

# Application of Some Natural Gelling Agents in Injection Molded Dense Ceramics

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## Abstract

Gelcasting, as a technique for near-precision shaping, concurrently satisfies the attributes of intricate form, heightened density, energy-efficient and eco-friendly preparation protocols. Its framework progressively matures. Gel precursors command a substantial share in the process, inimitable in their role, exerting a direct influence on the performance of the fabricated specimens. The conventional AM-MBAM system bears a toxic nature, thus, the quest for aptly low-toxic or non-toxic gel precursors has surged as a fervent area of inquiry. Natural gel precursors, distinguished by their low toxicity and non-toxic disposition, have found extensive utility in contemporary studies of gelcasting. This manuscript expounds upon the procedural intricacies and fundamental mechanisms of gelcasting, while also delineating the predominant characteristics of presently favored natural gel precursors.

## Keywords

Gelcasting; Natural Gel; Isobam; Dense Ceramics.

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## 1. Introduction

In recent years, with the continuous refinement of our nation's policies towards the manufacturing sector and its development, there has been an elevation in not only the efficiency and quality expectations concerning material preparation but also a heightened emphasis on energy conservation and environmental impact during the preparation process. The significance of green material preparation processes is second only to the production of high-performance and top-quality materials. Powder metallurgy technology has found extensive application across various industries in our daily lives and has emerged as one of the most pivotal cornerstones in the realm of modern materials science in our country. Traditional methods of shaping include powder compaction, cold isostatic pressing, powder rolling, powder extrusion, and powder explosive forming, with powder compaction being the most widely employed technique within the manufacturing sector presently. However, while powder compaction can yield high-density preforms, it has gradually been supplanted by other shaping methods due to issues such as its fixed and singular shape and uneven powder distribution.

Injection molding, on the other hand, is an almost net-size molding process that simultaneously embodies characteristics of intricate form, high density, energy efficiency, and environmental preservation. It was introduced in the early 1990s [1-3] and has steadily ascended as a focal point of research over the past few decades [4-7].

Initially, injection molding was primarily used for the production of dense materials. However, in recent years of research, porous materials have gradually emerged as the mainstream in injection molding. Through meticulous process control, injection molding has demonstrated its capability to fabricate materials with highly intricate forms [8-9]. The injection molding process itself is not overly complex; for instance, the removal of organic materials does not require additional steps but can be

accomplished during the sintering process[10], rendering it conducive to large-scale production[11]. The uniformity of materials produced via injection molding is notably high, owing to its molding methodology.

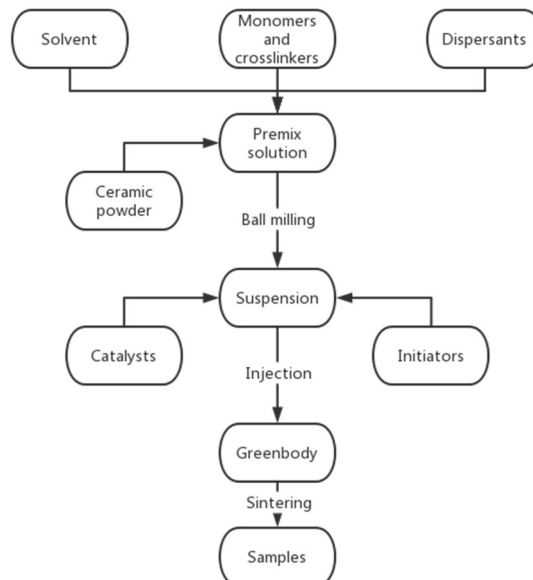
The initial incarnation of injection molding was the AM-MBAM system[12], which employed acrylamide (AM) as the monomer, methylene bisacrylamide (MBAM) as the crosslinker, ammonium persulphate as the initiator, and tetramethylethyl-ethylenediamine (TEMED) as the catalyst. This system created a three-dimensional mesh structure encapsulating ceramic particles using AM and MBAM polymers. However, due to the neurotoxicity associated with AM and society's growing environmental concerns, the usage of AM has gradually diminished. The pursuit of environmentally friendly, low-toxicity, or non-toxic alternatives to AM has become a prominent topic in injection molding research. Consequently, numerous synthetic monomers, as well as natural and biopolymer materials such as agar, agarose, gelatin, albumin, chitosan, and sodium alginate, have been investigated and tested[13-15].

