ARTICLE IN PRESS

Available online at www.sciencedirect.com

ScienceDirect

Ceramics International ▮ (▮▮▮▮) ▮▮▮–▮▮▮

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Development of a low cost micro-porous ceramic membrane from kaolin and Alumina, using the lignite as porogen agent

I. Hedfi[a,*], N. Hamdi[a], M.A. Rodriguez[b], E. Srasra[a]

[a]Centre National des Recherchesen Science des Matériaux, BorjCedria Techno-Park, B.P. 95-2050, HammamLif, Tunis, Tunisia
[b]Instituto de ceramica y vidrio, CSIC, c/kelsen, 2849 Madrid, Spain

Received 24 October 2015; received in revised form 16 November 2015; accepted 2 December 2015

Abstract

The manufacturing of low cost ceramic flat and tubular membranes via dry pressing and extrusion, using low cost material, namely, natural kaolin, Alumina, and lignite as a pore forming agent was the purpose in this study. These membranes are designed to be used as a support for multilayer ceramic membranes.

This study includes the preparation and the characterization of the different membranes.

The selected composition was 20% of lignite, 15% of Alumina and 65% of Kaolin, the membranes have been obtained at 1200 °C as a sintering temperature. The membranes show good behavior for both configurations, with a porosity above 36% and a mechanical strength of 39 MPa or the tubular membranes and 34% for the flat ones.

Keywords: E. Membrane; Kaolin; Lignite; Processing; Permeability

1. Introduction

The use of porous ceramic membranes has widely increased, in recent years; to gain importance in chemical technology they have been used in a wide range of applications [1–6]. This fact is due to their thermal, chemical and mechanical resistance, and environment friendly nature [7–9]. The microstructure of these membranes is controllable, thanks to the use of different types of pore forming agents [10,11]. Researches focused on the preparation of different types of membrane study, the influence of the type of the inorganic precursors on the morphology, stability and porous texture of the inorganic matrix.

Early research in inorganic membrane fabrication is focused on the utilization of α-alumina to fabricate the membrane [12,13] but it keeps an expensive precursor. In order to resolve this problem, more advanced research initiates the utilization of inorganic materials such as γ-alumina, zirconia, titania and silica [14–16]. But even using these materials, the cost remains to be significantly high. To resolve the issue of the cost, recent research is orientated to the use of cheaper raw materials such as fly ash [17,18], apatite powder [19], natural raw clay [20], dolomite, kaolin [21–25]. Of these inorganic materials, kaolin associated with an appropriate pore forming agent appears to be an important raw material that can be used for the fabrication of stable micro-filtration range inorganic membranes at a lower cost. It has been demonstrated in previous studies that the mechanical strength needs to be revised if an industrial use is targeted [26,27]. The objective of this study is to resolve such a problem and to prepare resistant microfiltration, flat and tubular, ceramic supports using natural kaolin, lignite as a pore forming, and a γ-alumina to improve the mechanical properties and chemical resistance of the support.

2. Experimental procedure

2.1. Raw materials

The raw materials used are kaolin, lignite and alumina.

The kaolin and Lignite are extracted from the region of Tabarka (North West of Tunisia). In order to determine their

*Corresponding author. Tel.: +34 603527304.
E-mail address: imen_hedfi@yahoo.fr (I. Hedfi).

http://dx.doi.org/10.1016/j.ceramint.2015.12.023
0272-8842/

composition, thermal behavior, and morphology, a characterization was carried out [25,26]. It was also necessary to determine the plasticity of the clay and of the selected mixture in order to know if the mixture can be extruded or not.

This study of the plasticity has been carried out by the Casagrande method, using the Atterberg limits, defining a range of percentage of water in which the mixture is plastic. The liquid limit of a soil is the moisture content, expressed as a percentage of the weight of the oven-dried soil, at the boundary between the liquid and plastic states of consistency. The moisture content at this boundary is arbitrarily defined as the water content at which two halves of a soil cake will flow together, for a distance of ½ in. (12.7 mm) along the bottom of a groove of standard dimensions separating the two halves, when the cup of a standard liquid limit apparatus is dropped 25 times from a height of 0.3937 in. (10 mm) at the rate of two drops/s [27]. The determination of the liquid limit and the plastic limit of the mixture has been defined using the procedure described by the Spanish standards UNE 103-103-94 [28] and UNE 103-103-93, respectively [29].

