



## Gelcasting as a Novel Processing Route to Fabricate Partially Stabilized Zirconia Ceramic Bodies

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### Abstract

Bioinert ceramics, like alumina and zirconia are used mainly for replacements of bones, hip joints and for dental implants. Partially stabilized zirconia ceramics (PSZ or TZP with 3 mol.% or 5 wt.%  $Y_2O_3$ ), appear as perspective bioinert ceramics because of their high strength and corrosion resistance. In order to fabricate complex shapes, it is essential to use a near net shape processing like gelcasting as an uncomplicated method which has appropriate potential of producing special shapes with suitable green and sintered properties (like high mechanical properties and almost full density and machinability of green bodies). Moreover, biocompatibility studies have proved that this processing route is nontoxic. In this experiment, microstructural observation performed to illustrate the effect of dispersant on homogeneity of gelcast 3Y-TZP bodies. It has been shown that it is possible to achieve uniform microstructure by means of appropriate amount of dispersant and ultrasonic wave. In addition, the machinability of these green bodies has been proven and some complex shaped bodies fabricated, in order to illustrate the capability of the process.

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### 1. Introduction

Bioceramics are more biocompatible with an organism than other implanted materials and have less effect on the immune system [1]. Broadly, bioceramics are divided in three main groups: resorbable (like tri-calcium phosphate), bioactive (like hydroxyapatite) and bioinert materials (like yttria-stabilized  $ZrO_2$ ). Bioinert ceramics, which have to satisfy

strict demands concerning mechanical and corrosion properties, chemical purity and biocompatibility, are used mainly for replacements of bones, hip joints and for dental implants [2]. Since Yttria-stabilized  $ZrO_2$  has high chemical bonds, which leads to high strength and chemical stability in corrosive environments like human body, it has become very attractive in biomedical and especially dental applications [3].

Among the main advantages of zirconia materials are their high values of mechanical strength and fracture toughness, which are attainable due to the phase transformation

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from the tetragonal to the monoclinic phase of  $ZrO_2$  [4]. The resulting mechanical strength of the material, however, depends largely on the selection of the starting powder and on the final microstructure of the body, which is a result of processing. As well, the processing route must be able to fabricate a homogeneous microstructure [3].

In this paper, a novel near-net-shape forming technique called gelcasting is employed to produce partially stabilized ceramic bodies. In this method, ceramic bodies are fabricated by means of *in situ* polymerization through which a macromolecular network is created to hold the ceramic particles together. Because of this special forming mechanism, the system contains only a small amount of the binder. The process enables slurries with high solid loading and strong, machinable green bodies to be produced [5]. Also, this process has potential for forming porous ceramics and has been used to create replamine-form inspired bone structures (RIBS) [6]. Moreover, biocompatibility tests' results, performed by Darsell et al. [7], have shown that sintered gelcast-bodies produced from exactly the same raw materials were not toxic; therefore, gelcasting is an appropriate process to

fabricate bio-ceramics.

## 2. Materials and methods

Partially stabilized zirconia powder (3Y-TZP) was used as bioinert ceramic. The average particle size of the powder was 0.2  $\mu\text{m}$  (Fritsch Particle Sizer, analysette22), and the powder density was 5.65  $\text{g}/\text{cm}^3$  (Micromereticx, AccuPyc1330 V3.00). Dolapix CE64 and Darvan 821A (a 40% aqueous solution of ammonium polyacrylate) were initially tested and evaluated as dispersants. Dolapix CE64 was then selected for further study because of the formation of more uniform suspension after dearing and its lower viscosity. The essential components of the gelcasting process are the reactive organic monomers: Monofunctional acrylamide,  $C_2H_3CONH_2$  (AM) (Merck), and difunctional N,N'-methylenebisacrylamide,  $(C_2H_3CONH)_2CH_2$  (MBAM) (Merck). These monomers were dissolved in deionized water to give premix solution. The premix solution undergoes free-radical initiated vinyl polymerization in the presence of an initiator such as ammonium persulfate,  $(NH_4)_2S_2O_8$ . The reaction is accelerated by heat and the resulting cross-linked polymer is an elastic hydrogel that serves as the binder.

