.%) as it causes pore blockage.

The flexural strength of the supports was evaluated by three-point bending strength method (Roell Z010, Zwick) at a test speed of 10 mm/min, and chemical stability was checked by keeping the membranes in standard solutions of HCl (pH ≈ 1) and NaOH (pH ≈ 13) for 7 d. The prepared ceramic supports were found to have very good mechanical strength (30–55 MPa) and chemical stability (less than 5% weight loss under acidic as well as basic conditions), which are desirable for membrane applications. It has been shown in literature by Harabi et al. [24], who prepared porous ceramic membrane supports using kaolin and 15 wt.% of calcite, that flexural strength is a strong function of sintering temperature. They found that the flexural strength of supports increases (from 67 to 87 MPa) with increase the sintering temperature (from 1,100 to 1,250˚C). Though the supports sintered at 900˚C in the present work possessed lower flexural strength than those prepared by Harabi et al. [24], they can withstand the operating pressures of microfiltration (0.1–0.4 MPa), ultrafiltration (0.2–0.7 MPa), and nanofiltration (0.6–1.0 MPa) processes.

3.2. Thermo-gravimetric analysis (TGA)

The TGA was performed by heating the raw material paste in an α-alumina crucible from ambient temperature (30 ± 10˚C)–1,000˚C at the rate of 10˚C/min. Fig. 2 presents the TGA and DTA graphs of raw material paste for four different compositions (viz. B0, N1, C2, and B2). The weight loss of about 10–16% observed in all four samples below 125˚C temperature was due to evaporation of free (unbound) moisture (indicated by an endothermic peak at around 70˚C in the DTA graph), about 1% weight loss between 200 and 400˚C was due to dehydration of crystal water (bound moisture) of boric acid, and a weight loss of about 3–5% between 450 and 650˚C was due to the transformation (dehydroxylation) of kaolinite ($Al_2Si_2O_5(OH)_4$) to metakaolinite ($Al_2Si_2O_7$) along with other phase changes [24,25]. For samples containing $CaCO_3$, the weight loss (8–12%) observed in the temperature interval 650–750˚C was due to thermal decomposition (calcination) of calcium carbonate resulting in the release of CO_2 (indicated by an endothermic peak at around 720˚C in the DTA graph), whereas for the samples containing Na_2CO_3, the weight loss (1–2%) in the temperature interval 750–850˚C was due to the release of CO_2 resulting from the reaction of silica with molten sodium carbonate (indicated by an endothermic peak at around 810˚C in the DTA graph). Further, a sharp decrease in weight of the samples in the interval 650–750˚C than the interval 750–850˚C indicates that the rate of decomposition of $CaCO_3$ is faster than that of Na_2CO_3. No weight loss was observed beyond 850˚C (in the temperature range studied), and hence, the sintering temperature for fabrication of porous ceramic supports using the raw materials presented in Table 2 should be above 850˚C. Therefore, the sintering temperature was chosen to be 900˚C, which is well above the minimum (850˚C). The same (i.e. 900˚C as the optimum sintering temperature) has also been recommended by many previous studies [7,10–12,20,26] on the preparation of low-cost porous ceramic supports/membranes using calcium carbonate because the decomposition occurs far below 900 ˚C and the product (CaO) reacts with silica of kaolin.

