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Research Article

Optimization of Gel Casting Parameters for High-Strength Fused Silica Green Bodies

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ABSTRACT

The gel casting of fused silica offers a promising approach for producing complex-shaped advanced ceramic components. Achieving a defect-free process, however, requires precise optimization of slurry formulation and gelation parameters. This study systematically investigates the effects of key processing variables on slurry rheology and green body strength, including dispersant concentration, solid loading, monomer-to-crosslinker (AM:MBAM) ratio, initiator concentration, and degassing time. Results indicate that a dispersant concentration of 0.4 ml per 100 g of powder minimizes viscosity, while a solid loading of 58 wt.% represents the upper limit for maintaining slurry stability. A balanced AM:MBAM ratio is critical to achieving both low viscosity during casting and high mechanical strength after gelation, as excessive crosslinker promotes premature microcracking. Furthermore, an initiator (APS) concentration of 0.5 wt.% and a degassing time of 20 minutes maximize green strength by ensuring complete polymerization and eliminating porosity without causing premature gelation. This study provides a comprehensive set of optimized parameters for reliably producing high-integrity fused silica green bodies via gel casting, forming a critical foundation for subsequent high-quality sintering.

1. Introduction

Fused silica is a highly valuable advanced ceramic material due to its unique combination of exceptionally low thermal expansion, high thermal shock resistance, and excellent chemical durability [1, 2]. These properties make it indispensable for applications in semiconductor processing, high-precision optics, and aerospace components. However, the fabrication of complex-shaped fused silica parts remains a significant challenge due to the intrinsic brittleness of ceramics and the limitations of conventional shaping methods such as slip casting and injection molding [3].

Gel casting has emerged as a powerful near-net-shaping technique to address these limitations. Originally developed in the 1980s [4], it involves solidifying a concentrated ceramic slurry in situ via the

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polymerization of organic monomers. This process produces green bodies with complex geometries, high green strength, and uniform microstructure [5-7]. Despite its potential, applying gel casting to fused silica requires precise control over numerous parameters, including slurry rheology, gelation kinetics, and drying behavior. The toxicity of early monomers like acrylamide has also been a concern, though it remains a widely studied system for its effectiveness [8-10].

The rheological behavior of the slurry is paramount, dictating its ability to fill complex molds without defects. This is governed by dispersant concentration, solid loading, and the monomer system itself [11-14]. Furthermore, the gelation kinetics, controlled by initiator and catalyst concentrations, must provide a sufficient time window for casting while ensuring rapid and robust setting to immobilize the particles [12, 15]. The mechanical strength of the resulting green body, essential for handling and machining, depends on the integrity of the polymer network formed during gelation. While previous studies have examined various aspects of gel casting, a systematic investigation linking the effects of slurry formulation, gelation parameters, and degassing on the final green properties of fused silica has not yet been established. This study addresses this gap through a comprehensive optimization of the gel casting process for fused silica. We systematically examine the effects of dispersant content, solid loading, monomer/crosslinker ratio, initiator concentration, and degassing time to establish a reliable protocol for producing high-quality fused silica green bodies, thereby enabling their successful densification.

2. Experimental Procedure

The present study systematically investigated the gel casting process for fused silica, focusing on the preparation of stable slurries and the fabrication of high-strength green bodies. The experimental workflow, shown in Fig. 1, was designed to isolate and evaluate the influence of key parameters on slurry rheology and green body properties.

2.1. Raw materials and slurry formulation

Raw materials were selected based on their established roles in the gel casting process, as summarized in Table 1.

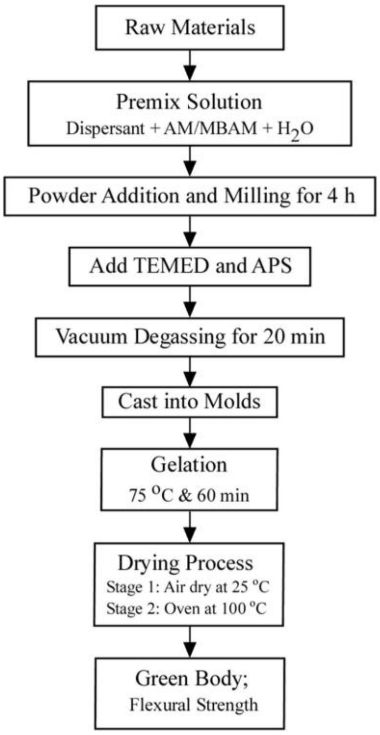

Fig. 1. Experimental workflow chart illustrating the sequential steps of slurry preparation, characterization, gel casting, drying, and green body testing.

