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Research paper

Physico-mechanical and gas permeability characteristics of kaolin based ceramic membranes prepared with a new pore-forming agent

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ABSTRACT

The objective of this study was to prepare low-cost macroporous ceramic membranes using natural kaolin obtained from deposits in Nigeria and Ghana, and powdery high density polyethelene (PHDPE) as porogen agent. The ceramic membranes have been prepared with porogen content wt% between (0 and 20)% by die pressing. Pellets were fired at 1150 °C and soaking time of 4 h. The raw materials were characterized using TG/DTA, particle size distribution (PSD) and Zeta potential. The membranes cast as circular disks were subjected for characterization studies using XRD and SEM analysis. In a bid to correlate the physico-mechanical properties vis-à-vis pore former content, the effect of the sintering temperature and pore former (PHDPE) content on porosity, density, water absorption and mechanical strength were evaluated. The membrane corrosion resistance was found to be unaltered with experimental conditions. Obtained membranes showed good porosity with maximum at about 62% with a mechanical strength that does not exceed 18 MPa. These membranes can be considered as efficient regarding the results shown in the gas permeation tests at different sintering temperatures. A PHDPE percentage of 20% and a sintering temperature of 1150 °C were chosen as the optimum for gas permeation based on enlarged pore diameter of sintered membranes.

1. Introduction

Ceramic membranes are more favourable than their polymeric counterparts, especially in gas applications, due to its high thermal stability, good chemical compatibility and exceptional flexion and compressive strengths (Kingsbury and Li, 2009; Wang and Lai, 2012). By virtue of having such characteristics that require no maintenance, the production of ceramic supports has gained attention widely among researchers. Also, ceramic membranes which can be utilized at relatively higher temperatures are finding increasing relevance, because it is more realistic to carry out many chemical reactions and separation at elevated temperatures (Mizukami et al., 1992; Xu and Anderson, 1993; Church et al., 1994; Lin et al., 1994; LeDuc et al., 1996). However, because ceramic membranes have higher cost than polymeric counterparts, their applications have been retarded in some traditional industries such as food, beverage, and pharmaceutical. Also, limited types of membrane materials (such as Al_2O_3, ZrO_2, TiO_2, and their composite oxides) hinder their further applications (Pandey and Chauhan, 2001; Khemakhem et al., 2009; Hamad et al., 2013). In light of this, it is imperative to find more low-cost alternatives. A significant effort has been provided in the field of membrane technology in order to find new low-cost ceramic materials (Li et al., 2001; Benito et al., 2007). Recent research in the fabrication of inorganic membranes is focused towards the utilization of cheaper raw materials such as apatite powder (Masmoudia et al., 2007), fly ash (Saffaj et al., 2004a, 2004b), natural raw clay (Saffaj et al., 2005, 2006), dolomite, kaolin (Almandoz et al., 2004; Bouzerara et al., 2006). Of these inorganic precursors, research is devoted to other raw materials that would be of low cost and suitable as a standard raw material replacement. Clays have been identified as the best cost-effective raw materials for membrane applications (Weir et al., 2001; Saffaj et al., 2004a, 2004b; Khemakhem et al., 2006). Precisely, kaolin appears to be an important inexpensive raw material that can be studied for the fabrication of durable micro-filtration range inorganic membranes at a lesser cost. In addition, kaolin is also one of the cheapest and the most abundant support raw material and it is easily obtained.

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Porosity is known to be an important characteristic as far as ceramic membranes for industrial applications are concerned. One of the basic methods to produce porous ceramics membranes is by using pore-forming agents (PFAs) (Liu, 1996; Rice, 1998; Rice, 2003; Scheffler and Colombo, 2006; Studart et al., 2006). Porous ceramics are nowadays being investigated for a variety of applications due to their specific properties like high surface area, high temperature stability, high permeability, low weight and low thermal conductivity.

Characterization of particle dispersion is important to optimize and control, for example, ceramic forming processes, etc. (Somasundran et al., 1997). Two very significant characteristics of aqueous dispersions are particle size distribution (PSD) and zeta potential (ξ-potential). The effect of the particle charge of clay minerals on the aggregation behaviour, sedimentation or filtration is the main object of several researches (Alkan et al., 2005a,b; Kosmulski, 2003; Hang et al., 2007).

A wide variety of natural (i.e. biogenic) and carbon based pore-forming agents (also called sacrificial templates or fugitive materials) have been tested or used so far, including saw dust (Saggio-Woyansky et al., 1992; Sheppard, 1992; Liu, 1996; Rice, 1998; Ducman and Kopar, 2001; Scheffler, 2005; Studart et al., 2006), wheat particles (Prabhakaran et al., 2007a, 2007b), carbon fibers (Isobe et al., 2007), cotton (Zhang et al., 2001), peas and seeds (Luyten et al., 2003), including poppy seed (Gregorová and Pabst, 2007).

A recent study (Obada et al., 2016a, 2016b), the potentials of fabricating porous ceramics bodies from kaolin clay mined from deposits in Nigeria and Ghana using different pore formers was investigated. Therefore, the objective of this study was to fabricate ceramic membranes from kaolin clay/powdery high density polyethylene mixture with varying ratios by weight to prepare macroporous membranes to improve the potential competitiveness of the low-cost ceramic membranes. The physical and mechanical properties of the kaolin based ceramic membranes and gas permeability test results are also presented.