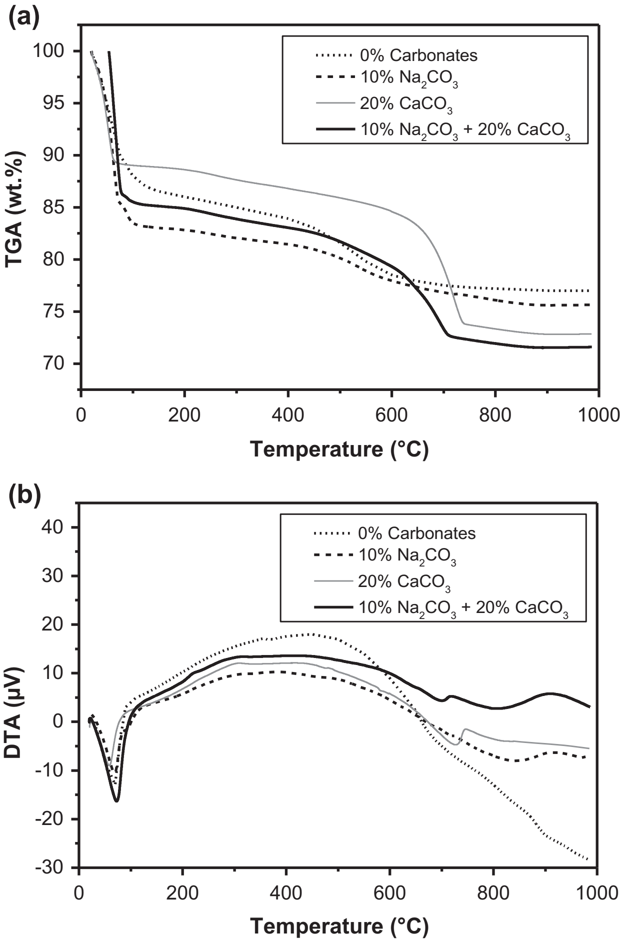

Fig. 2. Thermal analysis of raw material paste: (a) TGA and (b) DTA.

3.3. X-ray diffraction (XRD) analysis

Fig. 3 summarizes the XRD patterns of ceramic supports made of four different compositions (B0, N1, C2, and B2) and sintered at 900˚C for four hours. The XRD analysis conveys that quartz (SiO_2) and nephiline ($Na_2O{\cdot}Al_2O_3{\cdot}2SiO_2$) were present equally in all four compositions. Nephiline is produced by the reaction of sodium oxide (Na_2O) and metakaolinite ($Al_2Si_2O_7$) at a temperature of about 850˚C [7]. Metakaolinite was highly present in composition B0 as it contained the maximum amount of kaolin (95 wt.%) in comparison with other three compositions. Mullite ($3Al_2O_3{\cdot}2SiO_2$) phase was mainly present in supports prepared using sodium carbonate (i.e. compositions N1 and B2), whereas, illite ($K_{0.65}Al_2(Al_{0.65}Si_{3.35}O_{10})(OH)_2$) phase was present in the supports prepared using calcium carbonate (i.e. compositions C2 and B2). Inyoite ($Ca(H_4B_3O_7)(OH){\cdot}4H_2O$) was found to be present in trace amounts.

It can also be observed from Fig. 3 that anorthite ($CaAl_2Si_2O_8$) was present in the membranes prepared of calcium carbonate (C2 and B2), while albite ($NaAlSi_3O_8$) was present in the supports prepared of sodium carbonate (N1 and B2). Oligoclase ($Na_{0.8}Ca_{0.2}Al_{1.2}Si_{2.8}O_8$), an intermediate between albite and anorthite, was present in the membranes prepared using both carbonates (B2) and pyrophyllite ($Al_2Si_4O_{10}(OH)_2$) was found to exist in the membranes prepared without using any of the carbonates (B0).

