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Development and Characterization of Low-cost Ceramic Membrane

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ABSTRACT

Because of their better mechanical, chemical, and thermal properties, ceramic membranes are becoming more and more popular. They also have advantages over polymeric membranes. Many researchers have attempted to produce ceramic membranes by modifying their properties through various methods of production and raw materials. At present, a number of obstacles restrict ceramic membranes from being used widely, such as the expensive cost of raw materials and the high sintering temperature during synthesis. To overcome these limitations, low-cost raw materials like cenosphere and red mud were used for making ceramic membranes. Circular disk-type membranes were produced without the need of polymeric additives using a simple pressing technique. Throughout the synthesis process, the cenosphere and red mud compositions were changed to produce the composite membranes. The temperature range at which the membranes were sintered was 700 - 900 °C. The purpose of this research was to analyse the synthesized membrane through a variety of characterization techniques, such as thermogravimetric analysis (TGA), X-ray diffraction analysis (XRD), and scanning electron microscopy (SEM) etc. Also, each synthesized membrane's porosity has been examined and it was found that when the sintering temperature increased, the porosity decreased for a given composition. The cost of the produced membrane was found to be ₹1898.80/m^2.

Keywords: Cenosphere; Red mud; Membrane; Characterization; Cost.

1. INTRODUCTION

Global shortages of resources and environmental pollution steadily deteriorate as industrialization and economic growth pick up speed (Suttibak *et al.* 2008). Around the world, the pollution resulting from industrial solid waste from the metallurgical sectors is becoming a more significant issue. The production of different metals results in the annual generation of substantial amounts of metallurgical waste (Hou *et al.* 2017). As these residues contain soluble metals that cause a variety of problems with society, including contaminated water, they are regarded as hazardous wastes (Jabeen *et al.* 2013; Neiva *et al.* 2014). The use of industrial solid waste technology for sustainable development has received a lot of attention (Tang *et al.* 2014; Li *et al.* 2015). Solid wastes can be classified in general as red mud, cenosphere, and other categories (Hou *et al.* 2017; Qiu *et al.* 2018). As cenosphere has a high percentage of alumina and silica, it is a by-product of burning coal in thermal power plants. Recently, efforts have been made to use this by-product to prepare inexpensive ceramic membranes. In fact, due to the ensuing pollution, this permit efficient management of this by-product, which represents a major problem in many regions of the world (Jedidi *et al.* 2011).

A novel inorganic membrane material that has been attracting more attention recently is ceramic membranes. The most common materials utilized for producing these membranes are inorganic substances like SiO_2, TiO_2, ZrO_2, and Al_2O_3. Inorganic membranes provide a high degree of thermal and chemical stability and are repairable (Kaur *et al.* 2016). Ceramic membranes are used extensively in a variety of industries, including the food, pharmaceutical, and petroleum-chemical sectors. The importance of energy conservation has increased during the past 20 years. There are several steps in the industrial process. The purification or separation stage is one of them. In order to reduce overall energy usage at this level, membrane technology is needed. Membrane technology related to the development of novel ceramic membranes is important in a variety of applications (Singh *et al.* 2013). Researchers are dealing with the difficulty of developing novel ceramic membranes employing affordable materials and modern production techniques. Because of its economic competitiveness, low cost micro and ultra-filtration membrane technologies are crucial in industrial applications of ceramic membrane technology (Das *et al.* 2016).

This work used a variety of inorganic precursors and solid wastes to produce a low-cost sintered porous membrane. Producing a ceramic membrane with two different type of wastes as the raw material and an appropriate composition that could be sintered at low temperatures was the main objective of the current effort, which aimed to reduce the membrane's production costs. The synthesized membranes underwent a range of characterization processes, such as porosity and average pore diameter measurements, as well as techniques such as thermogravimetric analysis (TGA), X-ray diffraction (XRD), and Scanning electron microscopy (SEM).

2. EXPERIMENTAL SECTION

2.1 Materials and Methods

Red mud and cenosphere were used as the basic materials for this work. Other chemicals were used in addition to these main raw materials to produce the circular ceramic membrane. Energy dispersive x-ray analysis (EDX) was used to determine the chemical composition of the cenosphere raw material. The trace elements that were found in percentages are Fe (3.10), Ca (2.79), Ti (0.88), Mg (0.35), and so on. In terms of percentages, Si (21.65), Al (12.79), and O (57.90) are the major components. Wet chemical analysis was used to determine the red mud's chemical composition. Al_2O_3 (19.47), Fe_2O_3 (40.39), TiO_2 (15.73), SiO_2 (8.21), LOI (9.73), and Na_2O (5.1) are the elements, expressed as a percentage.

