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Humans - Valuable Resource for Foundry Industry Development

Split, June 16th – 18th, 2021

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PREPARATION AND CHARACTERIZATION OF POROUS ALUMINA CERAMICS USING WASTE COFFEE GROUNDS (WCG)

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Abstract

Different methods have been used to produce porous ceramics for various applications. The generation of pores in ceramic samples offers a new range of different properties. Moreover, that porosity can be achieved using all kinds of various waste such as industrial, agricultural, domestic etc. One example being the process of coffee brewing which produces a large amount of waste, that was therefore used as a pore-forming agent in the production of porous ceramics.
Suspensions of 60 wt.% alumina with different amount of waste coffee grounds were prepared. Viscosity of all suspensions was determined by means of rotational viscometer. Green bodies were prepared by slip casting in plaster mold. DTA/TGA analysis of waste coffee grounds revealed multistep endothermic and exothermic events related with release and burning of the precursor materials accompanied with mass loss. The sintering regime was determined according to the DTA/TGA analysis of thermal decomposition of waste coffee grounds. Density of the obtained sintered samples was investigated and calculated.

Keywords: *alumina, porous ceramics, coffee, density, slip casting*

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INTRODUCTION

Porous alumina ceramics have been widely manufactured for various applications such as filters and membranes [1], catalyst supports [2], refractory materials [3], construction materials [4] thermal insulators [5] and many other. This wide application scope is attributed to achieve bodies with high porosity, high surface area, low density, high thermal and chemical resistance as well as corrosion resistance. Manufacturing of porous ceramics can be executed with a number of different methods such as precipitation [6], replica technique

[2], sacrificial template [7], with a pore-forming agent [8–10], uniaxial pressing [4] and isostatic pressing [8].

With the decrease in non-renewable resources, the interest in using waste materials in production of new and improved ones has been rapidly growing. Waste is generated in all branches of society, from small households to large industry plants. Different kinds of waste are being used as pore forming agents in manufacturing porous ceramics. Liu et al. [8] used walnut shell powder as pore-forming agent combined with alumina sol impregnation for fabricating porous alumina ceramics with enhanced crushing strength and thermal insulation performance. Graphite waste, primarily from batteries, was used by Ali et al. [9] as pore-forming agent to fabricate porous alumina ceramics using a fugitive material technique. The increasing porosity decreased mechanical strength but improved deformation tolerance, resulting in higher strain at break. The same authors also used commercial rice husk ash via solid-state technique to obtain a porous ceramic composite [11]. Mechanical properties were improved for composites with higher ratios of rice husk ash. Eliche-Quesada et al. used various industrial wastes to produce ceramic bricks with improved thermal insulation properties [12]. Paper waste, corn starch and sawdust were used to manufacture low-cost porous ceramics by Salman et al. [10]. The paper waste was the most suitable pore-forming agent because it allowed preparing of porous ceramics displaying good mechanical properties, porosity and permeability.

Since coffee is one of the most popular and often consumed beverages in the world, huge amounts of coffee waste are produced during the brewing process [13]. This coffee waste can also be used as pore-forming agent in production of porous ceramics. Sena da Fonseca et al. studied the influence of coffee waste in structural ceramics for construction [4]. They concluded that coffee waste can be used as a secondary clay raw material to produce bricks with improved thermal insulation properties. The use of coffee waste for preparation of macroporous alumina ceramics resulted in high open porosity of the ceramic material as well as good mechanical strength performed by Alzukaimi and Jabrah [7]. Furthermore, spent coffee grounds were also used as filler for the formulation of lightweight clay ceramic aggregates by Andreola et al. [14]. Their results showed that the obtained material has an interesting set of properties that make it possible to use it in both urban and agricultural purposes.

In this paper, waste coffee grounds were used to produce porous alumina ceramics by slip casting method [15]. The preceramic suspensions containing different amounts of waste coffee grounds were described by rheological measurements, while sintering regime was discussed on behalf of achieved densities study and DTA-TGA analysis of thermal decomposition of coffee grounds.