The gelcasting flowchart is presented in Figure 1. A solution of AM and MBAM in deionized water with AM/MBAM ratio of 25/1 was first prepared as the precursor. The 3Y-TZP suspension was prepared with 45 vol% of the ceramic powder. The dispersant amount used for this purpose was 0.6 wt% and 0.4 wt% by total weight of suspension. The suspension was first stirred for 5 min. For comparison purposes, some of this suspension was placed in sonicator for 6 min to break weak agglomerations, and the other part of suspension was left without any further procedure. These two different suspensions were vacuumed in 200 mbar pressure. Then, 0.04 wt% of the initiator  $(NH_4)_2S_2O_8$  was added to each suspension, and they were poured in appropriate moulds. The gelation

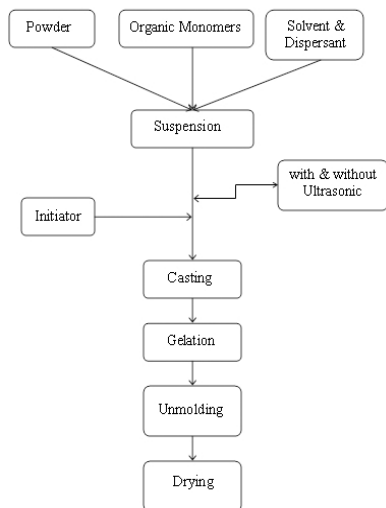
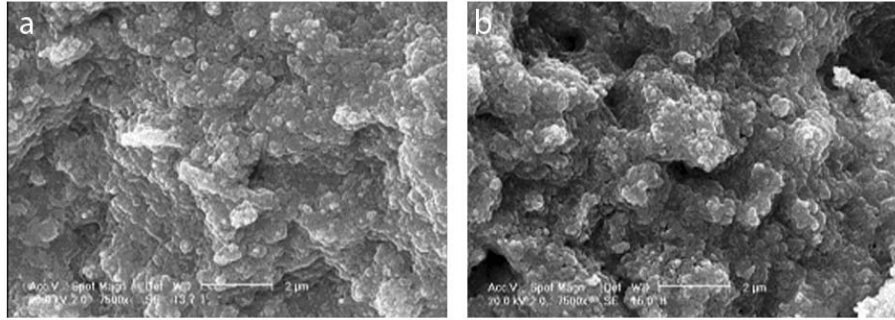


Figure 1. The gelcasting flowchart.



**Figure 2.** Figures show green microstructure of gelcast green bodies 2(a) with and 2(b) without using ultrasonic waves with 0.4 wt% of dispersant.

process was accelerated by temperature (about 50 °C). All specimens were first dried in 50 wt% solution of polyethylene glycol in water to avoid cracking in primary stages of drying and then they were dried in air. Microstructural observation was performed with SEM (XL30 Philips, Netherlands).

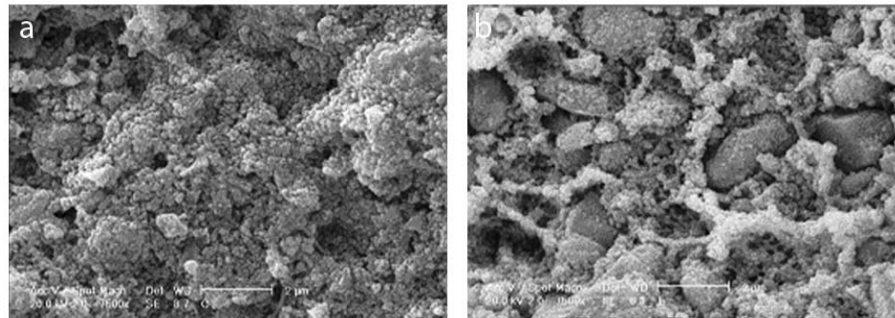
### 3. Results and discussion

#### 3.1. Green microstructure

The differences in the pore size, distribution and degree of densification in the gelcast bodies could be also visualized by microstructural observations, as shown in Figures 2 and 3. Application of ultrasonic waves on suspension, significantly improved homogeneity of the green body, while several entrapped pores and also weak agglomerations existed in the gelcast body without using ultrasonic waves. The entrapped pores and heterogeneity will lead to low green density and defects in the sintered products. Figures 2(a) and 2(b) show fracture

surface microstructures of gelcast green bodies with and without using ultrasonic waves with 0.4 wt% of dispersant, respectively, and Figures 3(a) and 3(b) show these microstructures for 0.6 wt% of dispersant.

Figures 2(a) and 3(a) represent homogeneous microstructure with using ultrasonic waves to break weak agglomerations and disperse the particles in the body, in contrast to some pores and agglomerates without using ultrasonic waves as mentioned in Figures 2(b) and 3(b). However, in Figure 3(b) the cluster consisted of particles was observed which was formed due to different amounts of dispersant. Even though the gelcast bodies with and without using ultrasonic waves showed much difference, it is clear that more homogeneous microstructure could be obtained with using ultrasonic waves.



**Figure 3.** Figures show green microstructure of gelcast green bodies 2(a) with and 2(b) without using ultrasonic waves with 0.6 wt% of dispersant.