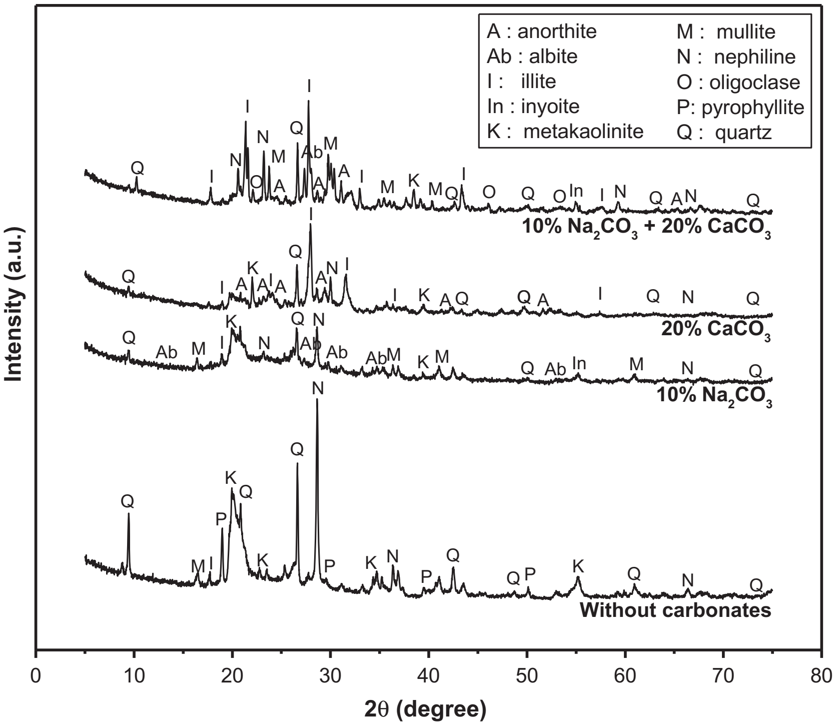

Fig. 3. XRD pattern of membranes sintered at 900˚C.

3.4. Scanning electron microscopy (SEM)

The surface SEM analysis was performed to determine the surface morphology and the surface pore size distribution of the membranes. Fig. 4 shows the surface SEM images (captured at a magnification of 2,000×) of four different membranes. It was observed that the prepared membranes were uniform and defect-free (i.e. no cracks were noticed). The figure also conveys that the supports prepared using no carbonates (a) or less amount of carbonates (b) are more homogeneous than others (c) and (d).

SEM images captured at a magnification of 10,000× or higher were used to study the surface pore size distribution of the membranes. Fig. 5 outlines a few of the pores identified on the surface SEM image of C2 membrane. About six to eight such images, each containing 100–150 pores (approx.), were analyzed to evaluate the pore size distribution of a sample. Estimation of pore size by this method has been proven to be as accurate as liquid permeation, if applied with utmost care [7,8,10,23]. Else, it may lead to errors (up to 10%) as the size distribution depends on the locations selected and may necessitate further analysis to validate the results.

Fig. 6 shows the surface pore size distribution of ceramic supports prepared using different compositions. From this figure, it is clear that the ceramic supports prepared using sodium carbonate have narrow pore size distribution in comparison with those prepared using calcium carbonate. This is probably due to slower reaction of molten sodium carbonate compared with faster decomposition of calcium carbonate, which is evident from the TGA analysis. The supports prepared using both carbonates also showed a wider pore size distribution as they contained more amount of $CaCO_3$ (10–30%) than Na_2CO_3 (10%). The pores noticed on the surface of membranes (B0) prepared without using carbonates were formed by the accumulation of fine and porous kaolin powders, as well as the release of H_2O during drying (<125˚C), dehydration (200–400˚C) and dehydroxylation (450–650˚C).

Further, no pores were observed on the surface of the supports N3 and N4. The average pore size values obtained from surface SEM analysis are presented in Table 2. The mean pore size decreased with the addition of 10–20 wt.% of carbonates and a further increase in the amount of carbonates caused an increase in the mean pore size. This observation indicates that small amounts of carbonates (10–20%) aid in the creation of new pores of smaller size, whereas, excess amount of carbonates (>20%) lead to overlapping of adjacent