Table 1. Raw materials used for gel casting of fused silica

Material	Abbreviation	Role/Function	Purity/Particle size
Fused silica powder	SiO_2	Ceramic phase	>99.5%, $D_{50} = 5$ µm
Acrylamide	AM	Monomer	>99%
N,N′-Methylenebisacrylamide	MBAM	Crosslinker	>99%
Ammonium persulfate	APS	Initiator	>98%
N,N,N′,N′-Tetramethylethylenediamine	TEMED	Catalyst/Accelerator	>99%
Lactic acid (85-90 wt.% aqueous solution, density ~1.2 g/ml)	--	Dispersant	85-90% aqueous solution
Distilled water	--	Solvent	--

A baseline slurry composition with 58 wt.% solid loading was chosen (Table 2), as this value was identified as the practical limit for maintaining a castable viscosity. Operating at this maximum solid loading is crucial for maximizing green density and minimizing drying shrinkage in the final components.

2.2. Slurry preparation protocol

The slurry preparation was a multi-stage process that was critical to achieving homogeneity and stability.

Premix solution preparation: A predetermined amount of distilled water was first poured into a Nalgene jar. The dispersant (lactic acid) was added at a fixed concentration of 0.5 ml (approximately 0.6 g) per 100 g of fused silica powder, unless otherwise stated for specific experiments. The monomer (AM) and crosslinker (MBAM) were then dissolved in the water-dispersant mixture. The total monomer content (AM+MBAM) was fixed at 10 wt.% relative to the fused silica powder mass for the baseline composition, with an AM:MBAM weight ratio of 96.7:3.3. This premix solution was mixed for 15 minutes using a magnetic stirrer to ensure complete dissolution.

Powder incorporation and milling: Fused silica powder was gradually introduced into the premix solution in multiple stages to prevent the formation of irreversible agglomerates and to achieve high solid loading more effectively. The jar was then placed on a roller mill and milled for 4 hours using zirconia balls (5 mm diameter) at a ball-to-powder ratio of 2:1. This step ensured thorough de-agglomeration and coating of the powder particles with the dispersant, resulting in a homogeneous and stable suspension.

2.3. Gel casting and curing process

Following milling, the slurry was prepared for polymerization:

Catalyst and initiator addition: The initiator, ammonium persulfate (APS), was initially dissolved in a minimal amount of distilled water and added to the slurry at a concentration of 0.5 wt.% relative to the total slurry weight, unless varied for gelation studies. Subsequently, the catalyst, TEMED, was added at a fixed ratio of 0.05 × the mass of APS.

Mixing and degassing: The slurry containing the initiator and catalyst was subjected to high-speed mechanical stirring for 2 minutes to ensure uniform distribution. It was then transferred to a vacuum desiccator connected to a diaphragm pump. Degassing was performed at an absolute pressure of 100 mbar for a duration of 20 minutes (established as optimal). Throughout degassing, the slurry container was gently and continuously shaken by hand to facilitate bubble escape and prevent premature surface skin formation. To mitigate the exothermic heat of polymerization, the container was partially immersed in an ice-water bath during this step.

Casting and gelation: The degassed slurry was promptly poured into acrylic molds, which had been pre-coated with a thin layer of silicone release spray. The filled molds were then transferred to a preheated forced-air oven and maintained at 75 ± 2 °C for 60 minutes to complete the in situ polymerization reaction and form the rigid gel network.

Table 2. Baseline composition for 100 g of slurry at 58 wt.% solid loading

Component	Mass (g)	Function
Fused silica (SiO_2)	58.00	Ceramic powder
Acrylamide (AM)	5.60	Monomer (10 wt.% relative to solids)
MBAM	0.19	Crosslinker (3.4 wt.% relative to monomer)
Ammonium persulfate (APS)	0.49	Initiator (0.5 wt.% of slurry weight)
TEMED	0.02	Catalyst (0.05 × APS weight)
Lactic acid	0.35	Dispersant (0.5 ml per 100 g powder)
Distilled water	34.35	Solvent

2.4. Drying and green body formation

The demolded gelled parts underwent a controlled two-stage drying protocol to minimize shrinkage stresses and prevent cracking:

Constant rate period (Air drying): The specimens were first dried at ambient laboratory conditions (25 °C, ~40% relative humidity). Their mass was recorded every 24 hours using a precision analytical balance (ABP-200, Kern, Germany) until approximately 50% of the initial water content had evaporated.