MATERIALS AND METHODS

Alumina suspensions were prepared with 60 wt.% of alumina powder. High-purity Al_2O_3 powder was used, with the average particle size of 300-400 nm (Alcan Chemicals, USA). Dolapix CE64 (Zschimmer & Schwarz Chemie GmbH, Germany) was used as a dispersant to stabilize highly concentrated alumina suspensions. Polyvinyl alcohol, PVA (Sigma Aldrich, USA) was added to the suspension as a binder. Different amounts (1 and 5 wt.%) of waste coffee grounds (WCG), obtained from a household coffee machine with a grinder, were added to alumina suspensions as a pore-forming agent.

Suspension preparation

0.2 wt.% of dispersant and 0.5 wt.% of binder were mixed with deionized water and placed in the grinding jar of the planetary ball mill. 60 wt.% of dry monolithic alumina powder was mixed with different amounts of waste coffee grounds (WCG) and added into the grinding jar. Ten alumina balls were used for the mixture homogenization, which lasted for 90 minutes at a speed of 300 rpm. Alumina balls were separated from the suspension after the homogenization using a sieve. The suspension underwent an ultrasonic treatment for 15 min in an ultrasonic bath – BRANSONIC 220 (Branson Ultrasonics Corp., USA) to remove air bubbles and achieve better homogeneity. Afterwards, suspensions were poured into plaster moulds to produce a ceramic green body.

Determination of rheological properties

Rheological properties of prepared ceramic suspensions were determined using a rotational viscometer ViscoQC™300 (Anton Paar, Graz, Austria) in a measuring cup C-CC12 with a measuring bob B-CC12. The shear rate was gradually increased from 0.1 to 180 s^{-1}, and then reduced back to 0.1 s^{-1} divided into 50 equal time frames. Rheological parameters were recorded just before each shear rate change.

Sintering of green bodies

The obtained green bodies were sintered in an electric furnace (Nabertherm P310, Bremen, Germany) according with the course of thermal decomposition of waste coffee grounds, which was determined by Differential Thermal Analysis (DTA) and Thermo-Gravimetric Analysis (TGA) using a simultaneous DTA/TGA device STA409 (Netzsch, Selb, Germany). Density of the sintered alumina samples was measured using Archimedes' principle on a laboratory scale by Mettler-Toledo while the density of samples with the addition of waste coffee grounds was calculated theoretically.

RESULTS AND DISCUSSION

Rheological measurements

Rheological properties were examined using rheological flow curves which show the dependence of shear rate (γ) on viscosity (η). They are used to predict the nature of interactions between particles in the suspension. Flow curves for alumina suspension without the addition of waste coffee grounds (WCG) as well as alumina suspensions with the addition of 1 and 5 wt.% of WCG are shown in Figure 1.