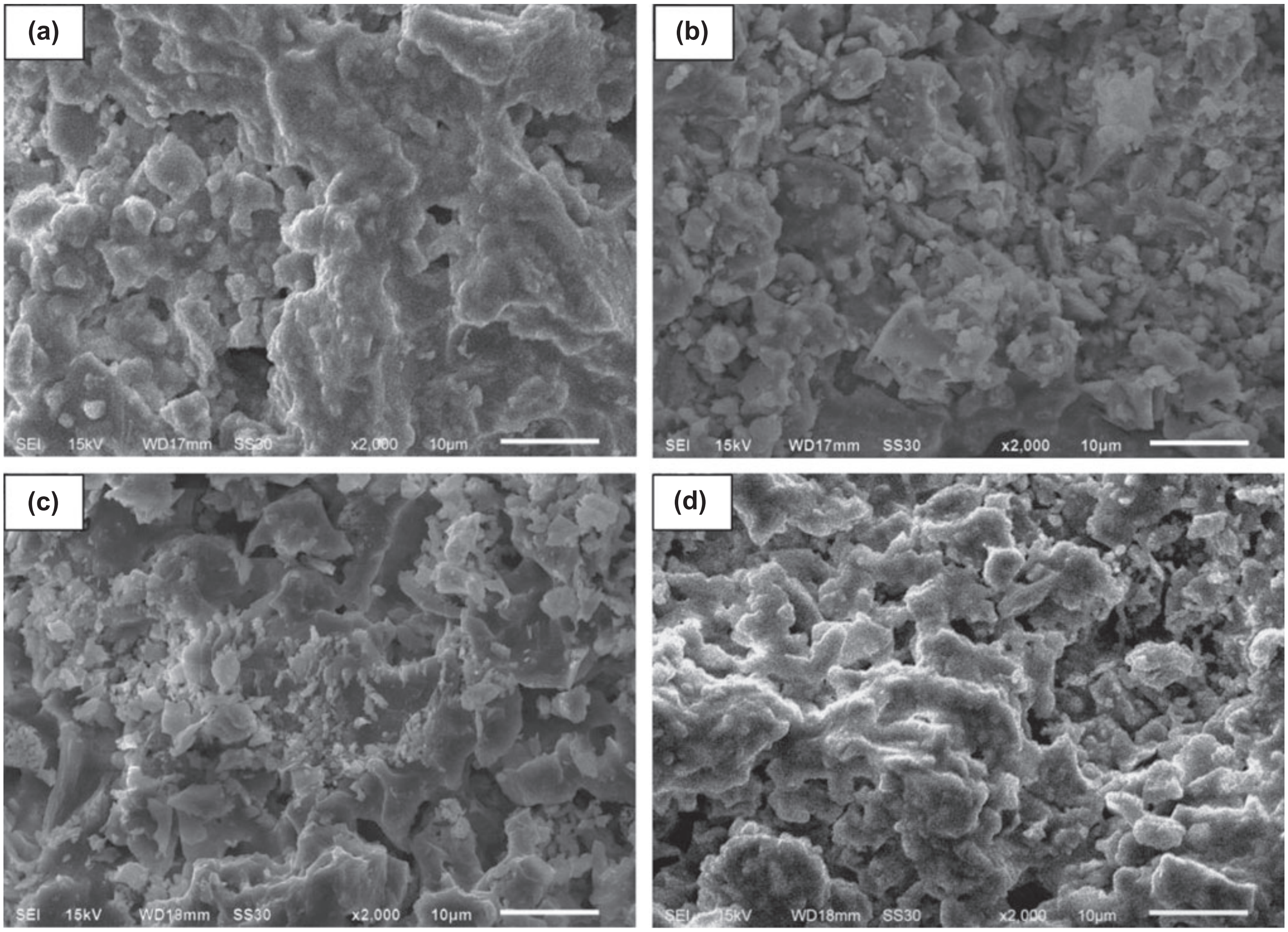

Fig. 4. Surface SEM images of membranes prepared using (a) 0% carbonates; (b) 10% Na_2CO_3; (c) 20% $CaCO_3$; and (d) 10% Na_2CO_3 + 20% $CaCO_3$.