Falling rate period (Oven drying): The samples were then transferred to a drying oven and held at 100 °C for 5 hours to remove the remaining bound water. The final dried green bodies were stored in a desiccator until characterization.

2.5. Characterization methods

2.5.1. Slurry rheology

The viscosity of the slurries was measured using a rotational viscometer (Fungilab, Spain) equipped with an L3 spindle. Measurements were conducted at room temperature. The shear rate was ramped from 10 to 100 rpm to assess shear-thinning behavior. The effects of dispersant concentration (0.2-0.6 ml/100g powder), solid loading (30-60 wt.%), and monomer/crosslinker ratio were systematically studied.

2.5.2. Gelation time measurement

The gelation onset time was determined by monitoring the temperature of the slurry using a K-type thermocouple connected to a data logger. The slurry containing APS and TEMED was placed in a water bath at 75°C. The gelation point was identified as the time corresponding to the sudden, sharp increase in temperature due to the exothermic nature of the polymerization reaction. The initiator (APS) concentration was varied from 0.1 to 1.0 wt.% to study its effect on gelation kinetics.

2.5.3. Green body flexural strength

The mechanical strength of the dried green bodies was evaluated using the ring-on-ring biaxial flexure test on a universal testing machine (STM-150, Santam, Iran), according to ASTM C1499-2003. Disk-shaped specimens (diameter: 30 mm, thickness: 4 mm) were tested with a crosshead speed of 0.5 mm/min. The biaxial flexural strength (σ_f) was calculated using the following equation:

$$\sigma_f = (3P / 4\pi t^2) * [(1-\nu)(D_s - D_L)^2 / 2D^2 + (1+\nu)\ln(D_s / D_L)] \qquad (1)$$

Where P is the fracture load, t is the specimen thickness, ν is the Poisson's ratio, estimated as 0.2 for a ceramic-polymer composite green body [16, 17], D_s is the support ring diameter, D_L is the load ring diameter, and D is the specimen diameter. A minimum of five specimens were tested for each condition, and the average value was reported.

2.5.4. Effect of process parameters on green strength

The influence of key variables on green strength was investigated:

Monomer system: The total monomer content (AM+MBAM) was varied from 5 to 15 wt.% relative to solids, and the AM:MBAM ratio was adjusted.

Initiator concentration: APS concentration was varied from 0.1 to 1.0 wt.% of slurry weight.

Degassing time: The vacuum degassing duration was systematically altered (0, 10, 20, and 30 minutes) while all other parameters were held constant at the baseline values.

3. Results and Discussion

The successful gel casting of ceramics is a delicate balancing act, requiring a slurry that is fluid enough to cast yet capable of forming a rigid, strong polymer network. The results of this study demonstrate a profound and interconnected influence of dispersant, solid loading, monomer chemistry, and processing parameters on both the rheology and the green strength of fused silica.

3.1. Rheological behavior: the foundation of castability

The rheological behavior of fused silica slurries is a key factor in determining the success of the gel casting process. The results demonstrate that the dispersant concentration plays a critical role in controlling slurry viscosity and its overall fluidity. As shown in Fig. 2, increasing the dispersant content initially reduces the viscosity, reaching an optimal at 0.4 ml per 100 g of powder. At sub-optimal concentrations, insufficient coverage of the fused silica particle surfaces fails to overcome van der Waals attractive forces, leading to flocculation and high viscosity. The subsequent increase in viscosity beyond this optimum point is a critical finding, often attributed to the phenomenon of "over-dispersing." In this regime, an excess of ionic dispersant increases the ionic strength of the solution, compressing the electrical double layer and reducing the inter-particle repulsive forces. This can cause particle re-agglomeration through a depletion flocculation mechanism, in which free dispersant molecules generate osmotic pressure that drives particles back into contact [11, 18]. Therefore, identifying this precise optimum is not merely beneficial but essential for achieving a stable, low-viscosity suspension. While the observed viscosity increase at high dispersant concentrations is consistent with the well-documented 'over-dispersion' mechanism involving double-layer compression and depletion flocculation, this interpretation would be further strengthened by direct zeta potential measurements, which are recommended for future work.