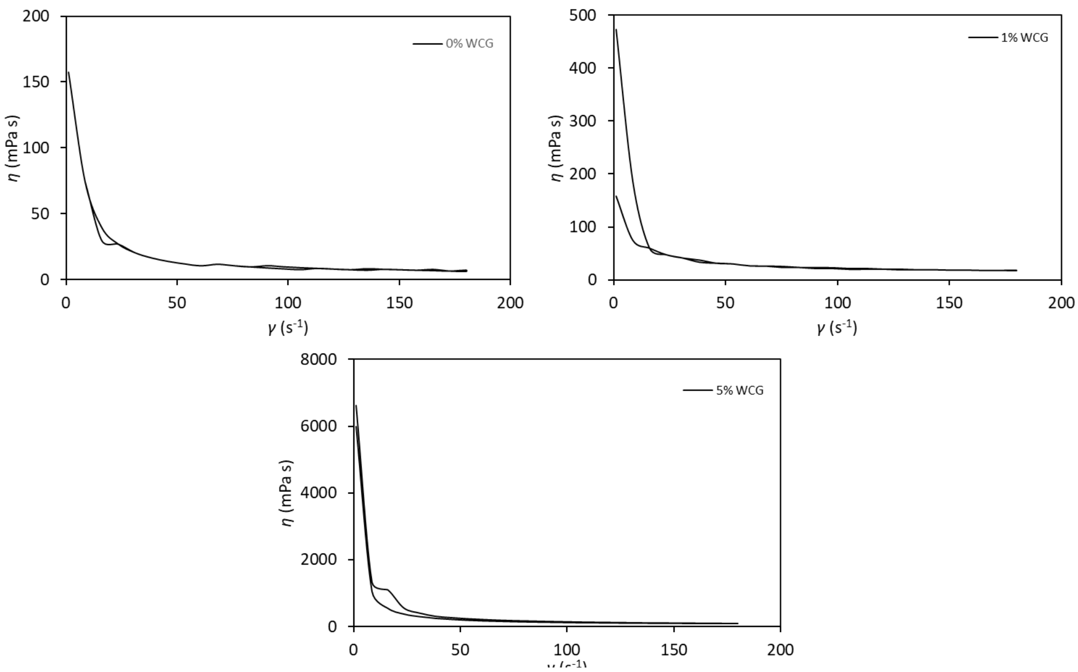

Figure 1. Rheological flow curves of prepared alumina ceramic suspensions with 0, 1 and 5 wt.% of waste coffee grounds (WCG)

The obtained results show that the suspension viscosity decreases with an increase of the shear rate, that is, all suspensions show typical pseudoplastic behaviour, characteristic of non-Newtonian fluids.

Suspension stability was estimated by the viscosity measurements at two shear rates, 50 and 100 s^{-1}. The shear rate of 50 s^{-1} is the exact shear rate usually achieved during gravity slip casting while the shear rate of 100 s^{-1} was chosen in purpose of better comparison. The obtained viscosity values of prepared alumina suspensions with the addition of waste coffee grounds (WCG) are shown in Table 1. Viscosity of prepared suspensions increased with the addition of waste coffee grounds.

Table1. Measured viscosity for prepared ceramic suspensions

w (WCG in powder mixture), %	*η* (mPa s)	
	γ, 50 s^{-1}	*γ*, 100 s^{-1}
0	11.78	8.87
1	29.44	19.22
5	241.40	143.40

Differential Thermal Analysis (DTA) and Thermo-Gravimetric Analysis (TGA)

DTA/TGA analysis (Figure 2) of waste coffee grounds revealed a single endothermic event up to 100 °C related with evaporation of the moisture accompanied with ~0.5 % of mass loss. After that, up to 200 °C another single endothermic event related with release of chemisorbed moisture accompanied with ~0.25 % of mass loss. Above 200 °C to about 450 °C from the DTA a single broad exothermic event related with burning of the organic material accompanied with TGA mass loss in what appears to be a three-step process; one from 200 °C to about 320 °C, second from 320 °C to about 390 °C, and the third from 390 °C to about 450 °C. From 400 °C to about 750 °C the DTA signal is dominated by broad endothermic envelope attributed to the subsequent release of the decomposition residuals. At 800 °C ambiguous overlapped endothermic and exothermic effects with small intensity are observed without mass loss. Above 900 °C carbonaceous residuals burn out and gas out which slightly increases mass loss.

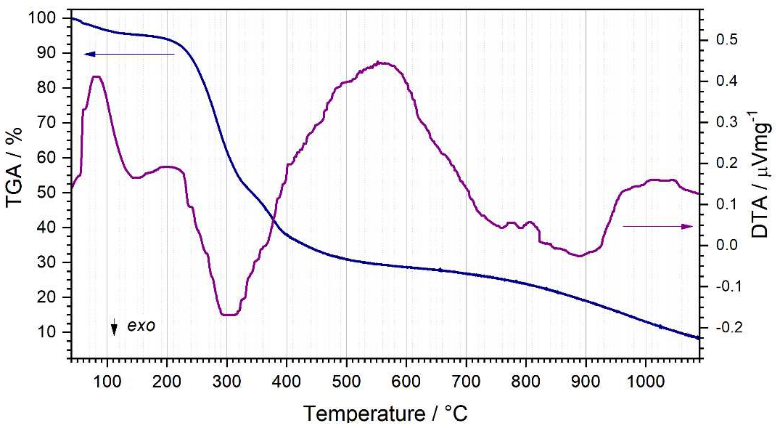

Figure 2. Differential Thermal Analysis (DTA) and Thermo-Gravimetric Analysis (TGA) curves of the waste coffee grounds

According to DTA/TGA curves, a sintering process was defined, as shown in Figure 3.