2.2. Synthesis of Membrane

Dry pressing was the primary technique used for making ceramic membrane supports. Using this procedure, a metal mould was filled with a certain quantity of binder, pore-forming agent, and raw material. All of the materials, including cenosphere, red mud and other chemicals were measured in weight percentage on a dry basis. To improve the inorganic chemical's dispersion characteristics and provide homogeneity in the membrane's structure, a chemical that increases strength was also added to the mixture. The synthesis process flow chart is shown in Fig. 1.

2.3 Membrane Characterization

Utilizing a scanning electron microscope with EDX (Jeol, JSM-IT300), the surface morphology of the synthesized membrane was determined. Utilizing an XRD analyzer (X-Pert Pro MPD, Netherlands), the different mineralogical phases for both raw materials used for the manufacture of membranes were analyzed using Cu Kα radiation (λ = 1.541 Å). The weight loss of the sample and the ideal sintering temperature can both be determined using thermogravimetric analysis, both of which are important. In this case, the analysis was carried out using an Exstar TG/DTA 6300 thermo-gravimetry analyzer.

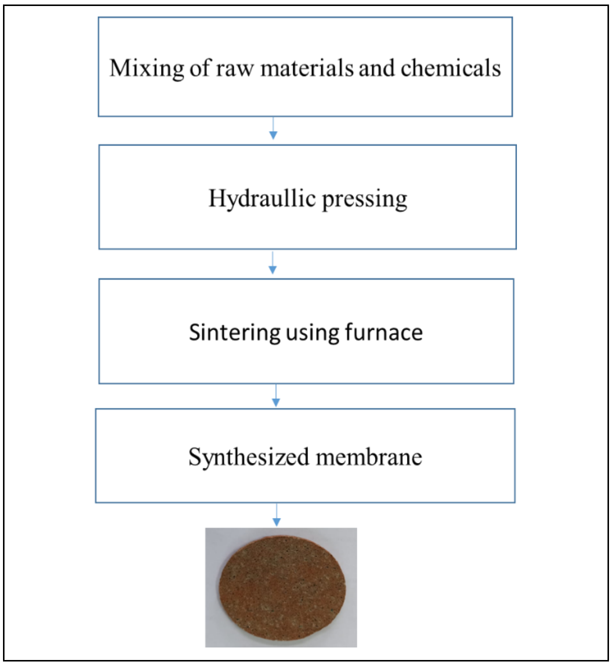

Fig.1: Synthesis process flow chart

2.4 Membrane Porosity

The membrane's void volume fraction is the main measure of membrane porosity. The gravimetric method was used to measure the manufactured membrane's porosity. The membranes were immersed in distilled water for a duration of one day. It was carried out using the Archimedes principle. A number of membranes were kept in an oven for an hour before the porosity test, and then they were turned off for the night. The thickness and diameter of the artificial ceramic membranes were measured using the Vernier calliper scale. These values were measured at different locations along the membrane, and the porosity was also computed by taking each membrane's volume into account, using the mean values. The prepared membrane measured 5 mm in thickness and 6 cm in diameter. The mean volume of the membrane was calculated using the thickness and radius of the membranes. Initially, the membrane was placed inside an oven that was preheated to an accurate temperature of about 110 °C before its dry weight (W_d) was measured. After that, these membranes were submerged in distilled water for a day. The new weight of the membrane, designated as (W_w), was then calculated after the surface water was removed. The porosity of the membrane was calculated using the following formula:

$$\varepsilon = \frac{W_w - W_d}{\rho_{water}} x \frac{100}{V_{mean}} \quad (1)$$

Where, ε is the porosity of membrane; W_w is the new weight of membrane after removing surface water (g), that is, wet weight; W_d is the initial weight of membrane after keeping inside the oven (g), that is, dry weight; ρ water is the density of water (kg/m^3); and $V_{membrane}$ is the volume of the membrane (m^3).

3. RESULT AND DISCUSSION

3.1 Structural Characterization

SEM, TGA, and XRD studies were employed to assess the structural characteristics of the membranes that were developed.

3.1.1 Thermogravimetric Analysis

The weight shift that takes place in a specimen as it is heated is monitored using TGA, an analytical technique used to assess a material's thermal stability. TG-DTG are typically used to see how a sample's mass changes with temperature. The TG-DTG curves that were captured during the analysis are shown in Fig. 2. The main objective of this thermal investigation was to determine the predominant weight loss and phase change of the produced membrane under a specific temperature regime. This study can be utilized to assess how the pore structure, pore width, and mechanical strength of ceramic membranes are affected by varying temperature regions. The synthesized membrane exhibits an overall mass loss percentage of around 1.28% at the sintering temperature of 800 °C. This analysis is useful to find the minimum sintering temperature (Singh *et al.* 2013).

