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Development of hollow fiber membranes with alumina and waste of quartzite

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The development of ceramic membranes with different geometries and compositions extends the possibilities of industrial applications, inducing advantages in terms of increased permeability, membrane area by volume module and chemical, thermal and mechanical resistance. The use of low-cost raw materials is a trend that has grown in scientific research. The aim of this work is to prepare membranes with hollow fiber geometry from alumina and residue of quartzite, by the technique of immersion precipitation in distilled water from a mixture of ceramic mass with a solution of polyethersulfone and, synthesized in temperatures of 1100 ºC to 1500 °C. The hollow fiber membranes were characterized by chemical analysis, X-ray diffraction, particle size distribution, scanning electron microscopy, apparent porosity, flexural strength and permeated water flow by the membranes. The results indicated that the sintering temperature has direct influence on the formation of the mullite phase, and the properties of apparent porosity and permeate flow. The higher the sintering temperature (1400-1500 ° C) increase the formation of the mullite phase, the lower the porosity, as well as the lower the permeate water flow in the membranes. However, there was increase in flexural strength in the hollow fiber membranes with high temperature.

Keywords: *Hollow fiber membranes, mullite, waste of quartzite, alumina.*

1. Introduction

The ceramic membranes have attracted significant attention when compared to polymeric membranes due to their properties, with regard to mechanical strength, high chemical resistance, thermal stability, insensitive to swelling and easy to clean[1,2]. Ceramic membranes are suitable for applications involving extreme processes and where long-term stability is required[3]. However, conventional tubular ceramic membranes when compared with the hollow fiber configuration, (surface area up to the volume of 9000 m^2 m^3)[1], have a much smaller membrane area, which can make them less competitive.

The phase inversion technique combined with sintering is a method of producing hollow fiber ceramic membranes quite promising. Initially, a suspension of ceramic particles in solvent is prepared with a polymer binder and then, through the phase inversion technique of the binder, via exchange with a non-solvent during spinning, the ceramic particles are immobilized. This method can form membranes with specific morphologies. After this preparation, the calcination and sintering are necessary to remove all the organic precursors of the membrane and to consolidate the membrane structure and the mechanical strength. This method of membranes preparation in hollow fiber shape has been used to fabricate membranes from a wide range of ceramics, and even metals[4-9].

Several ceramic materials have been highlighted in the manufacture of membranes, such as alumina, silica, zirconia and titanium. However, they have a high manufacturing cost, mainly due to the materials that are synthetic, so that they are limited for large-scale applications, thus demanding the research and development of sources of economically viable raw materials[10-11]. Recent studies point to the phase of mullite ceramics as a good alternative in order to reduce the cost of manufacturing, since it can be obtained by economically viable sources and presents properties that ensure good performance in the preparation of ceramic membranes[10-15].

Recent research points out several forms of mullite sintering, for the development of membranes from minerals that present alumina and/or silica in their compositions[10,11,16-18]. Among the studies addressing mullite sources, it may highlight those in which the authors make use of low-cost raw materials or materials from waste as from the rice husk silica [10], kaolin residue[19]; kyanite[20]; bauxite[13,14].

The quartzite is a rock, geologically classified as a metamorphic rock, consisting of more than 80% of quartz, and is an abundant ore in Brazil. The importance of the rock sector for the Brazilian economy is incontestable, but the waste generated is inadequately disposed in the environment,

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without the prediction of reuse[21-25]. Due to the large volume of waste generated, both fine and coarse particle size, the possibility of applying as raw materials in other industries, is seen as an imminent solution to a set of environmental problems that may arise due to improper disposal of this material. Researchers already use this residue to develop gres porcelain [21], red [22] and structural ceramics[23]. Indeed, utilizing this waste to make low-cost ceramic membranes can be a promising alternative to waste reduction and recycling.

The aim of this research is to prepare hollow fiber ceramic membranes from alumina and residue of quartzite (fine), to obtain the mullite phase.

