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Development of chalk layered microfiltration ceramic membrane

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ABSTRACT

In the present investigation, a low-cost ceramic microfiltration membrane was fabricated by using inexpensive raw materials through paste casting technique. The membrane was cast in the shape of a circular disk of 73 mm diameter on a metal plate by using local river soil, sodium hydroxide, sodium nitrate, oxalic acid and chalk powder. Initially, the paste was prepared by mixing the precursor materials in a predetermined proportion with the help of 1 M and 3 M NaOH solution. The casting process was followed by sintering at 970 °C and subsequent activation at this temperature for 30 minutes. Then the prepared membrane was allowed to cool down to room temperature in order to provide strength. The porosity of the prepared membrane was determined by pure water permeability test and it was found to be 0.43 whereas the permeability of pure water was evaluated as 3×10^{68} m^3/m^2-kPa-s. The compressive strength of the prepared membrane was calculated as 2.32 MPa. The cost of the prepared membrane was estimated to be 13.64/membrane respectively. Therefore, in the present study, a low-cost ceramic membrane had been effectively developed which could be used to remove various water pollutants from wastewater.

Keywords: *Ceramic, Low cost, Paste casting, Porosity, Permeability.*

1. INTRODUCTION

In recent years, ceramic membranes are used in a broad spectrum of various fields which includes household as well as industrial applications. Ceramic membranes offer good thermal stability, mechanical strength, chemical resistance, long life, defouling properties and good removal efficiency [1, 2]. But widespread use of commercial ceramic membranes is restricted due to their high costs. Besides, ceramic membranes prepared from alumina powder as a base material is generally sintered at a high temperature of about 1500 °C for which compromise has to be made between mechanical strength and porosity of the membrane [3]. Therefore, fabrication of low-cost ceramic membranes has become a challenging tusk which will offer the same membrane characteristics like commercial membrane such as, high porosity, high permeability, good mechanical resistance etc. Membranes can be prepared from various materials but among all these membranes prepared from clay minerals have gained lots of importance as clay materials offer good structural rheological and thermal properties. Therefore, development of low-cost membranes from inexpensive clay materials has emerged as an effective alternative [4]. In the present study, a low-cost ceramic membrane was prepared from locally available river soil by mixing it with sodium nitrate and oxalic acid through paste casting technique.

2. MATERIALS AND METHODS

2.1 Casting of membrane

In the present research, the ceramic membrane was prepared through paste casting technique by using the following compositions as prescribed in Table 1. The precursor materials such as river clay, sodium nitrate, and oxalic acid were mixed with 1 M and 3 M NaOH solutions in order to form the initial paste. For fabrication of the outer skin layers, 15 g soil was mixed with sodium nitrate and oxalic acid at an impregnation ratio of 0.15 (w/w) by using 3 M NaOH solution. On the other hand, the inner layers of the membrane were prepared by mixing 20 g of soil with sodium nitrate and oxalic acid at an impregnation ratio of 0.1 (w/w) by using 1 M NaOH solution. The membrane was cast in five subsequent layers such as two skin layers and two middle layers with similar compositions. Finally, one layer of chalk powder was sandwiched between the middle layers of 20 g soil to enhance the membrane porosity. Once the paste was prepared it was then cast over a metallic surface in the shape of a circular disc of 73 mm diameter and 0.6 cm thickness and kept at room temperature for 12 h for partial drying and finally it was subjected to controlled heat treatment in a programmable muffle furnace (Electronics and Electrical Engineering Co., Kolkata, India).

Table 1: Compositions of membrane

Materials	Value
Clay (%)	79.97
Sodium carbonate (%)	1.67
Oxalic acid (%)	14.40
Commercial carbon (%)	NIL
Calcium carbonate	15.99

2.2 Heating profile of ceramic membrane

Eventually, the casted membrane was subjected to step by step heat treatment in a programmable muffle furnace (Electronics and Electrical Engineering Co., Kolkata, India) to reach the desired sintering temperature of 970 °C in 3.5 h and kept at this temperature for 30 min. Initially, the prepared membranes were carefully heated from room temperature to 200 °C at a heating rate of 3.33 °C/min for 1 h. It was then heated from 200 to 500 °C at a rate of 5 °C/min for next 1 hour and finally, it was heated from 500 to 970 °C at a rate of 5.2 °C/min for 1.5 h. Once the sintering temperature was reached, the membrane was kept at this temperature for 30 min for subsequent activation and to provide the final strength to the membranes. Then, the temperature of the muffle furnace was allowed to come down to room temperature. Finally, the membrane was rubbed with silicon carbide abrasive paper (C-180) to give a nice look and the final shape of 73 mm diameter and 0.6 cm thickness.

