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A novel gelcasting of alumina suspension using curdlan gelation

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Abstract

A novel nontoxic system for gelcasting of alumina suspensions was investigated using curdlan as gelling agent. The rheological and gelling behavior of curdlan suspensions in the temperature range 10–85 °C either on heating or cooling were studied. Influences of concentration of curdlan and solid loading of alumina suspension on the rheological properties and gelling behavior of alumina suspension were investigated. Complex shaped green bodies with compressive strength around 3.7 MPa were obtained by heating 50 vol% alumina suspensions with 0.625–0.750 wt% curdlan (based on alumina powder) at 80 °C for 1 h and by subsequently cooling to room temperature. Dense alumina parts with relative density above 98% have been prepared by curdlan gelation with homogeneous microstructures and good mechanical properties.

Keywords: C. Mechanical properties; Gelcasting; Alumina; Curdlan; Rheology

1. Introduction

Colloidal process of ceramics leads to more uniform microstructure with smaller defects size which increases the reliability of ceramics in engineering aspects [1]. As a near-net-shape colloidal forming method combining slip processing with polymer chemistry, gelcasting has been used to prepare high-quality and complex-shaped ceramic parts by means of situ gelation through which a macromolecular network is created to hold the ceramic particles together [2,3]. Green bodies of gelcasting possess a high compressive strength up to 40 MPa which allows them to be easily machined before sintering, for example by a conventional lathe machine with a high-speed cutting tip [4,5].

The original report on gelcasting used acrylamide as basic monomer and gelation was achieved via polymerization assisted with cross-linker. Experiments have demonstrated that the acrylamide system can be easily controlled during gelcasting due to its good gelation properties. However, the main component of the monomer acrylamide system is a neurotoxin, which limits the application of acrylamide system [6]. To reduce the environmental pollution, low-toxicity or the nontoxic gelcasting systems have been developed to replace acrylamide. Low toxic systems, such as hydroxyethyl methacrylate (HEMA) [7], glycerol monoacrylate [8], acrylic acid [9], N,N-dimethyl acrylamide (DMAA) [10] and epoxy resin [11], have been developed in recent years. More recently, a novel and simple method for gelcasting of ceramics had been developed using a nontoxic and water-soluble copolymer of isobutylene and maleic anhydride (commercially called Isobam) [12,13]. Wang et al. reported a thermoresponsive gel system of poly(N-isopropylacrylamide) gel which had been applied for gelcasting of alumina suspension [14]. The gelling systems reported above are synthetic monomers which are gelated by the use of catalysts or initiators.

Environmentally friendly natural gel-formers have also been applied to the gelcasting of ceramics. Their gelation takes place with changes in temperature without the use of catalysts or initiators, which is one of the advantages compared with the synthetic monomer systems. Natural gelling systems, such as agarose [15], carrageenan gums [16], egg white [17], chitosan [18], gelatin [19,20], have been extensively investigated in the last few years. A novel nontoxic system for gelcasting of ceramics was also investigated using gellan gum as gelling agent assisted by divalent cations [21].

Curdlan is an extracellular bacterial polysaccharide composed entirely of (1,3) -ß-D-glucosidic linkages. It is insoluble in water or acidic solution. Curdlan can be suspended in water and produce gels via the heating of the suspension. However, it

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is soluble in alkali and gels can alternatively be formed, in the absence of heating, simply by the neutralisation of such solutions. Gels similar in character to these alkali-neutralised gels can be formed by heating aqueous suspensions to around 55 °C and subsequently cooling to ambient temperatures. Such gels have customarily been referred to as low-temperature or low-set gels, while heating samples at sufficient concentrations to higher temperatures (above 80 °C) can yield a more resilient, stronger gel [22–24]. Curdlan has been used to fabricate porous ceramics for building materials. The results indicated that the in situ gelation method using curdlan as a promising technique to prepare porous building materials [25].

The aim of this work is to research the use of curdlan as gelling agent for colloidal ceramic processing. The influences of concentration, temperature and pH value on the rheological and gelling properties of curdlan suspensions were studied. Curdlan was applied to alumina forming as a non-toxic galcasting system. Influences of concentration of curdlan and solid loading of alumina suspensions on the rheological properties and gelling properties of alumina suspensions were investigated. The properties of the green bodies and sintered ceramics were characterized.

