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Preparation and characterization of porous alumina ceramics through starch consolidation casting technique

R.M. Khattab, M.M.S. Wahsh *, N.M. Khalil

Refractories, Ceramics and Building Materials Dept., National Research Center, 12622, Dokki, Cairo, Egypt

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Abstract

This work aims at studying the influence of thermal treatment on the microstructure, resistivity and technological properties of porous alumina ceramics prepared via starch consolidation casting (SCC) technique. Colloidal suspensions were prepared with three different contents of alumina solid loading (55, 60 and 65 mass%) and corn starch (3, 8 and 13 mass%). The sintered samples at 1400, 1500, 1600 and 1700 °C, show open porosity between 46 and 64%, depending on the starch content in the precursor suspensions and sintering temperature. The pore structures were analyzed by SEM. The effect of corn starch content on the apparent porosity, pore size distribution, linear shrinkage and electrical resistivity as well as cold crushing strength of the sintered porous alumina ceramics was also measured. These porous alumina ceramics are promising porous ceramic materials for using in a wide range of thermal, electrical and bioceramics applications as well as filters/membranes and gas burners, due to their excellent combination properties.

Keywords: D. Al_2O_3; A. Suspensions; A. Sintering; B. Porosity; B. Electron microscopy

1. Introduction

Porous ceramics are widely used for a variety of applications, including burners, thermal barrier coatings, insulating layers, filters for gases and liquids, catalyst support and thermally or acoustically insulating bulk materials [1,2]. The use of pore-forming agents is one of the frequently used methods to produce porous ceramics with controlled microstructure (porosity and pore size) [3,4]. During heating up to the final firing temperature of the ceramics, these pore-forming agents are burnt out, leaving void pores in the ceramic. Among the various pore-forming agents, those of biological origin, in particular starch, have gained a prominent position. They are cheap, non-toxic, environmentally friendly and exhibit defect free burnout between approximately 300 and 600 °C [5,6]. A specific advantage for porous ceramics is the fact that the pore size can be controlled by choosing the appropriate starch type and that the pore size distribution is in most cases sufficiently narrow to make technological process control efficient [7]. The pore sizes achievable with commercial starch types ranging from ~5 μm in rice starch to ~50 μm in potato starch [6].

Starch grains are normally white, dense and water insoluble at room temperature. Most starches consist of mixtures of two polysaccharide types, a linear one, amylose, and a highly branched one, amylopectin. Amylose gives the starch its gelling property in aqueous suspensions. The glucose units that build up the polymeric chains in starch expose a large amount of hydroxyl groups and, therefore, give a strong hydrophilic character to starch granules (Fig. 1) [8]. These have some favorable characteristics such as good thickening, stabilizing, membrane-forming and gelling properties [8].

The water insolubility of starch granules below about 50 °C means that they can be handled and processed at room temperature without significant impact on the granule structure. However, when the starch suspension is heated to a temperature between 55 and 80 °C (depending on type and concentration), the intermolecular bonds holding the granules together are weakened. During this process the granules undergo a rapid and irreversible swelling by water

* Corresponding author. Tel.: +20 1007561987; fax: +20 233370931.
E-mail address: mmswahsh@yahoo.com (M.M.S. Wahsh).

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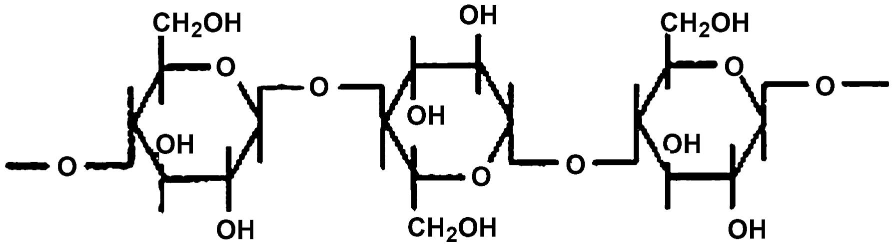