2.3 Determination of pure water flux and porosity of the prepared membrane

The water flux is determined as a function of time at different applied pressure varied from 196 – 392 kPa. The steady state permeability was attained over the experimental time. So the water permeability (L_P) of the membrane was determined from the slope of the graph between water flux and applied pressures by using the following equation

$$Jw = L_P \Delta P \quad (1)$$

Here, Hagen-Poiseuille equation was used to determine the average pore size of the membrane by considering the presence of cylindrical pores on the membrane surface [5].

$$J_w = \frac{\varepsilon r^2 \Delta P}{8 \mu \tau l} . \quad (2)$$

where ε is the porosity of the developed membranes
r is the pore radius of the membrane (m)
ΔP is the applied pressures across the membrane (196 – 392 kPa)
l is the pore length (0.015 m) which is generally taken as the thickness of the membrane
τ is the tortuosity factor (generally used as 1)
μ is the viscosity of water (0.00089 Pas).

By combining equation (1) and (2) the pore radius of the developed membranes were calculated as

$$r = \left[\frac{L_p 8 \mu \tau l}{\varepsilon} \right]^{1/2} \quad (3)$$

The porosity of the prepared membranes was calculated by using the following formulae [6]:

$$Porosity(\%) = \frac{W_{wet} - W_{dry}}{\rho_{water} \times V_{membrane}} \times 100 \quad (4)$$

Where W_{wet} is the wet weight of the membrane (placed in distilled water for overnight)
W_{dry} is the dry weight of the membrane (dried at 110 ◦C for 3 h)
$V_{membrane}$ is the total volume of the membrane
ρ_{water} is the density of the water.

3. RESULTS AND DISCUSSIONS

3.1 Determination of pure water flux of the prepared membrane

The water flux is determined as a function of time at different applied pressure varied from 196 – 392 kPa. The steady state permeability was attained over the experimental time. So the water permeability (L_P) of the membrane was determined from Darcy's law by using equation (1) and the water permeability was found to be 3×10^{-6} m/s-kPa. The similar kind of result was also obtained by Zhou et al., (2006) [7].

3.2 Determination of average pore size and porosity of the prepared membrane

Hagen-Poiseuille equation was used to determine the average pore size of the membrane and the by using this relation the pore radius of the membrane was calculated as 9.97 µm which lies in the range of microfiltration membrane. This value is comparable to the value obtained from the previous literature. [8, 9]. The porosity of the developed membranes was also determined by using equation (4) and the value was found to be 0.43. Finally, the overall membrane properties are summarized in Table 2. It can be speculated from Table 2 that the developed membranes have excellent membrane characteristic and hence it can be applied to various industrial applications.

Table-2: Properties of developed ceramic membrane

Parameter	Value
Membrane diameter (mm)	73
Average pore diameter (from H_2O permeability)	9.97 µm
Porosity	0.43
Water permeability (m^3/m^2-kPa-s)	3.0×10^{-6}
Compressive strength (MPa)	2.32

3.3 Cost estimation of membrane

In the present study, the cost of the developed ceramic membrane was determined on the basis of raw materials used and it is estimated to be Rs. 13.64/membrane as shown in Table 3. The raw materials cost of the membrane per unit area is estimated to be Rs. 2539.50/m^2 (38 \$/m^2) respectively. On the other hand, the cost of other commercial membranes prepared using α-alumina is found to be approximately \$500/m^2 whereas, the symmetric stainless steel membrane costs approximately \$3000/m^2 ([7]. Therefore, it can be said from the cost estimation that the prepared membrane is inexpensive as compared to commercial membranes, based on the sintering temperature and raw materials utilized in this study.

Table-3: Cost analysis of a single membrane

Raw Materials	Cost /500 g (INR)	Amount Required (g)	Cost of raw materials
Clay			
Oxalic Acid	Rs. 219.00	6	Rs. 2.63
Sodium Nitrate	Rs. 240.00	0.5	Rs. 0.24
Sodium Hydroxide	Rs. 314.00	11.68	Rs. 7.33
Chalk	Rs. 36.44	10	Rs. 0.72
Total cost for single membrane II			Rs. 10.92

4. CONCLUSION

In the present study, a microfiltration ceramic membrane was fabricated from inexpensive raw materials through paste casting technique. The pure water permeability was determined for the developed membrane and the value was found to be 3×10^{-6} m/s-kPa whereas the porosity was also calculated as 0.43. The pore sizes from the pure water permeability were evaluated as 9.97 µm which lie in the microfiltration region. The compressive strengths of the membranes were determined as 2.32 MPa for the casted membrane. Finally, the cost of the membrane was evaluated based on the cost of raw materials used and the cost was found to be Rs. 13.64/membrane which is quite inexpensive in compare to other commercial membranes. Therefore, it can be concluded that an inexpensive microfiltration membrane can be developed by using the above mentioned raw materials which can be further used for water purification purpose.

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