3.1.2 XRD analysis

Different phases for both raw materials, cenosphere and red mud were found by XRD studies using x-ray database (XDB) software and the XDB reference card. The diffractogram phases of two raw materials, namely cenosphere and red mud, are displayed in Fig. 3 (a) and Fig. 3 (b). In Fig. 3 (a), mullite, hematite and quartz are the different phases in cenosphere raw material. The phases sodalite, alumogoethite, hematite, quartz, anatase, and rutile are found in the red mud shown in Fig. 3 (b).

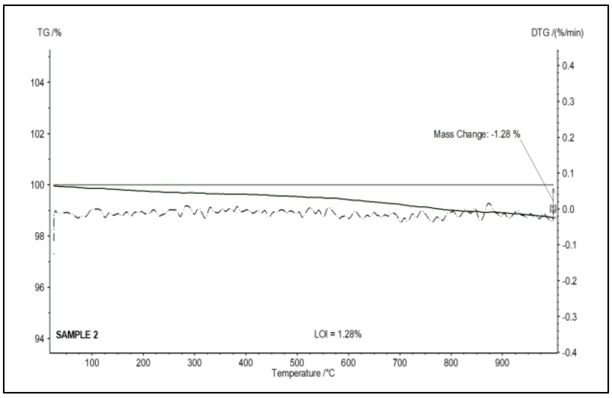

Fig. 2: TG-DTG of the sintered ceramic membrane (800 °C, Red mud: Cenosphere – 0.428)

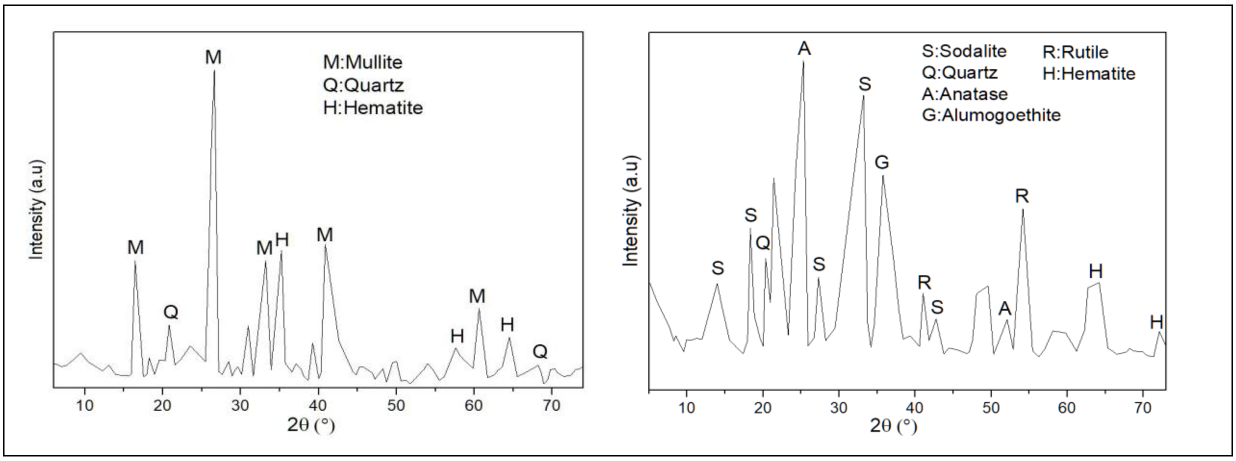

Fig. 3: XRD of (a) Cenosphere (b) Red mud

3.1.3 SEM Analysis

The produced cenosphere-red mud ceramic membranes were subjected to SEM analysis. The average pore diameter of each of the synthesized membranes was determined from the corresponding photographs. From the images it is seen that there are no cracks on the membrane surface (Rawat *et al.* 2018; Pradhan *et al.* 2024). SEM images of synthesized membranes at two different compositions are represented in Fig. 4 (a) and Fig. 4 (b). The average pore diameter of the membrane was 738 nm when the composition ratio of red mud to cenosphere was 0.33. The average membrane pore diameter, roughly 178 nm, reduced as the composition ratio of red mud to cenosphere increased from 0.33 to 0.428. A higher degree of densification during sintering may occur from an increase in red mud composition. This would then cause the overall pore size to decrease and the packing density to increase, resulting in a lower average pore diameter.