The observed shear-thinning behavior (Fig. 3) is highly desirable for a gel-casting slurry. At rest, the weakly flocculated structure or the network of particles trapped in a polymer solution gives the slurry a high viscosity, preventing sedimentation. Upon application of shear during mixing or pouring, these fragile structures are broken down, aligning particles and freeing entrapped solvent, which drastically reduces viscosity and facilitates mold filling. This behavior thus confirms that the slurry possesses the necessary thixotropy for a successful casting process.

The relationship between solid loading and viscosity, shown in Fig. 4, is non-linear, with a critical point identified at approximately 58 wt.%. Up to this point, the increase in viscosity is manageable, as the particles are well-dispersed and can slide past each other. However, beyond this threshold, the particle-to-particle distance decreases significantly, leading to intensified hydrodynamic interactions and frictional forces. The free volume of the solvent becomes insufficient to lubricate particle movement, resulting in a sharp, exponential rise in viscosity. This 58 wt.% represents the practical limit for achieving a castable slurry with this specific powder and dispersant system. While higher solid loadings are theoretically desirable to minimize drying shrinkage, exceeding this limit would render the slurry difficult to process and introduce casting defects.

To move beyond a qualitative description and extract quantitative rheological parameters, the flow curves (shear stress vs. shear rate) were fitted to the Herschel-Bulkley model ($\tau = \tau_0 + K\gamma^n$), which is highly suitable for describing the behavior of concentrated ceramic suspensions [16, 18]. The model parameters—yield stress (τ_0), consistency index (K), and flow index (n)—are summarized in Table 3 for key slurry compositions.

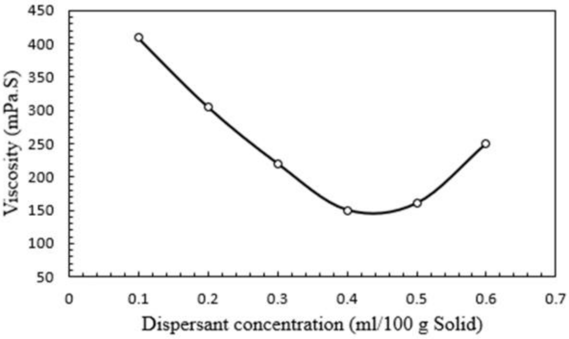

Fig. 2. Effect of dispersant concentration on the viscosity of a slurry with 40 wt.% solids, measured at a rotational speed of 100 rpm.

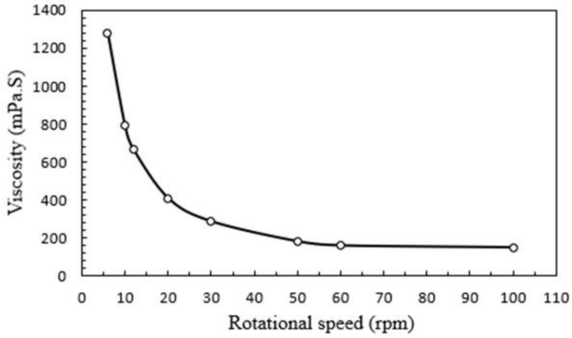

Fig. 3. Viscosity behavior of a slurry containing 40 wt% solids at different rotational speeds.

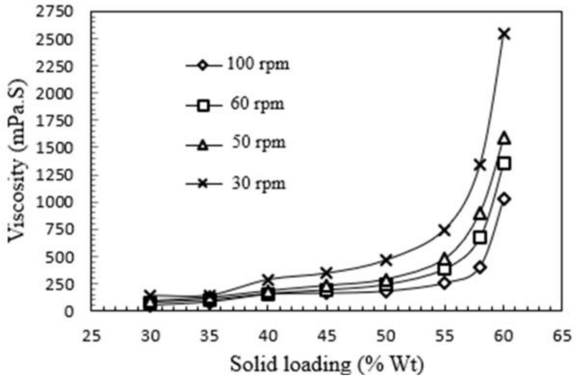

Fig. 4. Effect of solid loading on slurry viscosity at different rotational speeds, using a dispersant concentration of 0.5 ml per 100 g of powder.