In recent years, ISOBAM®, a water-soluble alternating copolymer composed of isobutylene and maleic anhydride, has garnered significant research attention. Transparent ceramics produced with ISOBAM® as an additive exhibit a dense and uniform microstructure, low organic content, low shrinkage, and porosity, even at high solid content levels of up to 80%[16-21].

This paper aims to provide a comprehensive review of research on the injection molding process of dense ceramics produced from single-phase or composite powders, with a focus on environmentally benign additives.

## 2. Dense Ceramics

As previously mentioned, injection molding offers distinct advantages over other traditional shaping methods. Its process flow is illustrated in Figure 1:



**Figure 1.** The process of gelcasting

Firstly, a mixture of solvent, monomers, crosslinkers, and dispersants is prepared to create a premix solution.

Ceramic powder is introduced to form a high-solid-content solution.

The powder is uniformly dispersed in the solution through ball milling, creating a suspension.

Catalysts and initiators are added to the suspension after ball milling and injected into molds.

The suspension gradually solidifies within the molds, taking shape.

Upon solidification, a green body is formed and demolded.

Drying takes place under controlled temperature and humidity conditions, followed by debinding (if necessary) and sintering.

In order to fabricate ceramic materials that meet the prescribed high-density, flawless criteria, every parameter must be rigorously governed and honed to perfection, particularly with regard to pivotal variables such as monomers and cross-linking agents. The majority of research endeavors are, indeed, centered around this very pursuit.

### 2.1 Non-toxic Monomers and Adhesive Agents with Low Toxicity.

As previously mentioned, the recent introduction of ISOBAM, a low-toxicity, water-soluble substance, has generated substantial interest[16-21]. This compound possesses the remarkable ability to serve as a monomer, crosslinker, and dispersant even at low concentrations. As depicted in Figure 2[27], the addition of ISOBAM triggers a significant shift in the suspension's isoelectric point towards the acidic region. Moreover, within the pH range of 8-11, the zeta potential of the suspension containing ISOBAM is lower than that of a dispersant-free suspension. This phenomenon can be attributed to the hydrolysis of ISOBAM in the suspension, leading to the formation of -COO- groups that readily react with the positive charges or -OH groups on the surface of BST50 particles. Additionally, the -CONH2 groups in ISOBAM can effectively adsorb onto the particles through hydrogen bonding[28].

Figure 3 illustrates research conducted by Lu et al.[29] on the modulus curves obtained during the high-temperature gelation process of the ISOBAM system in the presence of triammonium citrate (TAC). They proposed an alternative mechanism, a two-step bridging mechanism. In step one, ISOBAM molecules bridge from one particle to the surface of an adjacent particle, resulting in gelation speeds equivalent to the ISOBAM-only system. However, due to the limited area left uncovered, the gelation rate markedly slows down. Step two takes precedence, wherein ISOBAM molecules from neighboring particles replace TAC, leading to significantly longer gelation times compared to step one. The gelation rate in the second step is influenced by electrostatic repulsion and spatial hindrance, and it can be finely tuned by adjusting the content of TAC, ISOBAM, and ionic strength.

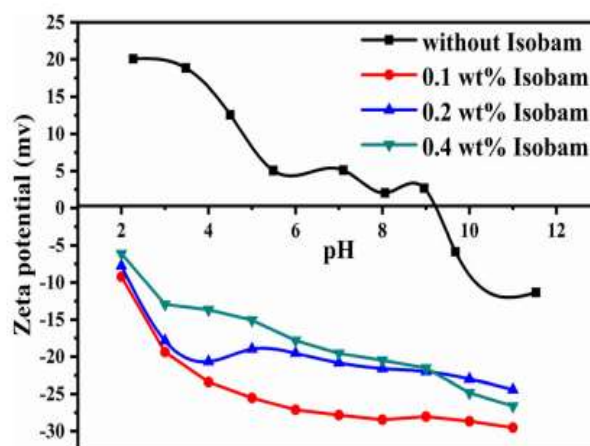
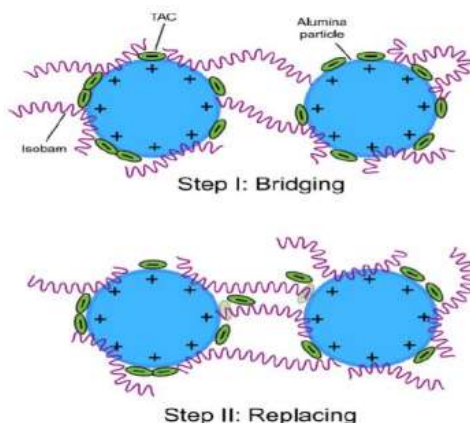