2. Experimental

2.1 Materials

For this study, it was used the following materials: Alumina powder with an average particle size of 0.4 - 8.0 μm [Imerys Fused Minerals, α-phase > 99.6%], and waste of quartzite supplied by industry located in Paraíba/Brazil were used as the membrane material. N-Methyl-2-pyrrolidone (NMP, Neon Comercial, purity 99.92%) was used as a solvent to prepare the initial solution. Polyethersulfone (PES, SOLVAY) and Polyethersulfone (PES, Ultrason, 6020P) was used as polymer and Polyvinylpyrrolidone (PVP Neon Comercial) was used as viscosity enhancer and de-ionized water as non-solvent.

2.2 Preparation of spinning dispersions

The quartzite residue was submitted to a beneficiation, consisting of grinding and sieving, using the mill CT-242, Servitech, for 45 minutes, and sieve ABNT 200 (aperture 0.074 mm).

The combined phase inversion-based extrusion method and sintering technique was used to prepare hollow fiber membranes. Suspension was prepared by mixing Arlacel P135, Polyethersulfone (1 wt%), in an N-methyl-2-pyrrolidinone (NMP) solvent (7.14 wt%). Afterwards, the mixture was rolled/milled at 100 rpm for 1h. Alumina (60 wt%) and waste of quartzite (40 wt%), and polyvinylpyrrolidone (PVP, 8 wt%) were added later as a binder and further rolled/milled for another 30min. Prior to extrusion, the suspension was degassed for 24 h to remove any entrapped air bubbles.

2.3 Preparation of hollow fiber membrane

After degassing, the spinning suspension was subsequently pressurized with nitrogen pressure of 3 Bar and extruded through a tube-in-orifice spinneret (Internal diameter = 1.17 mm; external diameter = 2.35 mm). Distilled water was used as the internal coagulant. The degassed suspension was extruded through a spinneret into distilled water at a constant speed of 250 mL/h with bore fluid of 250 mL/h. The fiber membrane green bodies passed through a certain air-gap distance of 10 cm were immersed in a water coagulant bath to allow the completion of the phase inversion process. Afterwards, they were dried at room temperature. Finally the hollow fibers were sintered at a target temperature ranging from 1100 to 1500 °C. The temperature was first increased to 500 °C at a rate of 2 °C /min to remove any remaining liquids and held for 1 h for the removal of organic polymer binder and dispersant. Next, the temperature was further increased to the target temperature at a rate of 5 °C/min to consolidate the ceramic membrane, up to the specified final temperature for 1 h.

2.4 Characterizations of hollow fiber membrane

The chemical compositions of the raw materials were determined by X-ray fluorescence spectrometry using a Shimadzu EDX 700 equipment; and particle size distribution was per-formed by laser scattering (Malver-Mastersiezer 2000) with a dispersion unit (HYDRO 2000G) at 8000 rpm stirring and ultra-sound. The ceramic hollow fiber membranes were characterized by X-ray diffraction (XRD), using a Shimadzu XRD 6000 equipment, with λ = 1.541 Å, operating at 40 kV and 30 mA and scanning angle from 5 to 80°, for analyze the developed crystalline phases. The images from scanning electron microscopy were obtained in SSX 550 Superscan - Shimadzu equipment, for analysis of the cross section. The porosity of the membranes was measured using the Archimedes method. For the flow measurement test with distilled water a bench system with tangential flow, at pressure of 1 bar and room temperature (25 °C), was used.

The flexural strength of the sintered hollow fiber from 1100 to 1500 ºC (three test specimens) was determined by using a three-point bending apparatus with a load cell of 5kN. The hollow fibers were positioned onto a planar base horizontally, and a planar crosshead is lowered down at a speed of 0.5 mm/min until fracture occurred. The bending strength was measured as follows[26]:

$$\sigma_F = \frac{8FLD_0}{\pi(D_0^4 - D_i^4)} \qquad (1)$$

Where, F is the force at which the fracture of specimen takes place, L is the span, which was kept at 40.3 mm and Do and Di are the outer and inner diameter of the hollow fiber, respectively.

3. Results and Discussion

The chemical composition of waste of quartzite and alumina (wt.%) is given in Table 1.

Alumina contains practically aluminum oxide (99.60%) and a small amount of other components (0.4%) resulting from contamination during the obtaining process.