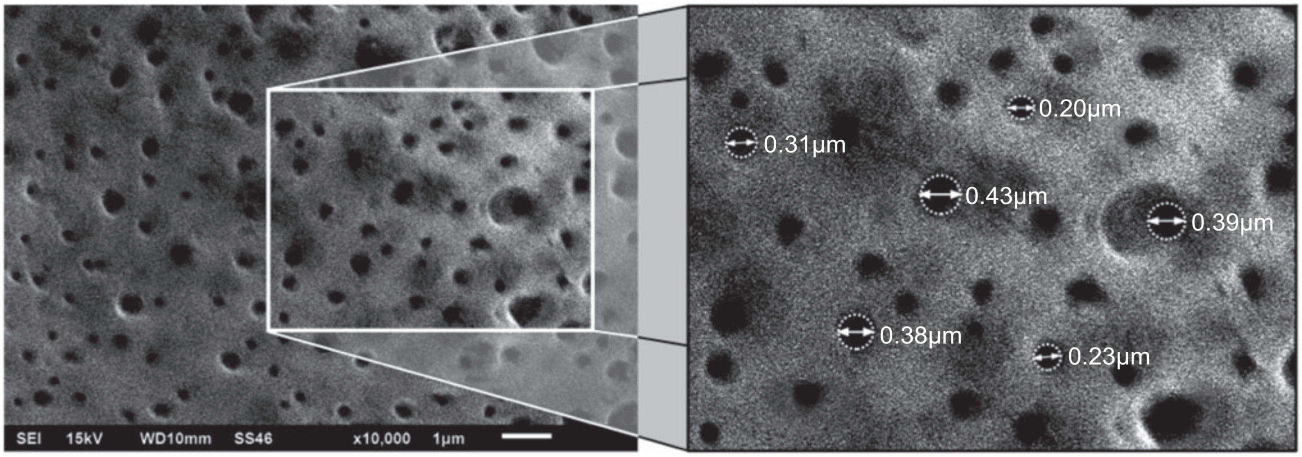

Fig. 5. SEM image of a membrane (C2) at high magnification (10,000×).

