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Influence of starch content on the properties of low cost microfiltration membranes

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ABSTRACT

This study is related to the development of low cost microfiltration (MF) membranes using cheap raw materials, namely clay and corn starch. Flat MF membranes were prepared by uniaxial dry pressing and sintered at firing temperature of 950 °C. Microstructure, porosity, permeability, shrinkage and flexural strength of the elaborated MF membranes were studied as function of corn starch content (0–20 wt.%). The experimental results indicated that the porosity and permeability of MF membranes increased with the increasing of corn starch content. However the flexural strength reduced from 16.3 to 10 MPa with the increasing of corn starch content from 0 to 20 wt.%. Moreover the addition of corn starch had no significant effect on the linear shrinkage. Elaborated MF membranes were used to treat wastewater from textile industry. The filtration results showed that the turbidity retention decreased with the increasing of corn starch content and all the turbidity retention values were greater than 80%. MF membranes with a satisfied porosity, permeability and mechanical strength were elaborated with the addition of 10 wt.% of corn starch.

1. Introduction

Porous ceramic membranes have been extensively employed in several industrial processes such as wastewaters treatment [1–4], food processing, and pharmaceutical manufacturing due to their high thermal, chemical and mechanical stability [5–8]. An excellent MF membrane should possess a high mechanical strength to withstand the stress induced by the applied trans-membrane pressure, a high porosity to minimize the permeation resistance, and narrow pore size distribution which mainly depend on the raw materials, sintering temperature and fabrication methods [9–11].

A lot of materials were used for membranes preparation; many researchers used kaolin [12–14], lixiviated phosphate powder [15] and fly ash [16,4]. Several works used natural clay to prepare membrane supports or microfiltration membranes [17,18]. The use of clay as raw material in the formulation of ceramic membranes is aimed to produce low cost supports and a strong ceramic body after sintering at a relatively low temperature as compared to the other materials.

Many methods were used to vary the properties of porous ceramic membranes which are mainly determined by membranes composition [19,20], pore former content [21,22] and the sintering temperature [23,24]. Generally, the introduction of pore former in the membranes composition increases the porosity and the permeability. However, the sintering temperature of porous ceramic materials is considered an important factor affecting their mechanical properties, microstructure, and porosity. The porosity and the mechanical strength increase with increasing of sintering temperature. The particle size of raw materials has an important effect on the microstructure and mechanical strength; with decreasing in particle size, the apparent porosity decreases and the mechanical strength increases.

As known, to elaborate a low cost MF membrane, cheap materials and economic methods should be used. So the addition of pore former such as starch in the membranes composition is the appropriate method to regulate the membranes properties. Plenty studies included starch in the membranes composition to increase the porosity [25–27]. Starch generates pores during its burning at sintering temperature between 300 and 600 °C [28,29], it is more preferable than other pore former such as calcium carbonate $CaCO_3$ because starch is very cheap, easy processing, environmentally friendly and has a complete burn out without ash residues.

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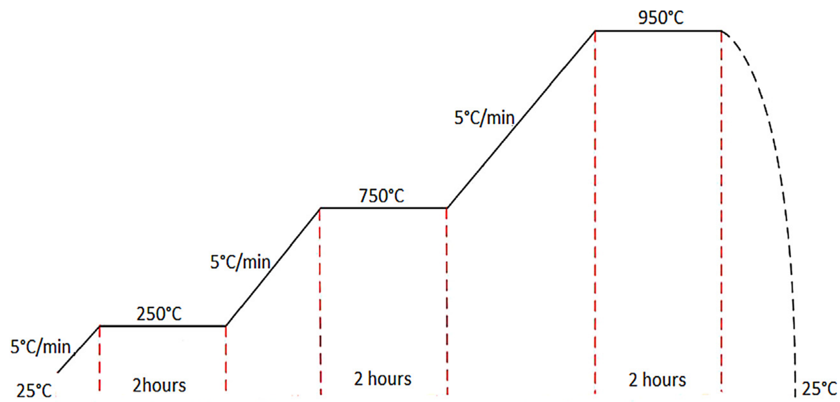

Fig. 1. Diagram of thermal program sintering.

Table 1
Chemical composition of natural clay (wt. %).

Oxides	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	CaO	Na_2O	TiO_2	Lo.I*
Mass%	41.28	12.29	4.131	2.803	1.736	14.23	1.02	0.62	20.2

* Lo.I: loss on ignition at 1,000 °C.

