Ceramics International 42 (2016) 13682–13688

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

2-carboxyethyl acrylate as a new monomer preventing negative effect of oxygen inhibition in gelcasting of alumina

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ARTICLE INFO

Article history:
Received 28 April 2016
Received in revised form
23 May 2016
Accepted 25 May 2016
Available online 26 May 2016

Keywords:
Gelcasting
Oxygen inhibition
Alumina
Acrylates
Low-toxic monomers

ABSTRACT

The paper presents the application of 2-carboxyethyl acrylate as a new monomer in gelcasting of alumina. The research showed that the use of 2-carboxyethyl has a positive influence on the properties of green bodies, limiting the negative effect of oxygen inhibition. Oxygen inhibition which hampers polymerization reaction is a well known negative phenomenon in gelcasting but still not fully overcome for samples formed in air atmosphere. As a reference monomer 2-hydroxyethyl acrylate was used in the research. Rheological behavior of alumina suspensions containing both monomers has been studied, as well as the properties of green and sintered bodies obtained by gelcasting have been measured. The differences in the rheological properties and thus ceramic-monomer interactions in the slurries has been analyzed. The high values of densities and mechanical strength, accompanied with the pictures of microstructures of sintered ceramic parts have been presented, as a result of a successfully completed gelcasting process of alumina powder.

1. Introduction

Gelation processes are commonly used in consolidation of ceramic powders. One of the methods based on gelation is gelcasting – a forming process which has been recognized as the very attractive route in the fabrication of elements of complicated geometry and of small series of pieces.

Gelcasting technique was developed in 1990s by Omatete and Janney from the Ceramic Processing Group at Oak Ridge National Laboratory (ORN), USA [1]. Gelcasting combines inorganic and polymer chemistry. Thanks to the combination of ceramic powder and organic additives it is possible to obtain high-quality complex-shaped and homogenous ceramic parts by means of an in situ polymerization or the gelation of polysaccharides or proteins [2–4]. In the process the macromolecular gel network which results from the in situ polymerization of organic monomers added to the slurry is created and holds ceramic particles in the desired shape. The main challenge in the gelation process is to prepare homogenous, highly concentrated and stable ceramic suspension which leads to high particle packing in the green state and full densification upon sintering [5–8]. Gelcasting allows to fabricate both dense and porous materials [9]. Gelation process has been also employed in the preparation of thin films, as a combination with tape casting technique and also for the fabrication of textured ceramics as a combination with magnetic field alignment method [10,11]. The main advantage of gelcasting process is the possibility to conduct the process at room temperature without the use of high-pressure apparatus.

The key role in this process is played by the suitable selection of organic additives such as monomers, dispersants, activators and initiators of polymerization. These substances influence on the rheological properties of ceramic suspensions and mechanical strength of green bodies. One of the most commonly used monomers in gelcasting is commercially available acrylamide which is toxic and probably carcinogenic. Therefore scientist started to replace it by methacrylamide which is less toxic. Nevertheless green bodies based on this substance exhibited worse mechanical strength. Third commonly applied water-soluble monomer is 2-hydroxyethyl acrylate (HEA). All mentioned substances are often used together with the external cross-linking agents what increases the total content of the organic phase in green bodies [12–15].

Oxygen is a known inhibitor of radical polymerization. Oxygen inhibition causes exfoliation of ceramic samples and decreases their mechanical strength due to lack of polymeric binders which would hold ceramic particles together. The problem of oxygen inhibition in gelcasting of ceramics is known and partially reduced by replacing air atmosphere by CO_2, N_2 or vacuum, but still not fully solved [16–18]. Shaping under nitrogen atmosphere is promising but complicated from the technological point of view, while vacuum only partially prevents from the exfoliation. For these reasons the Authors were searching for monomers which

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http://dx.doi.org/10.1016/j.ceramint.2016.05.166

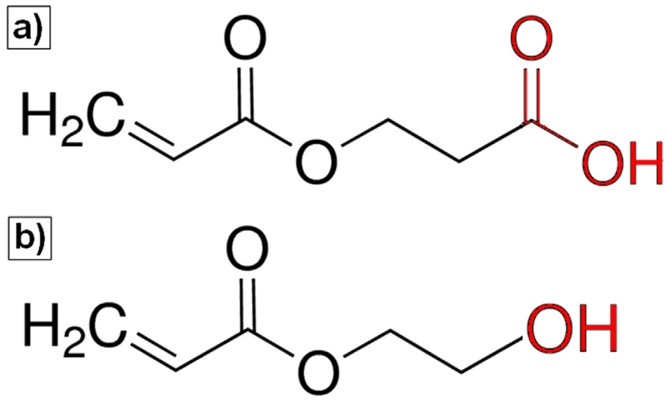

Fig. 1. Chemical formulas of: 2-carboxyethyl acrylate (a) and 2-hydroxyethyl acrylate (b).

would allow to overcome the above limitations. The promising alternative is the new monomer 2-carboxyethyl acrylate (CEA). This low toxic, water-soluble monomer modifies the rheological properties of the ceramic suspensions and additionally minimizes the negative effect of oxygen inhibition. 2-carboxyethyl acrylate (CEA) was chosen because of its molecular structure. Authors excepted that the presence of the carboxyl group in CEA molecule could have positive influence on the rheological behavior of ceramic suspensions and as a consequence the high quality of samples.