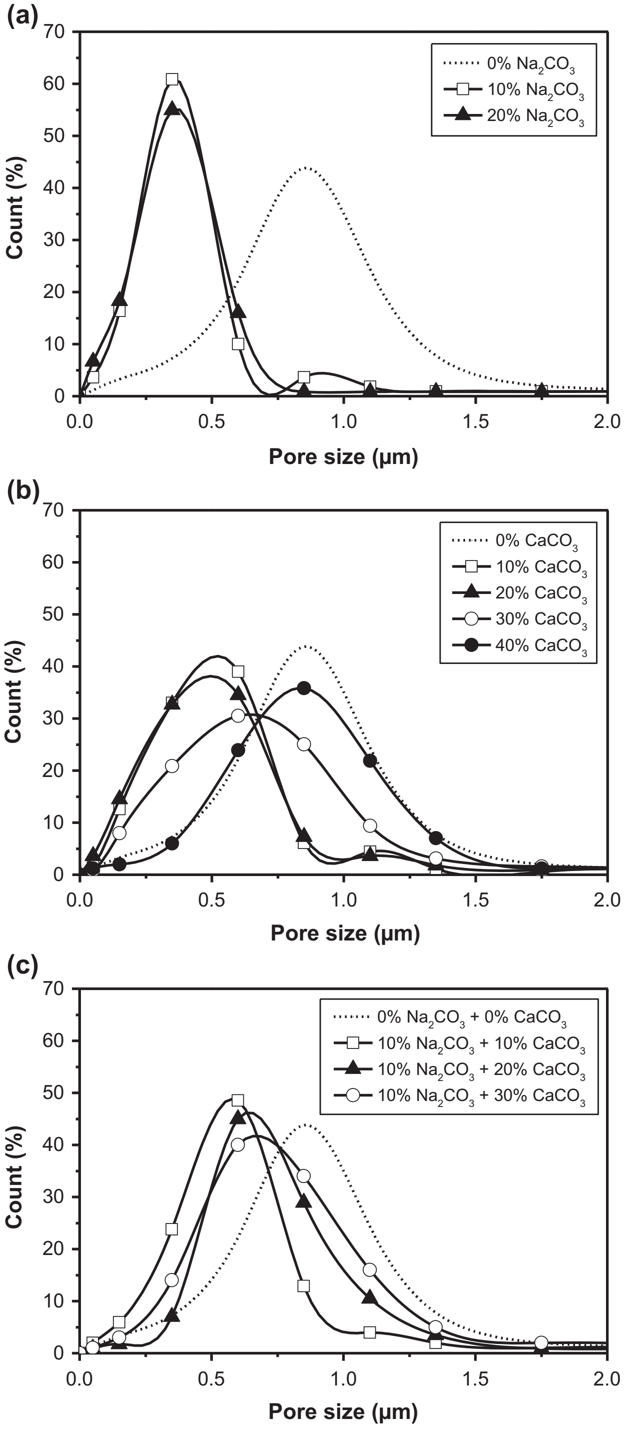

Fig. 6. Surface pore size distribution of membranes prepared using (a) Na_2CO_3; (b) $CaCO_3$; and (c) Both carbonates.

pores and/or creation of larger pores. Therefore, the optimum amount of $CaCO_3$ that should be used for membrane fabrication is 20 wt.%.

3.5. Porosity

The porosity values determined by pycnometric method are presented in Fig. 7. It can be observed from Fig. 7(a) that the porosity of the ceramic supports increased with increasing the amount of calcium carbonate. This is in accordance with the fact that carbonates impart porous texture to the membranes. During the sintering process, calcium carbonate dissociates into calcium oxide and carbon dioxide at 663–745℃, and sodium carbonate melts at temperatures above 800℃, reacts with silica of kaolin, and releases carbon dioxide. The vacant spaces created by the release of CO_2 lead to the formation of pores [15]. The ceramic supports prepared using $CaCO_3$ were more porous than those prepared using Na_2CO_3, and increasing Na_2CO_3 composition beyond 10 wt.% did not cause subsequent increase in porosity. In addition, the supports (N3 and N4) prepared using high amount of

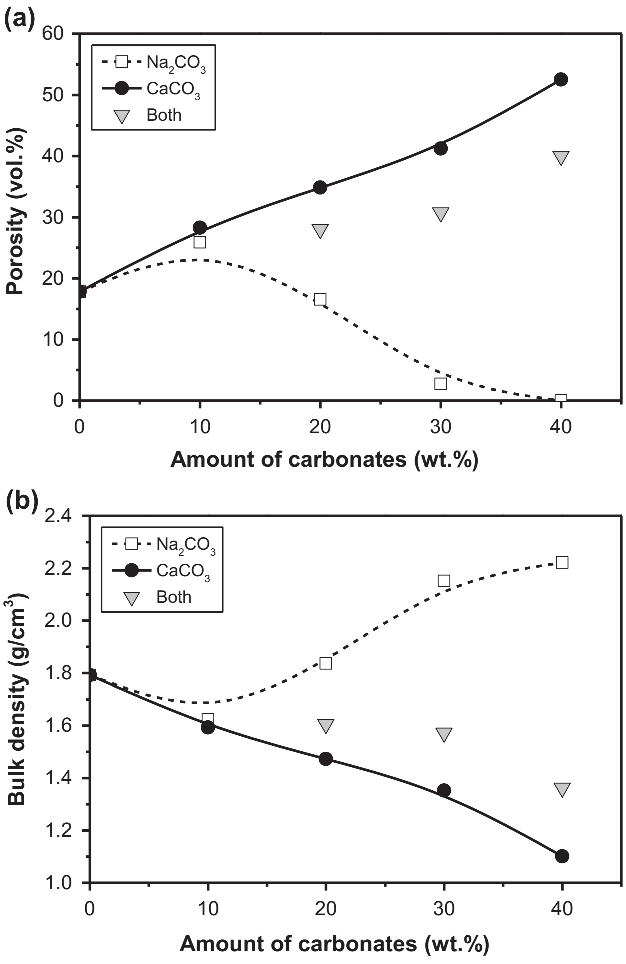

Fig. 7. Variation of (a) porosity and (b) bulk density with amount of carbonates.