2. Experimental procedure

2.1. Materials

A CT3000SG alumina powder (Almatis, Ludwigshafen, Germany) with an average particle size of 0.33 μm and a specific surface area of 8.08 m^2/g was used to prepare the concentrated alumina suspension evaluated in this study. Triammonium citrate (TAC) purchased from Guoyao, Beijing, China, was used as the dispersant. Curdlan (Beijing JINNUO-CHENG Chemical Co., Beijing, China) was used to be the gelling agent. The chemical formula of curdlan is $(C_6H_{10}O_5)_n$ [26], shown in Fig. 1. Deionized water was used in all preparation process.

2.2. Processing details

The purchased curdlan is gel particles with an average size of 35 μm. The size is too large for suspension preparation. In order to make curdlan well-dispersed in water, curdlan suspensions with concentration of 1–5% (w/v) were prepared by ball milling for 24 h. Then the curdlan suspensions were used to prepare alumina suspensions. The process for alumina ceramics prepared by curdlan gelation was illustrated in Fig. 2. Alumina suspensions with solid loadings of 40–50 vol% were prepared by tumbling the alumina powder, curdlan suspensions and the dispersant in polyethylene containers for 24 h. For the suspensions with negatively charged ions on the surface of the alumina particles, 0.3 wt% triammonium citrate based on alumina powder was used as the dispersant. Zirconia balls with a diameter of 5–10 mm were used as grinding media. The mass ratio between grinding media and alumina powder was 1:2. The suspensions were degassed in vacuum condition for 15 min at room temperature and then cast into a plastic mold. The cast samples were prepared by heating the alumina suspension to 80 °C for 1 h. The samples were put in the air at room temperature for 2 h, and then demolded. The green body was dried carefully at room temperature for 24 h, heated in an oven at 80 °C for 24 h. The sample was then sintered at 1550 °C for 2 h with a heating rate of 3 °C/min in the temperature range 25–500 °C and 5 °C/min in the temperature range 500–1550 °C.

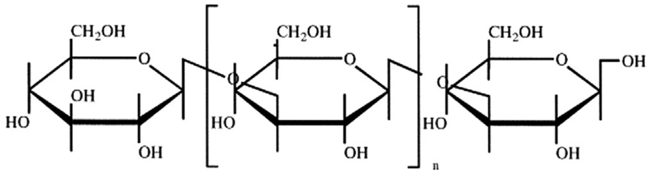

Fig. 1. Chemical formula of curdlan.

2.3. Characterizations

The rheological properties of the curdlan and alumina suspensions were measured using a KINEXUS rheometer (Malvern Instruments, UK). In all viscosity measurements, the shear rate was set at 100 s^{-1}. The measurements were taken at temperatures ranging from 10 °C to 85 °C, and the heating or cooling rate was 5 °C/min. The scanning time was 10 s per point. Thermotropic behavior of green body with addition of curdlan was investigated by a DSC-2910 differential scanning calorimeter (TA Instruments, USA) equipped with a thermal controller. Cylindrical bodies with 25.5 mm in diameter and a height between 25 and 30 mm were cast for wet compressive strength measurements. The wet green strength was measured by an AG–IC20KN (Shimadzu, Japan) testing machine with a crosshead speed of 0.5 mm/min. The dried samples were sintered at 1550 °C for 2 h. The samples were cut into 3 mm × 4 mm × 36 mm and then grinded. The samples were polished by serial diamond rubbing powders, W40, W28, W14, W7 and W1 (Henan Tingxin Abrasives Co., Zhengzhou, China). The bending strength was measured with a mechanical machine

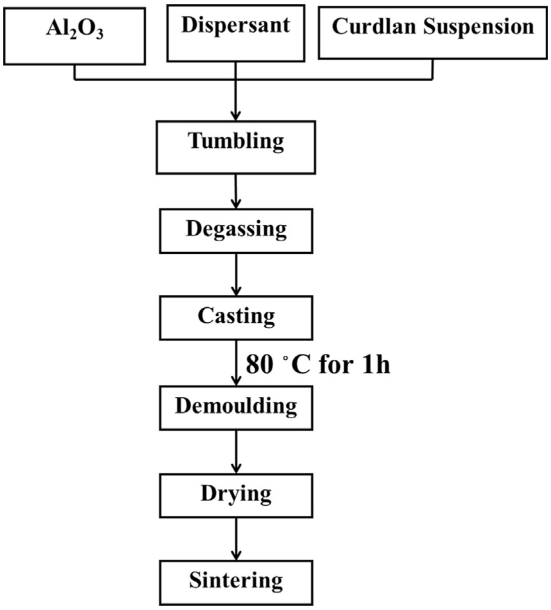

Fig. 2. Gelcasting process of alumina suspension by curdlan gelation.