2. Materials and methods

2.1. Processing of raw materials

The raw materials used in this study are kaolin and powdery high density polyethylene. The kaolin was obtained from Kankara and Kibi deposits in Nigeria and Ghana respectively. The powdery high density polyethylene powder was processed at the Materials Laboratory of the University of Ghana. The kaolin was beneficiated as reported in our previous studies (Obada et al., 2016a). Moreover, the raw materials used in this work serve for different functional attributes. The kaolin was the base material while the powdery high density polyethelene (PHDPE) serves as a pore-forming agent.

The kaolin clay was mixed with the powdery high density polyethylene in four different ratios (clay-to-pore formers) by weight (Table 1). The XRF and XRD of the kaolin clay used for membrane fabrication in this present study have been presented in our previous works (Obada et al., 2016a, 2016b). It was observed that the composition of the kaolin consists of kaolinite, illite, mica and montmorillonite.

Table 1
Composition of test samples by weight (total weight = 100 g).

Sample code	Kaolin (g)	Plasticizer (Kibi kaolin) (g)	High density polyethylene (HDPE) (g)
0% CS	80	20	–
5% HDPE	75	20	5
10% HDPE	70	20	10
15% HDPE	65	20	15
20% HDPE	60	20	20

2.2. Materials characterization

2.2.1. Thermal analysis

In order to know the thermal behaviour of the kaolin and more information about its composition, a thermogravimetric analysis has been performed between 100 and 700 °C. This was studied by TG/DTA/DTG under air flow of 50 ml/min. The samples were thermally analysed by placing ≈ 25 mg of the specimens in an alumina (Al_2O_3) crucible (100 mg capacity), subjected to a linear heating ramp between 100 °C and 700 °C at a rate of 20 °C/min in NETZSCH, STA 449C Jupiter TG/DTA instrument. The test measurements were made for the mass change (loss) of the sample as a function of the temperature and the phase changes by the adsorption or the emission of energy.

2.2.2. Particle size distribution (PSD) and zeta potential

The essential properties of the raw materials (kaolin and powdery HDPE) such as particle size ranges were experimentally determined in a wet dispersion mode. A laser method, low-angle laser light scattering (LALLS), was used for the particle size analysis with levels of sensitivity in the 0.3 nm to 8000 nm range using a Horiba Scientific, SZ-100 nanoparticle analyzer. 10 mg of the kaolin samples and pore formers which were prepared using an analytical mill to reduce the sizes, were dispersed in freshly deionized water and subjected to continuous ultrasound treatment in an ultrasonic bath for 30 min to ensure dilution and homogenous dispersion. The zeta potential of dispersed kaolin was also performed on the same equipment as discussed for PSD. After 15 min ultrasonication of the suspension, the zeta potential was measured as a function of pH by titration with HCL and NaOH.

2.3. Membrane fabrication

The raw materials as listed in Table 1 were mixed in a ball mill at 50 rpm for 30 min. The resulting powdery mix was then compacted in a hydraulic pressing machine at a pressure of 3.5 MPa with the help of stainless steel die. The membranes were shaped as disks of 5 cm in diameter and 0.5 cm thicknesses. The obtained circular shaped membranes were first dried at ambient temperature before further drying at 150 °C for 24 h in a hot air oven. This was to remove moisture and also reduce any thermal stress which may hamper the successful fabrication of the ceramic membranes. Subsequently, the membranes were sintered at 1150 °C for 4 h in a muffle furnace at 5 °C/min. The sintering process was run in two steps; firstly, sintering temperature was setup at 500 °C at a rate of 2 °C/min and held for 2 h so that the pore former (PHDPE) would be burnt off. Then, the sintering temperature was increased up to 1150 °C at a rate of 5 °C/min and held for 4 h to produce ceramic membranes for the physical and mechanical tests, and up to 850 °C, 1000 °C, 1150 °C at a rate of 5 °C/min and held for 4 h to produce ceramic membranes for the gas permeation tests.Finally, temperature was then reduced to room temperature at a rate of 5 °C/min. After sintering, the compact and porous ceramic membranes were polished on both sides using silicon carbide abrasive paper to obtain ceramic membranes with uniform surface and considerably free from defects. These membranes were washed with deionized water in an ultrasonic for 15 min to remove the loose particles adhered on the surface of membranes.

For the mechanical properties testing of the membrane formulations, the kaolin and pore former mixture was completely filled into a rectangular mold and pressed to the same level to ensure compactness and dimensional homogeneity using a hydraulic pressing machine with pressure corresponding to 3.5 Mpa. The as-prepared green body samples were removed from the mold and dried at room temperature on boards for three (3) days after which they were further dried at 150 °C for 24 h in a hot air oven to remove moisture. The dried batch-formulated green bodies were then sintered at 1150 °C for 4 h in an electric muffle furnace at a heating rate of 5 °C/min after the systematic pore former oxidation process which was set at 500 °C at a rate of 2 °C/min and held for 2 h.