Table 3. Herschel-Bulkley rheological parameters for fused silica slurries under different conditions

Slurry condition	Yield stress, τ_0 (Pa)	Consistency index, K (Pa·s^n)	Flow index, n	R^2
Varying Dispersant				
0.3 ml/100g	2.15 ± 0.21	1.88 ± 0.15	0.62 ± 0.03	0.998
0.4 ml/100g (Optimal)	**0.98 ± 0.11**	**0.95 ± 0.08**	**0.72 ± 0.02**	**0.999**
0.5 ml/100g	1.45 ± 0.16	1.21 ± 0.09	0.68 ± 0.02	0.998
Varying solid loading				
52 wt.%	0.51 ± 0.08	0.41 ± 0.05	0.78 ± 0.03	0.997
55 wt.%	0.74 ± 0.09	0.67 ± 0.07	0.75 ± 0.02	0.998
58 wt.% (Optimal)	**1.12 ± 0.13**	**1.05 ± 0.10**	**0.69 ± 0.02**	**0.999**
60 wt.%	3.85 ± 0.35	2.54 ± 0.22	0.58 ± 0.03	0.996

Note: All measurements were conducted at the baseline AM:MBAM ratio of 96.7:3.3, unless otherwise stated.

The yield stress (τ_0) represents the minimum shear stress required to initiate flow, a critical parameter for preventing particle sedimentation after casting and during the initial gelation phase. The data in Table 3 clearly show that the optimal dispersant concentration of 0.4 ml/100g resulted in the lowest yield stress (0.98 Pa) and consistency index (0.95 Pa·s^n), confirming its effectiveness in breaking down agglomerates and producing a well-dispersed, fluid suspension. The subsequent increase in these parameters at 0.5 ml/100g quantitatively supports the over-dispersion phenomenon, where excess dispersant compresses the electrical double layer and promotes flocculation.

The flow index (n) quantifies the degree of shear-thinning behavior. All slurries exhibited $n<1$, confirming shear-thinning (pseudoplastic) character. The value closest to 1 ($n = 0.72$) was also observed at the optimal dispersant concentration, indicating a more Newtonian-like flow under shear, which is highly desirable for mold filling as it minimizes viscosity transients. Furthermore, the effect of solid loading is clearly quantified: increasing from 58 wt.% to 60 wt.% caused a more than threefold increase in yield stress and a significant drop in the flow index ($n = 0.58$), revealing a much more pronounced shear-thinning and solid-like behavior that aligns with the observed sharp rise in viscosity and loss of castability.

A comparison of the optimized parameters with those reported for other ceramic systems underscores the unique requirements of fused silica. The optimal dispersant concentration of 0.4 ml/100 g powder and the maximum practical solid loading of 58 wt.% are notably lower than values typically reported for high-density ceramics like alumina (Al_2O_3) or zirconia (ZrO_2), where solid loadings of 60-65 wt.% are common [11, 19]. This discrepancy can be primarily attributed to the lower particle density of fused silica (~2.2 g/cm³) compared to alumina (~3.9 g/cm³) or zirconia (~5.7 g/cm³). At an equivalent weight fraction, fused silica occupies a significantly larger volume fraction, leading to higher viscosity and a lower critical solids loading for casting. Furthermore, the acidic nature of the lactic acid dispersant used is particularly well- suited to the surface chemistry of silica, whereas other ceramics often require different dispersant types (e.g., polyelectrolytes like ammonium polyacrylate for more basic surfaces) [8]. The initiator concentration of 0.5 wt.% APS aligns well with established practice across systems, indicating that the gelation kinetics are less material-dependent and more a function of the universal free-radical polymerization mechanism. Thus, the primary novelty of this work lies in defining the specific formulation window for fused silica, which is dictated by its distinct physical and chemical properties, enabling its reliable processing via gel casting with unprecedented clarity.