2.2. Shape forming

The preparation of the flat supports was carried out by pressing the clay powder at the chosen pressure using an electro-mechanical pressing machine with pressure control (Tonindustrie GmbH, Germany). The tubular membranes were prepared by extrusion using a pneumatic extruder made on the laboratory of the ICV and shown in Fig. 1.

2.3. Characterization

The characterization of the supports includes the evaluation of the porosity using a mercury porosimeter Auto pore II 9215 (Micromeritics, USA), the measurement of the density geometrically, and the evaluation of mechanical strength through compressive test for flat membranes and diametric compression test for tubular ones, using a universal testing machine with a cell of 5000 N (Instron, UK). The theory and formulas of calculations are described elsewhere [31,32].

The microstructure was studied using a Field Emission Scanning Electron Microscopy S-4700 (Hitachi, Japan). Both gas and water permeability of the porous samples has been carried out using respectively N2 as permeate with a system described elsewhere [30] and water permeability system described in Fig. 2.

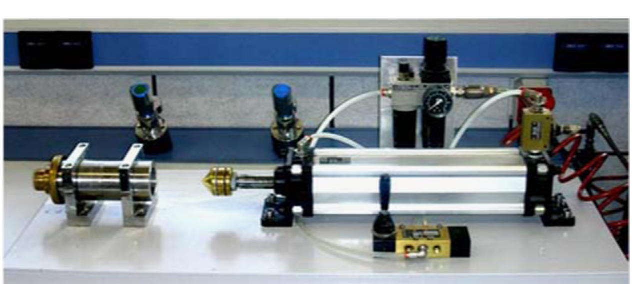

Fig. 1. Pneumatic extruder used for the preparation of tubular supports.

3. Results and discussion

The main characteristics are shown in Table 1 [25,26], including values of particle size, specific surface. Kaolin and Lignite present Quartz crystallographic contaminant. The transformation gap of temperature is defined as the temperature where the Kaolin shows main structural transformations by DTA–TG previous studies [25]. The pressing pressure also has been defined previously.

After analyzing the plasticity diagram of the clay (Fig. 3), it can be seen that it is located into the extrudability window. So, it can be considered that the kaolin could be shaped by extrusion due to its plastic character [33].

In order to define the best composition for porous and resistant membrane and, as continuity of previous researches [25,26], different mixtures have been prepared (Table 2). The compositions were selected on trial basis to get the best results in terms of microstructural considerations, mechanical stability and processing cost.

3.1. Pressed samples

Cylinders of around 25 mm in diameter and 5 mm in thickness were prepared by pressing at 23 MPa. They were sintered at three different temperatures: 1100 °C, 1150 °C and

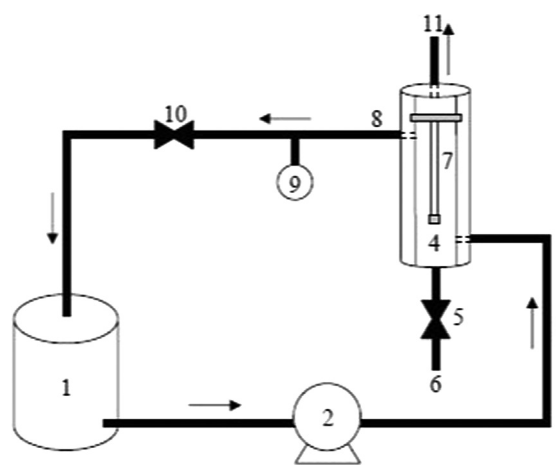

Fig. 2. Water permeability system, (1) feed, (2) pressure pump, (3) feed line, (4) membrane module, (5) valve, (6) purge, (7) membrane, (8) recirculation line, (9) pressure transducer, (10) valve, and (11) permeation flux line.

Table 1
Raw material main characteristics [25,26].

Materials	Particle size (μm)	Specific surface (m^2/g)	Transformation zone (DTA–TGA)	Pressing pressure (MPa)
Kaolin	3	18	880–1100 (°C)	23
Lignite	1	18	–	–
Alumina	5–8	20	–	–

Please cite this article as: I. Hedfi, et al., Development of a low cost micro-porous ceramic membrane from kaolin and Alumina, using the lignite as porogen agent, Ceramics International (2015), http://dx.doi.org/10.1016/j.ceramint.2015.12.023