Figure 2. The impact of dispersants on the zeta potential of BST50 suspension[27]

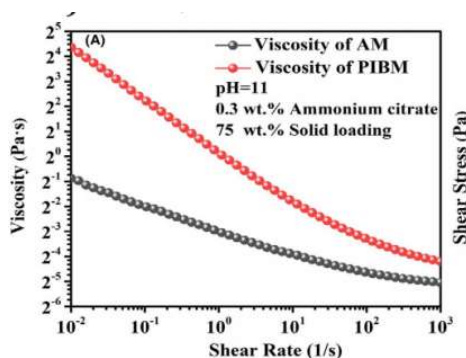


**Figure 3.** The two-step bridging procedure during ISOBAM gelation[29]

## 2.2 Suspension

The suspension is a critical component in the injection molding process, and the rheological properties of the suspension play a vital role. The better the rheological properties of the suspension, the fewer fine pores can be formed in the resulting green body. Consequently, the mechanical properties of the green body are enhanced. Therefore, the rheological properties of the suspension can effectively mirror the mechanical performance of the green body. Hence, it is imperative to strike a balance between low-viscosity suspension and high solid content, which is one of the key aspects of injection molding.

Zeng Jinzhen and colleagues[30] investigated three different morphologies of Al<sub>2</sub>O<sub>3</sub> raw materials and characterized their rheological properties. The results indicated that flat-shaped particles were more conducive to preparing low-viscosity suspensions compared to granular and rod-shaped particles. This was attributed to the unique morphology of flat-shaped particles, which resulted in less particle collision and aggregation in the suspension due to Brownian motion. Additionally, the parallel movement of lamellar particles with respect to the shear plane contributed to a more orderly arrangement of particles. As depicted in Figure 4, although ISOBAM-based systems are non-toxic and can function both as a dispersant and an initiator, when compared to suspensions prepared using the traditional AM system, the latter exhibited higher flow performance before the addition of the initiator. This allowed the former to produce suspensions with higher solid content and lower viscosity. Research by Qing and colleagues[21] revealed that suspensions in the AM system exhibited lower viscosity (0.05 Pa·s at a shear rate of 100 s<sup>-1</sup>), shorter gelation times (92.9 s), and higher flexural strength (approximately 24.46 MPa) compared to ISOBAM-based systems.



**Figure 4.** The rheological properties of suspensions prepared using the AM system and the PIBM system[21]

Gelation time serves as another crucial parameter in suspension, where a delicate balance is essential. When the gelation rate is too low, powder particles tend to settle before gelation, resulting in uneven

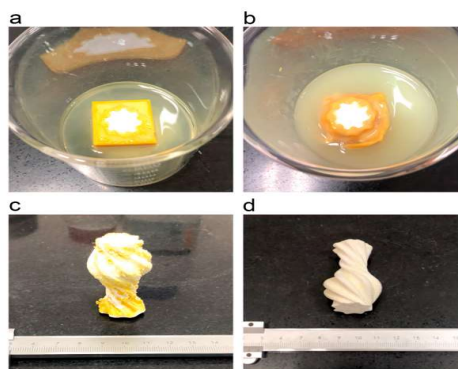
and less dense green bodies. Conversely, an excessively high gelation rate can increase the difficulty of preparation or even cause the suspension to solidify prematurely, leading to flocculation. Three key factors influencing gelation time include monomer content, the ratio of monomer to crosslinker, and temperature. Mahani and colleagues[31] conducted research on these three factors in the injection molding of silver nanoparticles. The results revealed that the monomer-to-crosslinker ratio had the most significant impact on gelation time, with a MBAM/MAM ratio of 1:10 being the optimal balance. Omid Sharifi and colleagues[32] proposed that at a high AM/MBAM ratio, the critical chain length is reached in a very short time, explaining the phenomenon of faster gelation with higher monomer-to-crosslinker ratios.