sodium carbonate (30–40%) had negligible porosity. This might be because of the blockage of pores by sodium silicate resulting in size shrinkage. Therefore, the optimum amount of Na_2CO_3 that can be used for preparing porous ceramics is 10 wt.%. The variation of bulk density of supports (determined by gravimetric method) with the amount of carbonates is shown in Fig. 7(b). From this figure, it can be observed that the bulk density decreases with increasing the amount of total carbonates due to increased porosity (except N3 and N4). This is because of the fact that the pore volume is also accounted in the calculation of bulk density. The membrane containing no carbonates in its composition (B0) also exhibited a porosity of about 18%. Therefore, it should be noted that the release of H_2O also induces porosity to the membranes and this could be the reason for the observation of highly porous nature of membranes prepared using wet paste method compared with those prepared using uniaxial dry compaction method by Ghosh et al. [20].

Increasing the amount of Na_2CO_3 from 10 to 20 wt.% did not cause an increase in porosity (Fig. 7), but resulted in a slight decrease in mean pore size (Table 2). In addition, the supports prepared using both carbonates viz. B1 (10% $CaCO_3$ + 10% Na_2CO_3 = 20% total carbonates), B2 (30% total carbonates), and B3 (40% total carbonates), had lower mean pore size than those prepared using calcium carbonate alone, i.e. C2 (20% $CaCO_3$), C3 (30% $CaCO_3$), and C4 (40% $CaCO_3$). Also, the supports prepared using sodium carbonate had a narrow pore size distribution (Fig. 6) than others. Based on these observations, it can be inferred that sodium carbonate when used in small quantities serves as a pore modifier instead of acting as a pore-forming agent.

3.6. Cost comparison

Cost is a major factor that limits the applicability of inorganic membranes over polymeric membranes. Therefore, this work attempts to prepare low-cost ceramic membrane supports excluding expensive raw materials such as quartz, feldspar, and pyrophyllite that have been used in previous works [23,26]. The unit prices of different raw materials were taken from the retail prices quoted in the supplier's catalog, which are also available elsewhere [8]. Since the sintering conditions and preparation procedures were similar for these membranes, the total cost of raw materials makes the difference in the overall membrane cost. The material cost per unit area of the membranes (0.5–0.9 μm) prepared in this work (60–72 $/m^2) is only one fifth of that reported [23] for membranes with pore size 0.3 μm (352 $/m^2) and is less than half of that reported [26] for 0.7 μm membranes (150 $/m^2). This indicates that the ceramic supports prepared in this work can serve as a cheaper alternative to those prepared earlier using expensive materials viz. quartz, feldspar, and pyrophyllite.

4. Conclusions

The role of carbonates in the preparation of kaolin-based low-cost microfiltration membranes has been studied in this work. The prepared ceramic supports (except N3 and N4) exhibited very good permeation characteristics viz. pore size and porosity. The TGA and XRD analyses revealed the occurrence of complex phase transformations during sintering process. It was also noticed that the rate of reaction of sodium carbonate is slower than that of calcium carbonate resulting in narrow pore size distribution on the surface of the membranes. Use of mixed carbonates yielded intermediate results. The supports prepared using calcium carbonate were more porous than those prepared using sodium carbonate as the latter melts and forms a sodium silicate layer in the interior of pores as well as on the support surface under the sintering conditions. Therefore, calcium carbonate should be preferred over sodium carbonate as pore-forming agent in the preparation of inorganic membranes. The average pore size decreases with increasing the amount of carbonates from low to moderate quantities (0–20 wt.%) due to the creation of new pores of smaller size, while excess amount (>20 wt.%) of carbonates leads to overlapping of adjacent pores resulting in increased average pore size. Therefore, the optimum amount of calcium carbonate that can be used is 20 wt.%. Sodium carbonate should be used cautiously in low quantities (≤10%), and it serves well as a pore modifier than as a pore-forming agent. The ceramic supports prepared in this work using kaolin are cheaper than those reported in previous works.

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