CHARACTERIZATION OF POROUS CERAMICS PRODUCED BY A DRY METHOD USING WAX SPHERES

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Porous ceramics have been used as filters, thermal insulation materials, catalyst supports, gas sensors and biomaterials due to properties such as: low density, stability at high temperature, permeability, large surface area and thermal shock strength. This paper describes the characterization of porous ceramics obtained by a dry method using wax spheres with 75 microns. Alumina samples with 12 mm in diameter and 10 mm in thickness were produced by uniaxial pressing under 40 MPa and then heated to 550ºC to remove spheres and sintered at 1550ºC for one hour. Afterwards, they were analyzed by SEM and XRD. Results obtained so far have shown a satisfactory microstructure with uniform porosity, hence porosity influence on mechanical properties will be investigated further.

Keywords: Porous ceramics, wax spheres, characterization.

INTRODUCTION

Ceramic materials are used in several applications, since useful products such as wine and grain recipients to highly searched ornamental treasures, like plates made by Romans, Chinese and English artisans. Nowadays, there are ceramic products used for high technology, serving as heat insulators, for example. For most of the applications, ceramic bodies may be thought as dense, hard, extremely resistant and with functional shapes for human being needs. These general types of ceramics may be found in everything, since China until optic fiber wires, however, there are other types of ceramics that are developed because of their porous nature. A porous ceramic body is similar to most well known natural substances, like soils, rocks, and bones, for example.

There are several methods to produce porous structures such as burning at low temperatures associated with talc and clay, the polymeric sponge method, the addition of organic materials methods, etc.. Most porous ceramic bodies are made by porcelain and alumina and may be used as filters, aerators, for example.

This paper describes the characterization of porous ceramics obtained by a dry method using wax spheres with 75 microns.

LITERATURE REVIEW

The interest on porous ceramics is increasing, since new processes have been developed and their applications represent technological and economical opportunities [1]. For each application, there is at least a considerable property that must be evaluated, depending on its function: physical, chemical or biological. Porous ceramics are uses as battery cell separators, burners, catalysis supports, filters, gas sensors, etc. [1]. For structural parts, porosity may be useful when there is a necessity of weight reduction, whereas for non structural components, it may be useful for other functions, such as permeability or for a high surface area substrate. Aluminum oxide (Al_2O_3) is one of the most raw materials used for advanced materials because of its beneficial properties, such as high hardness, high wear resistance, low friction coefficient, high resistance to corrosion by chemical reagents, high resistance to high temperature corrosion in air, thermodynamic stability, that is, the absence of phase transformations within the entire temperature range of solid state, and the fact that the material retains its strength even at very high temperatures (about 1500ºC – 1700ºC) [2]. These properties make ceramic materials adequate for a variety of applications, where thermal and mechanical tensions do not allow to use metallic or polymeric materials [3].

In general, the range of porosity, the pore morphology and pore size distribution are determined by the fabrication method selected [4]. There are several methods to produce porous ceramics, such as the method of the polymeric sponge, the foaming method and the method where organic agents are incorporated inside the ceramic bodies [3]. The polymeric sponge method is based in an introduction of a ceramic paste into a sponge, where the sponge is burned out after a slow burning operation and then the

material is sintered and hence, a material with open porosity is obtained. The foaming method consists on the addition of a foam agent into a ceramic suspension. By agitation, there is a formation of a foam that makes a pore structure essentially closed after liquid phase removal. The most common applications for open-cell porous ceramics are molten-metal and diesel engine exhaust filters while reticulate ceramics are used as catalyst supports and industrial hot-gas filters [5]. The method of addition of pore formers consists in a addition of organic materials inside the ceramic parts, which will be removed during burning, where pore size is related with pore formers particle size. Natural and Synthetic waxes are also used not only as pore formers but when a low viscosity is desired on processes such as low pressure ceramic injection [6].

Porosity is almost always present in ceramics prepared by powder compaction and heat treatment and may be defined as the volume fraction of pores present [7]. There are two types of porosity: open and closed. When the pores hit the body external surface, they are named open porosity whereas pores closed with the particle are named closed porosity [6]. The open porosity may be measured by Mercury porosimetry and gas sorption. Open porosity is useful for ceramic filters fabrication and closed porosity is important for insulators fabrication. The pores distribution obtained by addition of organic materials will depend on the pore formers distribution into the ceramic body and then it may have open and closed porosity [3].