3.2. Monomer system: a delicate balance between flow and strength

The influence of the acrylamide (AM) and MBAM system is twofold, affecting both the slurry's rheology and the final gel's mechanical properties. The increase in viscosity with higher total monomer content (Fig. 5) is expected, as the dissolved polymers increase the continuous phase's viscosity and can cause bridging flocculation between particles. More critically, the ratio of monomer to crosslinker (AM:MBAM) is a decisive factor for the gel's integrity. A high crosslinker (MBAM) content creates a densely cross-linked network with short polymer chains between nodes. While this might seem beneficial for strength, it often leads to a brittle gel. This brittle network is unable to accommodate the significant thermal stresses generated during the highly exothermic polymerization, which resulted in a temperature rise of over 20°C during gelation (see Section 2.5.2), leading to the formation of microcracks. This phenomenon has also been reported in other gel-casting studies using the AM-MBAM system [13]. Conversely, a system with insufficient MBAM produces long, linear polymer chains with few cross-links. This results in a weak, viscous gel that may not adequately immobilize the ceramic particles, leading to low green strength and potential particle settling. The optimal AM:MBAM ratio, therefore, creates a flexible yet strong three-dimensional network with long polymer segments between cross-linking points, capable of dissipating stress and forming a robust green body.

3.3. Gelation kinetics: controlling the polymerization window

The gelation onset time, controlled by the initiator (APS) concentration (Fig. 6), is a critical processing parameter. The inverse relationship is logical: a higher initiator concentration generates more free radicals, accelerating the propagation step of the polymerization. While a very short gelation time might be advantageous for rapid production, it significantly curtails the available window for de-airing, mixing, and complex mold filling, which can lead to defects. An initiator concentration that is too low, however, risks incomplete polymerization or premature sedimentation of the powder before the gel sets. The chosen optimum of 0.5 wt.% APS provides a reliable and reproducible gelation time that is practical for laboratory-scale processing, ensuring the slurry remains fluid long enough for thorough degassing and casting before transitioning to into a rigid solid.

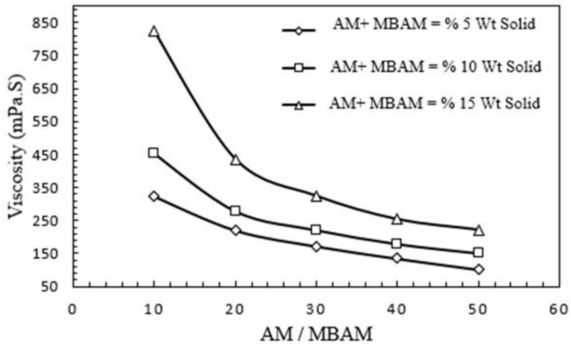

Fig. 5. Effect of the AM:MBAM monomer-to-crosslinker ratio on the viscosity of a slurry with 58 wt.% solid loading, measured at different total monomer (AM+MBAM) concentrations.

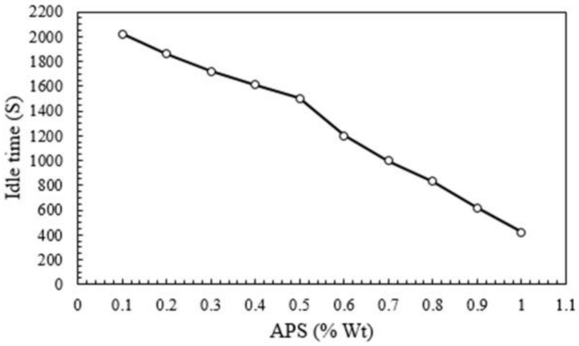

Fig. 6. Effect of initiator (APS) concentration on the gelation onset time.

3.4. Properties of green bodies

The mechanical strength of the green bodies depends on the polymer network formed during gelation. Increasing monomer concentration generally leads to an increase in green strength, as faster polymerization leads to earlier chain growth and more effective cross-linking (Fig. 7). However, excessive monomer relative to cross-linker caused rapid setting, generating microcracks and reducing the resulting strength. This highlights the critical role of MBAM in controlling polymer network density and mitigating thermal stresses during gelation.

Initiator concentration also had a non-linear effect on green strength (Fig. 8). At low initiator levels, insufficient polymerization resulted in weak gels. At high levels, localized polymerization led to heterogeneous networks and reduced mechanical integrity. An optimum initiator concentration is therefore required to maximize green body strength [20].

Degassing time was another key factor. Increasing degassing time up to 20 minutes improved strength by removing trapped air and reducing porosity. However, longer degassing times caused premature gelation within the slurry (Fig. 9), which reduced mold filling time and weakening the green bodies. The optimal degassing duration depends on solid loading and slurry viscosity, but excessive times are detrimental.