The results of chemical analysis of quartzite residues showed that the silica is the main component (~79%), followed by alumina (~12%). The high levels of SiO_2 and Al_2O_3 are typical of metamorphic rocks. The silica present in the

Table 1. Chemical compositions of raw materials (wt%): Alumina and waste of quartzite (WQ).

	SiO_2	Al_2O_3	MgO	K_2O	Fe_2O_3	CaO	TiO_2	SO_3	Others
Alumina	0.27	99.60	-	-	0.04	-	-	-	0.09
WQ	79.62	12.43	0.95	4.44	1.11	0.63	0.13	0.44	0.25
Formulation	32.34	63.56	1.54	1.52	0.49	0.28	-	-	0.27

WQ - Waste of quartzite.

residues comes from free silica, feldspar and mica. In smaller proportions, there are the melting oxides, usually present in the quartzite residues as impurities in the form of feldspar and micaceous mineral. The K_2O content (< 4%), probably is related to the presence of mica and microcline detected in these samples. As for calcium and magnesium oxides, they may be associated with minerals such as dolomite. The low percentage of iron oxide, around 1.0%, is fundamental for the production of white ceramics, because in levels higher than 5%, this oxide can develop reddish color in the ceramic piece during the sintering[21-23].

Other researchers studied the incorporation of quartzite residues in ceramic masses and in the chemical analysis it were detected contents of silica, alumina and fluxes oxides similar to those found in this work[21-24].

The results of the chemical analysis of membrane formulation indicated that, the mixture contains 32.35% of silicon oxide from quartzite residues, 63.56% of aluminum oxide and 4.1% formed by other oxides. In general, the chemical analysis indicates that the obtained composition can confer suitable contents of SiO_2 and Al_2O_3 with potential for the mullite phase formation.

In Figure 1 it was observed the granulometric distribution of the ceramic mass formulation.

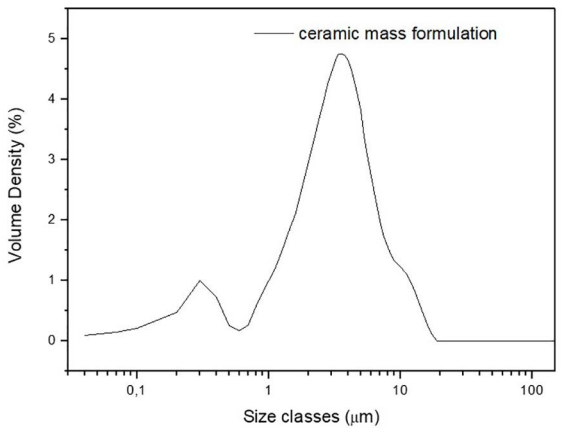

Figure 1. Particle size distribution of the ceramic mass formulation.

The result of the particle size analysis of the ceramic mass formulation, Figure 1, indicates a bimodal behavior, 10% of the particles have an equivalent diameter of less than 0.9 μm; 50% below 3.69 μm and 90% of the particles below 18.30 μm; the equivalent mean diameter was 2.85 μm. The narrower the distribution presented by the curve of particle diameter, the greater homogeneity in terms of the distribution, geometry and pore size of the membrane[27]. Considering that the presented distribution is narrow, it is expected that the membranes distribution, size and geometry of pores are suitable for the separation processes.

The Figure 2 illustrates the results from X-ray diffraction of the membranes sintered at 1100 °C, 1200 °C, 1300 °C, 1400 °C and 1500 °C.

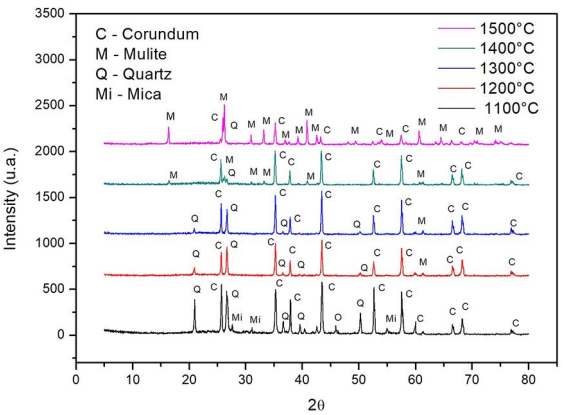

Figure 2. XRD analysis of hollow fiber membranes from alumina and waste quartzite sintered at 1100 °C, 1200 °C, 1300 °C, 1400 °C and 1500 °C.