The aim of the research was therefore the use of 2-carboxyethyl acrylate (Fig. 1a) as a new organic monomer in gelcasting of alumina. Rheological properties of the slurries have been measured as well as the mechanical strength, density and eventual exfoliation of green bodies have been determined. The properties and microstructures of the sintered samples have been also analyzed. For comparison, analogous measurements have been done for samples containing polymerized 2-hydroxyethyl acrylate (Fig. 1b).

2. Experimental procedure

2.1. Materials

The ceramic powder used in the research was α-Al_2O_3 A16SG (Almatis) of the average particle size $D_{50}=0.5$ μm, specific surface area of 7.5 m^2/g and density of 3.90 g/cm^3. Deionized Milli-Q water was used as a solvent. Commercially available 2-carboxyethyl acrylate, CEA (Sigma-Aldrich) and 2-hydroxyethyl acrylate, HEA (Sigma-Aldrich) were used as organic monomers, SYNTRAN 8250 was used as dispersing agent, L-ascorbic (Sigma-Aldrich) acid was an activator of the polymerization and ammonium persulphate, APS (Sigma-Aldrich) was the initiator of the polymerization. According to the information given by the supplier (Interpolymer Company) SYNSTRAN is a medium molecular weight aqueous solution based on polyacrylic acid homopolymer and was used in the form of 40% aqueous solution. The initiator was used in the form of 10% aqueous solution.

2.2. Preparation of slurries and samples

The ceramic samples were obtained according to the method schematically shown in Fig. 2. The first step was the preparation of the ceramic slurry in alumina containers. Dispersant was first dissolved in deionized water. Then, the appropriate amounts of ceramic powder and activator of polymerization were added and after prior homogenization the monomer was dropped into the slurry. Preliminary works showed that dissolving monomer in a solvent before the addition of alumina powder has negative influence on the rheological properties of ceramic slurry (high

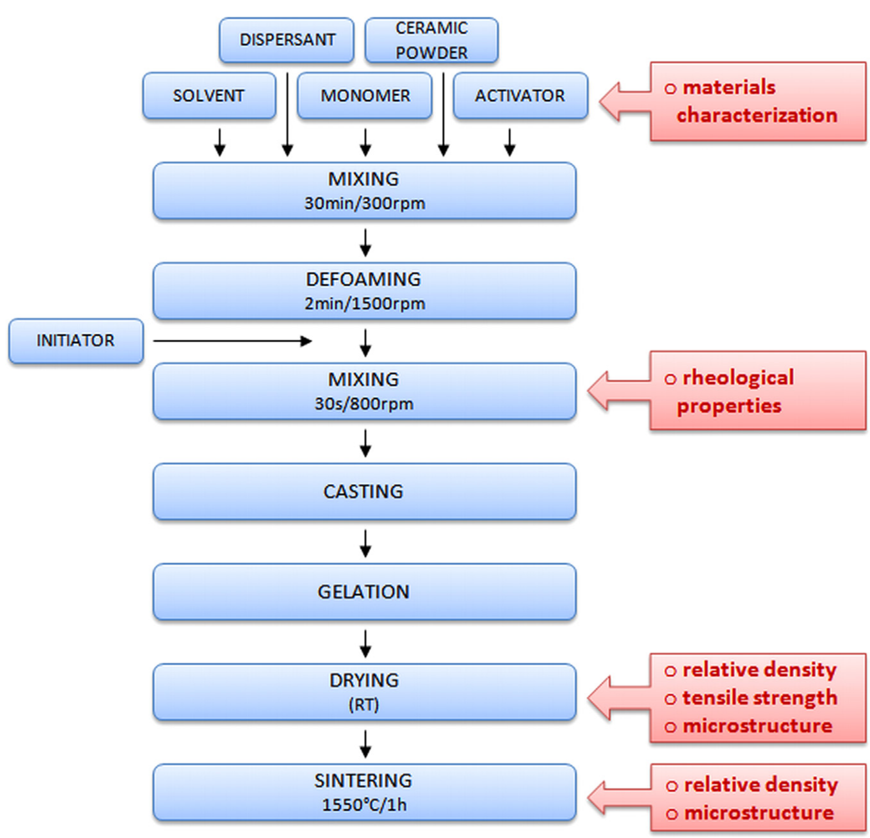

Fig. 2. The scheme of the gelcasting process used in the research.