EXPERIMENTAL

MATERIALS

Porous ceramics bodies were produced using three types of alumina, named A, B and C, (RC-LS, ERC DBM and RC-HPT DBM respectively) with addition of 30% vol. of wax spheres. The aluminas with differentparticle distributions were supplied by Reynolds, and the Licowax 520 supplied by Clariant S/A. The alumina's properties are shown on Table 1.

METHODS

Firstly, it was manually mixed the alumina with 30% vol. of wax to produce the ceramic material. Samples with 12 mm in diameter and 10 mm in thickness were produced by uniaxial pressing under 40 MPa and then heated to 550ºC to remove spheres and sintered at 1550ºC for one hour. Afterwards, they were analyzed by SEM and XRD, using a conventional Rigaku difractometer with two axes θ e 2θ for polycrystalline samples, operated at 40 kV / 30 mA with incident radiation of Cu ($\lambda_{k\alpha}$) and diffracted beam monochromatized by a curve graphite monochromator. Samples were measured at a range of 2θ: 20-80^{o} in continuous scan mode with 0.5^{o}/min and passes of 0.02^{o} at room temperature (23^{o}C), and a Scanning Electronic Microscopy Phillips XL 30, both from the Mechanical Engineering Department Universidade Federal do Ceará, Brazil.

RESULTS AND DISCUSSION

Figures 1 and 2 show the micrographies of A, B and C aluminas with 30% vol. of wax spheres after sintering at 1550ºC with 50x and 10000x respectively.

The porosity due to wax spheres evaporation may be seen in Figure 1. There isn't a good mixing homogeneity of the alumina and wax spheres because of a manual mixing. Figure 2 shows particle aspect. It may be seen that Sample A (Figure 2a) didn't syntherized well at 1550ºC, whereas Sample B showed low syntherization at 1550ºC (Figure 2b). Sample C seemed better syntherization than sample B at 1550ºC (Figure 2c).

XRD of samples A, B and C are shown in Figures 3, 4 and 5 respectively.

It may be seen in Figure 3 that three phases are present on sample A: Al_2O_3, Na_2O and SiO_2. XRD of sample B showed in Figure 4 that the same three phases presented on sample A were presented on it. Figure 5 shows the sample C XRD. Again, there are three phases are presented. There isn't phase of wax spheres presented for each sample.

CONCLUSION

By employing the organic wax addition method to produce samples, porous alumina bodies with 12 mm in diameter and 10 mm in thickness have been prepared.

The microstructure of these bodies depended on the way wax spheres were added on alumina. SEM showed that there wasn't structural changes on samples and there was pores control after wax spheres burnt, as shown in Figures 1 and 2. XRD showed that there wasn't wax spheres presented on samples struts and that the main peaks of Al_2O_3 were presented, such as for some impurities (NaO_2 and SiO_2). In terms of syntherization, sample C showed a few better than samples A and B. Although samples B and C seemed to be similar, sample B has a notable hydrated surface determined by DRIFTS analysis that made it less compatible with non – aqueous systems, like to that used on this paper, which may lead to bubbles and weak bonds.

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TABLES

Table 1 – Typical properties of aluminas used on this paper.

Powder	**Purity (%)**	**Fired Density (g/cm^3)**	**Surface Area (m^2/g)**
RC-LS [a]	99.75	3.85	3.3
ERC DBM	99.80	3.92	7.5
RC-HPT DBM	99.97	3.83	3.5 – 4.5

a – sintered at 1620ºC for 1 hour.

FIGURES

Figure 1 – SEM of: (a) sample A, (b) sample B and (c) sample C – 50x.

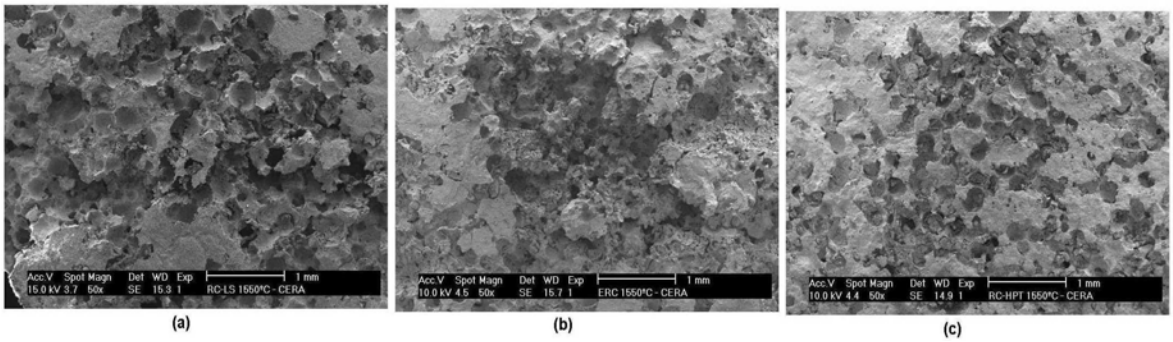

Figure 2 – SEM of: (a) sample A, (b) sample B and C – 10000x.