When the monomer content is relatively low, a higher monomer content leads to a tighter mesh structure formed by the monomer and crosslinker and smaller interparticle spacing, resulting in enhanced mechanical properties of the green body. However, as gelation occurs, two major kinetic parameters come into play: an increase in monomer content and an increase in gelation temperature. This causes the monomer chains to become thicker and shorter, increasing the likelihood of microcrack formation and, consequently, a decrease in the mechanical properties of the resulting green body[33].

The remaining processes involved in preparing the suspension, such as ball milling time and rotational speed, also have a significant impact on suspension viscosity. Suspension viscosity changes with increasing ball milling time: after a short period of ball milling, viscosity decreases, reaching a minimum at a specific point. This is because, after initial ball milling, the suspension may still contain coarser or larger particles, and the adsorption of monomers and dispersants onto the powder may not be fully realized. As ball milling time increases, the powder becomes thoroughly mixed with the solution, and the adsorption of monomers and dispersants onto the powder gradually saturates, leading to an increase in electrostatic repulsion and the formation of spatial hindrance, resulting in reduced suspension viscosity[34].

However, as ball milling time continues to increase, it can lead to a rise in temperature, making organic monomers more prone to polymerization reactions. It can also further refine powder particles, increase their specific surface area, and saturate the adsorption capacity of dispersants, leading to the possibility of aggregation. These factors collectively contribute to an increase in suspension viscosity. In summary, the preparation of a suspension requires a comprehensive consideration of the factors mentioned earlier, and the selection of the most suitable process flow and additives.

### 2.3 Shaping Or Molding



**Figure 5.** Photograph of the dissolution and dismantling of the mold

- (a) An image of the mold immersed for 1 minute.
- (b) An image of the mold immersed for 2 hours.
- (c) A photograph showing the mold after manual removal of softened portions.
- (d) A photograph of the completely dismantled and dried greenware.[35]



Once a qualified suspension is prepared, it needs to undergo the injection molding, degassing, gelation, demolding, and drying processes to form a green body.

The gelation process is a low-temperature process, which can occur within a range from room temperature to 80°C, making the choice of mold materials quite diverse. However, as there will inevitably be a shrinkage process during gelation, especially when preparing complex shapes without affecting the green body during demolding, the characteristics of the mold material need to be considered in conjunction with mold design.

As shown in Figure 5, Liu and colleagues[36] employed Fused Deposition Modeling (FDM) 3D printing technology to create plastic molds that are soluble in acetone. After injection molding and gelation, the demolding process was effortlessly completed by immersing the green body and the mold entirely in acetone, along with simple manual operations. This approach resulted in the production of smooth-surfaced zirconia green bodies without visible cracks.

After determining the mold, the degassing process following the injection of the suspension becomes a crucial step in reducing macroscopic defects in the green body. Typically, there are two methods for degassing: the first, traditional method involves vacuuming after injection, until all air bubbles disappear, which also requires mold designs that facilitate the release of air bubbles. The second method, as exemplified by Liu et al.[35] in a previous section, is to remove the mold directly. Complex molds may even require degassing twice, as demonstrated by Zhao et al.[36], who prepared the suspension, injected it under vacuum, and developed an auxiliary system to expel air from the mold after injection. As mentioned earlier, in polysaccharide systems as gelators, the use of defoamers is necessary to prepare dense ceramics. Chen et al.[37] proposed that in the case of using sucrose as a gelator, the addition of polyethylene glycol (PEG) hardly affects the stability and rheological performance of silicon carbide suspensions but effectively prevents surface flaking of the green bodies.