In this paper, natural Moroccan clay and corn starch with different quantities were used to prepare MF membranes by dry pressing method. The effect of starch addition at different weight percentage on the microstructure, porosity, permeability, shrinkage and mechanical strength of elaborated MF membranes was investigated in the aim of optimizing the corn starch quantity. The clarification of colored solution from textile industry was used to test the performance of the elaborated MF membranes in the filtration process.

2. Materials and methods

2.1. Preparation of MF membranes

The starting materials used to prepare the MF membranes are clay and corn starch. Natural Moroccan clay was extracted from Fès region located in the eastern lowest part of Saiss basin. Table 1 shows the chemical composition of raw clay. The main components of raw clay are SiO_2 and Al_2O_3. Corn starch was used as porosity agent at different contents of (0–20 wt.%)

The elaboration process of MF membranes from powder preparation to sintering step are the following:

- Crushing the raw clay in order to minimize the particle size;
- Sieving the powder through a sieve of 63 µm;
- Mixing of clay powder and corn starch at different weight percentage of (0–20 wt.%) using mixer machine in order to obtain a good homogeneity;
- Shaping by uniaxial pressing using an hydraulic press to form flat MF membranes with 4 cm of diameter and 1.5 mm of thickness.
- Sintering of the MF membranes using a programmable oven (Nabertherm L9/13/P320).

The thermal program used in this work is shown in Fig. 1. The MF membranes were heated up to 250 °C for two hours in order to eliminate the adsorbed surface water. To remove the structure water and corn starch completely, the MF membranes were sintered at 750 °C for two hours. Finally the MF membranes were sintered at 950 °C for two hours followed by a slow cooling ramp to avoid cracks. Heating rate of 5 °C/min was used for the three thermal program steps.

2.2. Characterization of natural clay

The characterization methods of chemical composition, crystalline phase composition (XRD) and Thermo-gravimetric analysis (TGA/DTG) were already explained in a previous work [30]. The particle size distribution of clay was obtained by dry laser diffraction (Malvern Mastersizer 2000) and the characteristic diameters D_{10}, D_{50} and D_{90} were calculated.

2.3. Characterization of MF membranes

The surface morphology, the presence of possible defects and the pore structure of the sintered MF membranes were analyzed by Scanning Electron Microscopy (SEM) (Hitachi, S-4500). The apparent porosity, water adsorption and apparent density were measured in accordance with ASTM C373-88 method using distilled water as an immersion medium.

Rectangular-shaped samples with 50 mm of length, 20 mm of width and 2 mm of thickness and sintered at 950 °C were used to measure the flexural strength. The bending tests were performed at a constant cross head speed of 0.1 mm/min using a three-point bending fixture with a span of 0.24 mm.

The flexural strength was calculated according to the following expression (Eq. (1)).

$$\boldsymbol{\sigma} = \frac{3\boldsymbol{Pl}}{2\boldsymbol{bh}^2} \quad (1)$$

where $\boldsymbol{\sigma}$ is the fracture strength (Pa), $\boldsymbol{P}$ is the fracture load (N), $\boldsymbol{l}$ is the span length (m), $\boldsymbol{b}$ is the width of samples (m), and $\boldsymbol{h}$ is the thickness of samples (m).

The average pore size of MF membranes sintered at 950 °C was calculated using the extended Hagen–Poisseuille equation (Eq.2) [31]

$r = \sqrt{8J\gamma \frac{\tau}{\varepsilon} \frac{\Delta X}{\Delta P}}$ (2)where $\boldsymbol{r}$ is the pore radius (m), $\boldsymbol{J}$ is the water flux (m/s), $\boldsymbol{\gamma}$ is the water viscosity is 10^{-3} Pa s, $\boldsymbol{\tau}$ is the tortuosity (2.5 for sphere particle packing), $\boldsymbol{\varepsilon}$ is the porosity (%), $\boldsymbol{P}$ is the pressure difference (Pa), and $\boldsymbol{X}$ is the membrane thickness (m).

The linear drying and sintering shrinkage of the samples were determined by the diameter measurements before and after drying and sintering process using a vernier caliper.

2.4. Filtration experiments

This study aims to test the membranes performance in the filtration process; the hydraulic permeability and the filtration test were realized using a laboratory setup made from glass. The clarification of textile industry effluent was released using the MF membranes prepared from natural clay and corn starch at differ-

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