From the X-rays diffraction curves of the hollow fiber membranes, Figure 2, it can be identified that, in general, the main characteristic peaks were: mullite (JCPDS: 79-1276), corundum (JCPDS: 10-0173) and quartz (JCPDS: 46-1045). Only for 1100 ºC appeared the peak of mica (JCPDS 83-1808). The formation of these peaks corroborates with the chemical analysis and composition of suspension used for membrane formation, including residue of quartzite and alumina.

Increased sintering temperature of the membranes from 1100 ºC to 1200 °C generated the disappearance of the crystalline phase of mica, due to the fluxes oxides, and also a decrease in the intensity of the corundum and quartz peaks, and appearance of the peaks characteristic of the mullite phase with low intensity.

According to the X-ray diffractions results, the compositions of the phases of each sample were calculated, as listed in Table 2.

From the Table 2, it was found that the increase in the sintering temperature, the mullite content was increased and the quartz and corundum were reduced, as is also perceived that crystallinity decreases, indicating that the vitreous phase content has increased.

Table 2. Phase compositions of the membranes sintered at 1100°C, 1200°C, 1300°C, 1400°C and 1500°C.

Sample	C (wt%)	Corundum (wt%)	Quartz (wt%)	Mullite (wt%)	Mica (wt%)
HFM -1100ºC	93.62	77.16	14.76	-	1.7
HFM -1200ºC	92.76	76.23	13.22	3.31	-
HFM -1300ºC	85.06	*69.93*	*8.31*	*3.56*	-
HFM -1400ºC	79.21	51.41	6.33	21.47	-
HFM -1500ºC	80.01	13.27	2.31	64.43	-

HFM – Hollow fiber membrane C – Crystallinity.

Even for higher temperatures of 1400 ºC and 1500 ºC it is observed the presence of corundum and quartz, showing that these temperatures were not enough to these phases reacted, not achieving complete mullitization[20,28-30]. These results corroborate with research, which combined silica and alumina raw materials to obtain membranes and observed mullite phase formation at temperatures above 1300 °C [20, 28,30].

The Figure 3 presents the SEM images of the hollow fiber membranes.

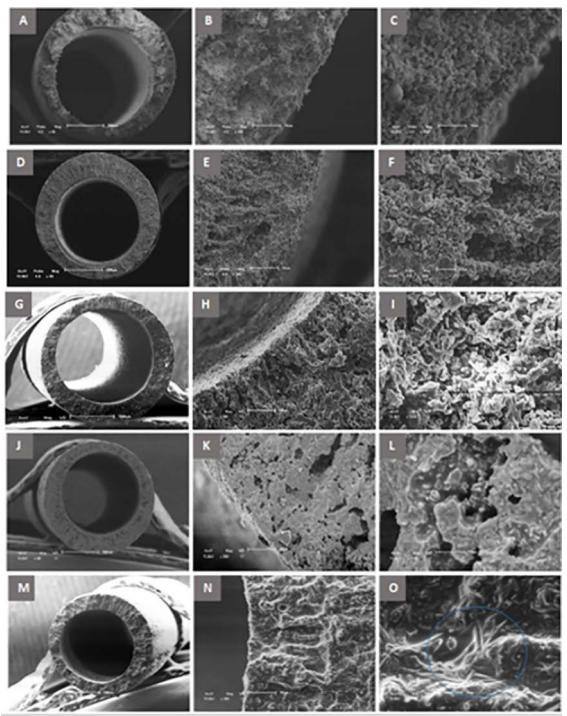

Figure 3. SEM images for membrane: HFM 1100 °C with magnification of (a) 40x, (b) 300x, (c)1500x ; HFM 1200 °C with magnification of (d) 40x, (e) 300x, (f)1500x; HFM 1300 °C with magnification of (g) 40x, (h) 300x, (i)1500x; HFM 1400 °C with magnification of (j) 40x, (k) 300x, (l)1500x and HFM 1500 °C with magnification of (m) 40x, (n) 300x, (o)1500x.