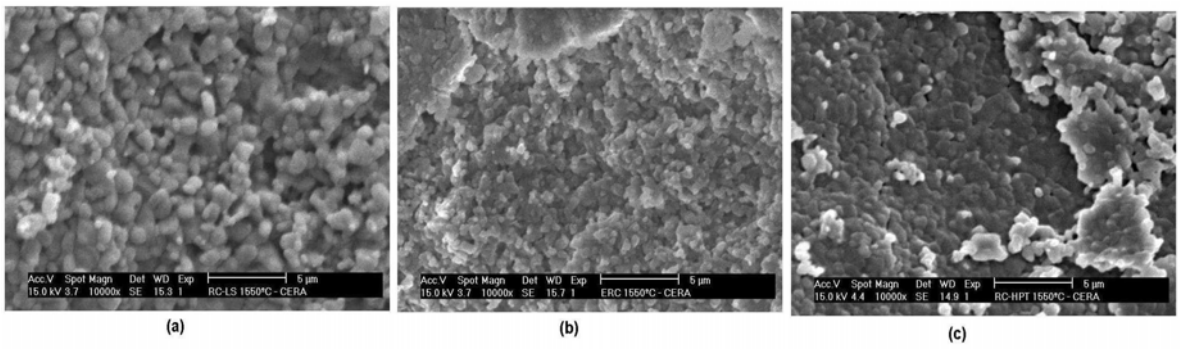

Figure 3 – DRX of sample A at 1550ºC.

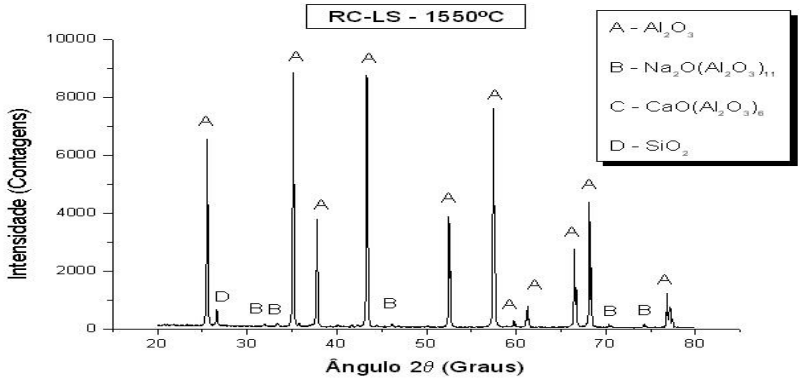

Figure 4 – XRD of sample B at 1550ºC.

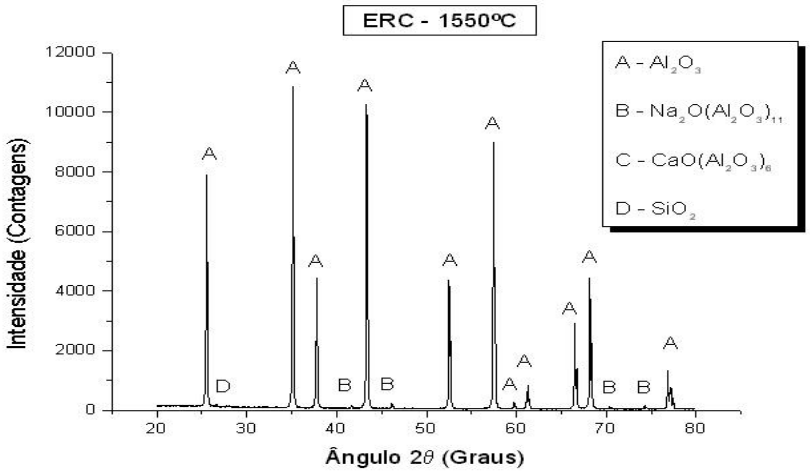

Figure 5 – XRD of sample C at 1550ºC.