Once the suspension solidifies, samples prepared using water-based injection molding contain approximately 20% water, necessitating a drying process. The temperature and relative humidity of the drying medium significantly affect the evaporation of water from the green body. Higher temperatures and relative humidity levels during drying lead to faster water evaporation, resulting in more cracks and voids in the green body. Guo et al.[38], after comparing several drying conditions, adopted a three-stage drying method, involving high humidity and low temperature for 10 hours in the first stage, maintaining increased humidity and temperature for 8 hours in the second stage, and high temperature and low humidity for 6 hours in the third stage. This method achieved a drying degree of 97.21% for AlN green bodies without deformation or cracks. Wu et al.[39] successfully overcame cracking issues in the preparation of large-sized samples using a stepwise drying method. However, traditional air drying or controlled humidity and temperature drying can be time-consuming, especially for large green bodies. To address this, shorten drying times, the use of liquid desiccant drying methods has been developed. This involves immersing the undried green body in an appropriate liquid desiccant, allowing chemical potential differences to form between the polymer chains in the desiccant and the solvent in the polymer grid of the green body. Gong et al.[40] compared liquid desiccant drying with traditional drying methods and found that in traditional drying, water evaporates rapidly from the green body's exposed surfaces, while it evaporates very slowly from other surfaces, leading to residual stresses. In contrast, liquid desiccant drying results in nearly uniform drying rates across the entire green body, reducing residual stresses. Additionally, by providing buoyancy to the green body during the drying process, it can minimize or completely eliminate deformation caused by uneven shrinkage.

## 2.4 Green Body and Sintered Bodies

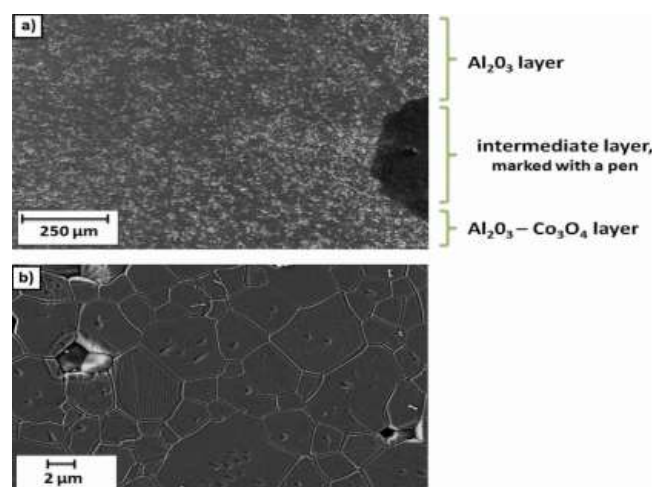
In order to prepare uniform and highly dense green bodies, maximizing the solid loading of the suspension is crucial to achieve the required high relative density after sintering. Gai et al.[41] analyzed the effect of various dispersant concentrations on the zeta potential of SiC ceramic suspensions and studied the relationship between SiC rheological properties and solid loading in the

suspension. The results indicated that when using polyethyleneimine as a dispersant, 0.2 wt% was the optimal concentration, allowing the suspension to achieve the highest solid loading of 50 vol%. Wang et al.[42] conducted repeated experiments and adjusted the particle size distribution of the raw materials, involving coarse, medium, and fine particles to fill each other. They increased the solid loading of a fused quartz suspension from 70% to 84%, resulting in a sintered sample density of 1.83 g/cm<sup>3</sup> and a flexural strength of 43.62 MPa, with tightly packed particles.

Traditional polysaccharide hydrogels like agarose, due to their high viscosity and low water solubility, can only be used for low-concentration injection molding, typically not exceeding 4 wt%. However, Santacruz et al.[43] dissolved agarose at high pressure, preparing a 5.6 wt% agarose solution, improving dissolution efficiency, reducing suspension viscosity, and allowing the solid loading of ceramic powder in the suspension to increase from 80 wt% to 84 wt% (equivalent to 50 vol% to 55 vol%).

When the solid loading of the suspension is relatively high, relying solely on ISOBAM-based dispersants may not achieve the desired dispersion effect. Lv et al.[44] addressed this issue by adding TAC to assist in dispersion. However, this approach reduced the strength of the green body and even led to collapse. The study explored three different dicarboxylic acids added to TAC and ISOBAM bases to enhance suspension performance. Results showed that for a solid loading of 52 vol% Al<sub>2</sub>O<sub>3</sub> suspension, phthalic acid (PA) reduced viscosity by approximately 30%. However, when the solid loading increased to 54 vol%, oxalic acid (OA) became the preferred additive, reducing viscosity by about 37%. When a suspension with 54 vol% solid loading was prepared with 0.5 wt% ISOBAM, 0.04 wt% PA, and 0.03 wt% OA, the flexural strength reached 448 ± 44 MPa. Ding et al.[45] significantly improved the gelation rate of AlN suspensions in the ISOBAM system by adding an appropriate amount of APS as an additional dispersant. After adding 1 wt% APS, the viscosity of a 50 vol% AlN suspension at a shear rate of 100 s<sup>-1</sup> was approximately 0.21 Pa·s, and the flexural strength of the green body reached 2.5 MPa.