From the SEM images (Figure 3) of the hollow fiber ceramic membranes, especially HFM 1100 °C and HFM 1200 °C, there was an asymmetric structure consisting of a sponge-like region and by voids similar to "fingers", which is a typical structure for the hollow fibers prepared by the spinning method. The structure similar to a "finger" originated from the inner surface until half of the cross section. The spongy structure was located in the outer region of the cross section. During the spinning process, the macroporous similar to the "fingers" were generated due to rapid exchange between solvent (NMP) and non-solvent (water) [4,31]. The macroporous structure is the result of interfacial instabilities between the ceramic suspension and coagulant during the phase inversion process. This phenomenon that occurs naturally can be attributed to many different mechanisms, as a result of the difference in viscosity, density and/or interfacial tension between two fluids, which destabilizes the interface during phase inversion process[4,6,11].

From the cross section of the membranes, (Figures 3a and 3b) the presence of grains is visible, presenting as rounded particles[6], and pores with different sizes and preferably spherical shapes, distributed in a non-uniform way.

When the sintering temperature is increased above 1300 °C, a change in the morphological characteristic in the internal part of the membranes is detected, marked by the decrease in the number and size of pores, with a decrease in the size of the macroporous and densification of these membranes, especially at the highest temperature of 1500 °C (Figure 3c). This difference in morphology is probably related to the increase in the amount of vitreous phase formed during sintering of alumina and quartzite residue. The presence of silica from quartzite that did not react with alumina, has a strong influence on the sinterability properties of mullite in hollow fiber membranes. During the sintering process, these impurities favored the bridge between the grains and densification of the membrane[20,28].

The microstructure of the sample HFM 1500 °C (Figure 3o) have essentially mullite (needles) as the primary phase, without quartz as a crystalline phase, and it is detected the mullite covered by a vitreous phase. The excess of SiO_2 can increase the densification of a sample, due to liquid phase formation during sintering[20,28,29].

Figure 4 shows the apparent porosity of the hollow fiber ceramic membranes in relation to the sintering temperature.

Observing the apparent porosity of hollow fiber membranes, Figure 4, it is observed that the membranes with sintering temperature of 1100 ºC showed higher apparent porosity of 27.3 ± 0.4%, and the membranes sintered at 1400 °C have lower apparent porosity of 6.7 ± 0.5%.

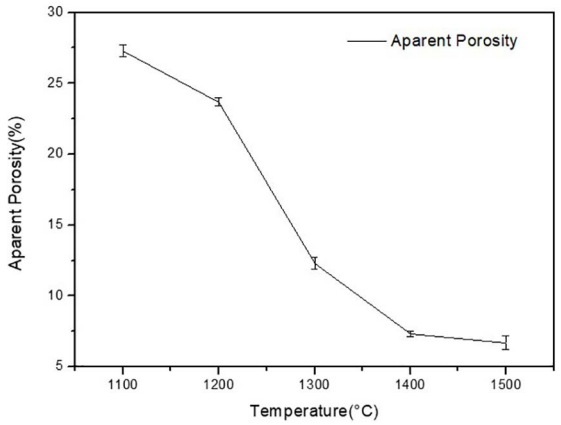

Figure 4. Apparent porosity of hollow fiber membranes at different sintering temperatures

In general, the increase in the sintering temperature caused a decrease in the apparent porosity, this behavior can be explained by the presence of liquid phase formed during heating at high temperatures, that eliminate small pores, leading to decreased porosity[20,32]. These results are in agreement with SEM images, Figure 3. The porosity directly influences the permeability and mechanical resistance of membranes. More porous, the lower the resistance to the flow passing through the membrane[32].

Figure 5 shows flow measurements with distilled water by hollow fiber membranes, sintered at 1100 °C, 1200 °C, 1300 °C and 1400 °C, with transmembrane pressure of 1.0 Bar.