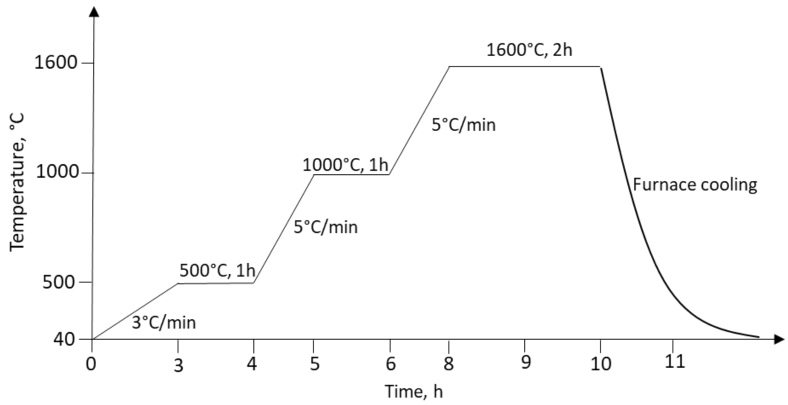

Figure 3. Scheme of sintering regime of alumina ceramics with the addition of waste coffee grounds

Firstly, heating to 500 °C with heating rate of 3 °C/min and 1 h holding time at 500 °C to enable the organic material to burn out. Next, the samples were heated to 1000 °C with heating rate of 5 °C/min and 1 h holding time at 1000 °C for carbonaceous residuals burn out and gas out. Afterwards, the samples were heated to the sintering temperature of 1600°C with heating rate of 5 °C/min and 2 h holding time at 1600 °C. After the cooling of the furnace, sintered samples were taken out for further characterization.

Density of pure alumina samples was measured using Archimedes' principle on analytical balance by Mettler-Toledo. Density of samples with the addition of waste coffee grounds was calculated theoretically using the following equation:

$$\rho = \frac{m}{V} \quad (1)$$

Where *m* (g) is mass of the sintered sample, and *V* (cm^3) is volume of the sintered sample. The obtained density values are shown in Table 2.

Table 2. Measured and calculated density of sintered ceramic samples

w (WCG in powder mixture), %	Density, g/cm^3
0	3.759
1	3.089
5	2.496

The results of density measurements showed that the density decreased with an increase of the amount of waste coffee grounds added. This trend could indicate the formation of pores in sintered ceramic samples due to the burn out of waste coffee grounds. For confirmation of pore formation, further characterization is planned in form of pore size analysis by means of Brunauer-Emmett-Teller (BET) theory from absorption-desorption isotherms.

CONCLUSIONS

Porous alumina ceramic samples were successfully prepared with the addition of waste coffee grounds as a pore-forming agent. Highly concentrated alumina suspensions were prepared with 1 and 5 wt.% of waste coffee grounds. Viscosity of the prepared ceramic suspensions decreased with an increase of the shear rate, while an increase in the amount of waste coffee grounds leads to an increase of viscosity.
DTA/TGA analysis of waste coffee grounds revealed multistep endothermic and exothermic events related with evaporation and release of the moisture, burning of the organic material and subsequent release of the decomposition residuals as well as carbonaceous residuals burn outs accompanied with mass loss. According to DTA/TGA measurements, a suitable course of thermal treatment for sintering was implemented. Density of obtained sintered ceramic samples was measured and calculated. With an increase in the amount of waste coffee grounds, the density decreased which indicated the possible generation of pores during burn out of waste coffee grounds during the sintering process. Further characterization is necessary for confirmation of abovementioned conclusions regarding pore formation.

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