Fig. 1. Starch as a polymer consisting of condensed glucose units.

uptake which results in an increased granule size to many times the original size [9]

The new shaping technique has been presented: starch consolidation casting (SCC). This method based on the ability of starch to swell and finally gelatinize in water at elevated temperature (60–80 °C), so that ceramic green samples can be formed from suspensions in impermeable molds (usually metal molds) [7,10–18]. As the starch granules or particles swell they will also act as a binder adding strength to the consolidated body, which enables demolding prior to drying. After burn-out of the starch and sintering of the ceramic matrix a material is obtained with a porosity corresponding to the original amount, shape and size of the starch particles [8]. Today SCC has become a competitive shaping technique beside traditional slip casting with starch added as a mere pore former [7].

This work was intended to fabricate and investigate of porous alumina ceramics by starch consolidation casting (SCC) technique using different contents of alumina (as solid loading) and corn starch after sintering at different temperature (1400, 1500, 1600 and 1700 °C).

2. Materials and experimental methods

2.1. Starting materials

The starting materials used for preparation of the porous alumina ceramic samples are:

(1) A commercial calcined alumina (α-Al_2O_3) powder with mean particle size $\leq$10 μm, its chemical composition is given in Table 1.

Table 1
Chemical composition of the starting alumina powder, mass%.

Oxides	Calcined alumina
SiO_2	0.74
Al_2O_3	98.20
Fe_2O_3	0.41
TiO_2	0.23
CaO	0.27
MgO	0.07
Na_2O	0.10
K_2O	0.05
Cl^-	–
SO_3	–
LOI	–
Total	100.07

Table 2
Composition of the colloidal suspensions, mass%.

Sample	Alumina solid loading	Corn starch content	Polyethylene glycol	Water content
A	65	3	2	30
B	60	8	2	30
C	55	13	2	30

(2) Poly ethylene glycol (PEG of molecular weight 10,000) used as a dispersant agent and also required for obtaining the significant mechanical resistance before the final consolidation by sintering [19], it is supplied by Fluka.

(3) Native corn starch, commercially available in the market for use in cooking.

2.2. Experimental procedures

To prepare slurry, different amount of calcined alumina ranging from 65 to 55 mass% (as seen in Table 2) was added stepwise to a fixed amount of premix solution containing poly ethylene glycol (PEG) and de-ionized water followed by milling for 2 h using a mechanical mill then different contents of starch ranging from 3 to 13 mass% were added to the premix solutions that containing alumina thoroughly. After 1 h of mixing, the slurry was subjected to de-aeration to remove undesired entrapped bubbles. After that, they were poured into the molds, which heated in air (80 °C, 1 h) for the gelatinization process.

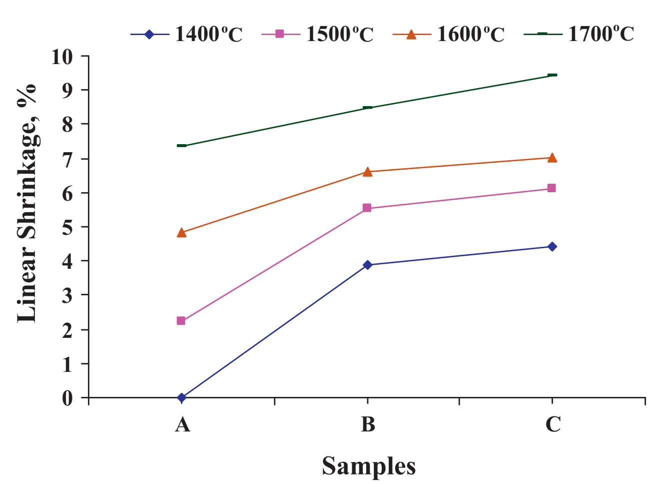

Fig. 2. Linear shrinkage of the porous alumina samples sintered at different temperatures 1400, 1500, 1600 and 1700 °C.