Wiecinska et al.[46] proposed a new method where they re-injected the surface of the gelled green body multiple times, resulting in multi-layered dense ceramics composed of Al<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub>-Co<sub>3</sub>O<sub>4</sub>. This involved alternating layers of two Al<sub>2</sub>O<sub>3</sub> layers and two Al<sub>2</sub>O<sub>3</sub>-Co<sub>3</sub>O<sub>4</sub> layers. When studied, the multi-layered samples exhibited higher tensile strength in both the green body and sintered body compared to single-layer samples. As shown in Figure 6, there was good bonding between the layers without additional delamination, and the Al<sub>2</sub>O<sub>3</sub>-Co<sub>3</sub>O<sub>4</sub> layers exhibited good physical properties.



**Figure 6.** SEM images of the multilayered sample,  
(a) The interconnection between the two layers,  
(b) The microscopic structure of the Al<sub>2</sub>O<sub>3</sub>-Co<sub>3</sub>O<sub>4</sub> layer.[46]

### 3. Conclusion

This passage delineates the evolving trends in the injection molding process of dense ceramic materials, encompassing the utilization of natural gelling agents within said process. Research into low-toxicity and non-toxic monomers is well underway, with these gelling agents having been verified as viable contenders alongside AM system's original monomers. However, for natural gelling agents to surpass AM systems comprehensively, substantial research is still required concerning pivotal processing parameters within the injection molding process, such as those pertaining to molding and drying techniques.

The majority of low-toxicity and non-toxic gelling agents can be processed within a water-based gel system. The primary objective of this research is to substantiate the environmental friendliness and multifunctionality of injection molding. These agents are employed at exceedingly low concentrations and do not necessitate a separate step for removal during combustion. Natural gelling agents have indeed been confirmed as capable of crafting high-density ceramics with both simple and intricate structures, including advanced materials like transparent polycrystalline ceramics.

In the nearly past decade of development within the injection molding process, substantial progress has been achieved in the healthcare sector. Endeavors are being made to avoid the use of carcinogenic and neurotoxic substances akin to those within AM systems. Instead, there is a shift towards employing less harmful materials such as ISOBAM® – a patented water-soluble copolymer of isobutene and maleic anhydride – and food-grade biopolymers like polysaccharides and proteins. ISOBAM can function both as a dispersant and gelling agent, reducing the concentration of organic substances to exceptionally low levels and giving rise to unique polycrystalline transparent ceramics like yttrium aluminum garnet (YAG). Biopolymers, on the other hand, enable the creation of dense, high-strength ceramics with properties nearly identical to traditional methods such as AM system injection molding. These gelling agents require minimal quantities to attain the necessary green body strength, eliminating the need for de-gelling before sintering, coupled with their low toxicity, rendering them potentially applicable within an industrial context.

Nevertheless, despite the aforementioned developments, the utilization of low-toxicity and non-toxic gelling agents in the injection molding process still presents certain limitations:

The high viscosity and low solubility of certain natural gelling agents result in a lower maximum solid content of ceramic powder within the suspension compared to traditional AM systems. Consequently, the green bodies produced by the former exhibit relatively low density and necessitate meticulous control of the drying process to prevent cracking and deformation, thereby elongating the drying period.

Some natural gelling agents exhibit a degree of foaming, requiring the incorporation of an appropriate de-gassing step before and during the injection molding process, in conjunction with the addition of specific defoaming agents.

The elevated cost of natural gelling agents imposes constraints on large-scale industrial production.

Should these aforementioned challenges be effectively addressed, injection molding in China could achieve environmentally sustainable industrial-scale production, significantly impacting the production of intricate high-density ceramics in our country.

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