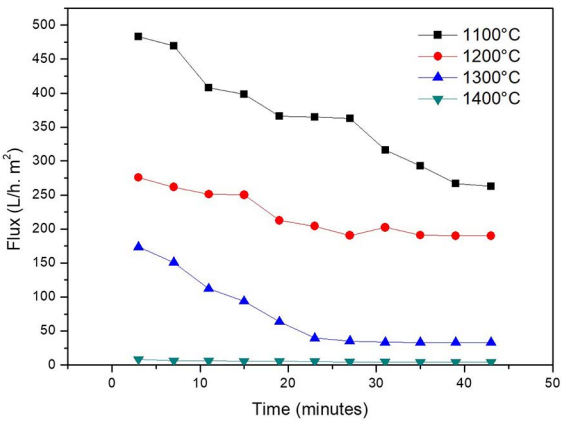

Figure 5. Water permeate flow of the hollow fiber membranes sintered at 1100 ºC, 1200 ºC, 1300 ºC and 1400 ºC and 1Bar of transmembrane pressure.

The permeate flow measurements with distilled water by the hollow fiber membranes, Figure 5, decreased over time until reaching a constant value from approximately 35 minutes of operation. This decay as a function of time may be associated with the hydration of the membranes[33,34,35].

The membranes sintered at 1100 °C presented higher stable flow value of 263.2 L/h. m², which may be associated with the greater apparent porosity value presented among the hollow fiber membranes, Figure 4. With the increase of the sintering temperature there was a decrease in porosity and, thus, a decrease in the permeate flow with distilled water by these membranes. The membranes sintered at 1400 °C showed lower flow value of 4.43 L/h. m² when compared with the other membranes.

Figure 6 shows the result of flexural strength of the hollow fiber ceramic membranes sintered at temperature of 1100 ºC, 1200 ºC, 1300 ºC and 1500 ºC, with values changing from 25 MPa (at 1100 ºC) to 82 MPa (at 1500 ºC). In this study, the flexural strength of sintered hollow fibers at 1100 °C is comparable to those of the mineral base, which are usually less than 30 MPa[31,34]. It can be seen that flexural strength increases gradually with increased sintering temperature. This increase may be explained by neck formation and increased densification, which corroborates the reduction of open porosity, as shown in Figure 4. Flexural strength improves dramatically when the sintering temperature increases to above 1300 °C, which can be attributed to increased mullite needles and the presence of vitreous phase leaving the membrane with a structure with entangled crystals and more denser.

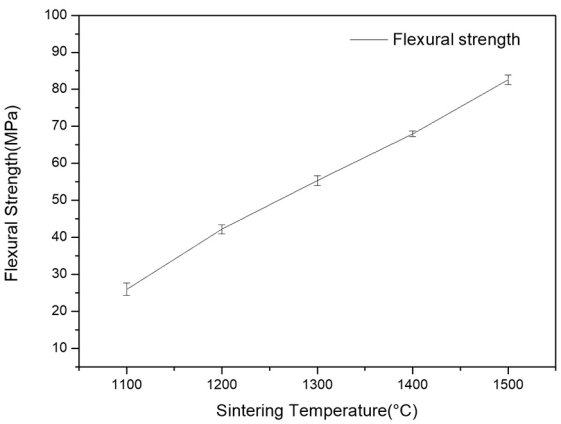

Figure 6. Flexural strength of hollow fiber membrane sintered at 1100 ºC, 1200 ºC, 1300 ºC and 1500 ºC.

4. Conclusions

The results of this research indicate that it was possible to obtain hollow fiber ceramic membranes from quartzite residue and alumina. The quartzite residue and alumina presented chemical and mineralogical properties suitable for obtaining the mullite phase. The sintering temperature had a direct influence on the formation of the mullite phase, as well as in the properties of apparent porosity and water permeate flow. The higher the sintering temperature (1400 - 1500 ºC) the greater formation of the mullite phase, confirmed by the intensity and presence of mullite characteristic peaks obtained by X-ray diffraction. Regarding apparent porosity, it is perceived that the increase of the sintering temperature caused a decrease in porosity, since the higher the temperature occurred the formation of more liquid phase and, consequently, elimination of the small pores,

leading to a decrease in porosity, which caused a decrease in the water permeate flow in the membranes. The densification of the membranes due to sintering reflected in the increase of the flexural strength, where the membranes sintered at 1500ºC had a value of 82.6 MPa.

5. Acknowledgements

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