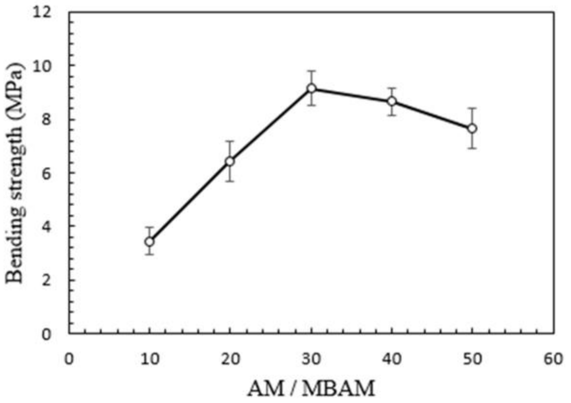

Fig. 7. Effect of the AM:MBAM monomer-to-crosslinker ratio on the flexural strength of green bodies. Specimens were fabricated from a slurry with 58 wt.% solid loading, a total monomer content (AM+MBAM) of 10 wt.% relative to solids, and a constant initiator concentration.

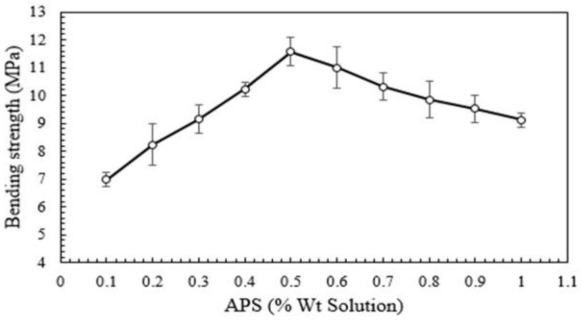

Fig. 8. Effect of initiator (APS) concentration on the flexural strength of green bodies. Specimens were fabricated from a slurry with 58 wt.% solid loading and a total monomer content (AM+MBAM) of 10 wt.% relative to solids.

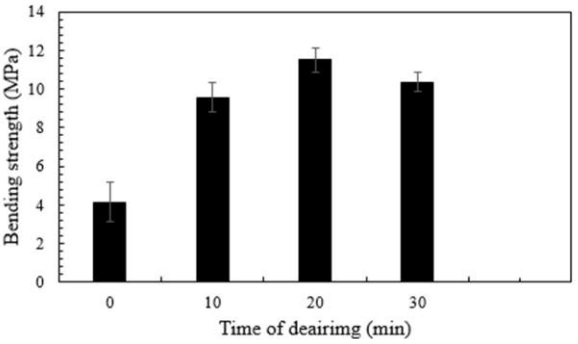

Fig. 9. Influence of de-airing time on the flexural strength of green specimens.

4. Conclusions

This study systematically optimized the gel casting process for fused silica, establishing the critical interplay between slurry formulation, gelation parameters, and degassing on the rheological behavior and mechanical properties of green bodies. The results revealed that:

- A dispersant concentration of 0.4 ml per 100 g of powder effectively minimizes viscosity while preventing flocculation.
- The slurry achieves optimal stability at 58 wt.% solid loading, beyond which viscosity increases sharply and castability is significantly compromised.
- The AM:MBAM ratio plays a pivotal role in balancing fluidity and structural integrity, where excessive crosslinking induces brittleness and microcracking, while insufficient crosslinking weakens the polymer network.
- An initiator concentration of 0.5 wt.% provides a practical gelation window, ensuring complete polymerization without premature setting.
- A degassing time of 20 minutes effectively eliminates porosity and enhances strength, while longer durations increase the risk of premature gelation.

Under these optimized conditions, the resulting green bodies exhibited high flexural strength and structural uniformity. This work establishes a robust, comprehensive protocol for slurry preparation, gel casting, and green body strengthening, providing a reliable foundation for the near-net-shape fabrication of high-integrity fused silica components. Such components are critical for demanding applications including radomes, precision optical substrates, and refractory molds for investment casting.

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Authors’ contributions

M. Afgarazordeh: Methodology, Data curation, Formal analysis, Visualization, Investigation

M. H. Paydar: Conceptualization, Supervision, Validation, Writing original draft, Writing-review and editing

Conflict of interest

The authors declare no conflict of interest.

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