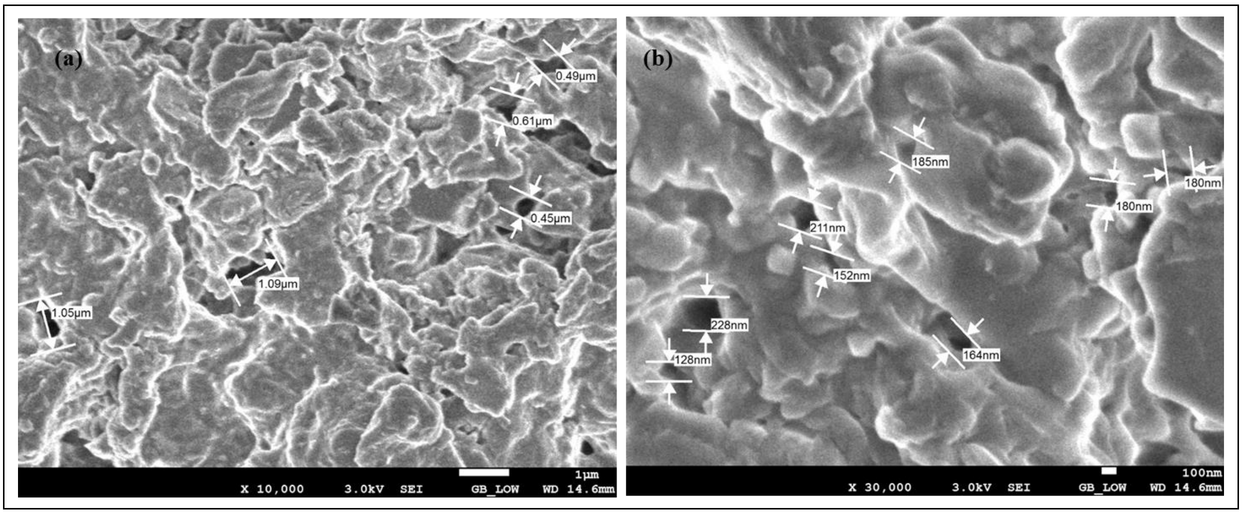

Fig. 4: Scanning electron microscopy (SEM) micrographs of 800 °C sintered ceramic membrane (a) Redmud: Cenosphere-0.33 (b) Redmud: Cenosphere-0.428)

3.2 Porosity

The properties of the membrane are affected by the sintering temperature. The relationship between membrane porosity and membrane sintering temperature is shown in Table 1. At high temperatures, the synthesized membrane exhibited low porosity, but at low temperatures, it exhibited high porosity. Table 1 clearly shows the correlation between the porosity and the sintering temperature. It also shows the relationship between the porosity and the composition ratio of red mud to cenosphere. Changes in composition can affect the mechanism by which pores are formed during membrane synthesis. Higher porosity can result from an increase in some components, which promote the formation of pores through the emission of gases or other volatile species. Higher amounts of iron in the red mud during this synthesis may cause the membrane's porosity to increase.

Table 1. Porosity of the synthesized membranes

Temperature (°C)	Composition ratio (Red mud: Cenosphere)	Porosity %
700	0.33	22.89
700	0.43	50.14
800	0.33	21.04
800	0.43	41.66
900	0.33	13.53
900	0.43	28.63

4. CONCLUSION

In this study, cost-effective basic raw materials, cenosphere and red mud, were formed into circular disc-type cenosphere ceramic membranes by uniaxial pressing, and these membranes were subsequently analysed. A range of sintering temperatures, including 700, 800, and 900 °C, were used during the synthesis. Porosity of the synthesized membrane was impacted by the sintering temperature. When the red mud to cenosphere composition ratio was 0.33, the porosity was 22.89% at 700 °C sintering temperature. This dropped to 21.04% at 800 °C, and a further increase in temperature to 900 °C resulted in a porosity of 13.53%.In a similar way, higher porosity was seen at all sintering temperatures when the composition ratio increased from 0.33 to 0.43.The average pore diameter, as determined by the scanning electron microscope, was about 178 nm at a composition ratio of 0.43 and 738 nm when the red mud: cenosphere ratio was 0.33.It was shown from the thermogravimetric study that the produced membrane had a very little mass change– roughly 1.28%.Using XRD analysis, distinct phases of the two basic raw materials were determined. The total estimated cost, which accounts for the cost of raw materials as well as the cost incurred during the sintering process, was found to be ₹5.36 for the circular synthesized ceramic membrane with a diameter of 6 cm. The cost per square meter of the membrane was determined to be around ₹1897.31, in comparison to other ceramic membranes that are available in the market.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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