



Role of Mixing Method and Solid Content on Printability of Alumina Inks for Stereolithography 3D Printing Process

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ABSTRACT

Additive manufacturing of ceramics via stereolithography method is a promising way to fabricate high-resolution ceramic parts with complex geometry, which is hard to obtain with traditional ceramic shaping methods. In order to shape the ceramics with the Digital Light Processing (DLP), a mixture of photocurable resin and ceramic powders, called ink, must be prepared. In this paper, the printability of the Alumina-glass inks, with different solid contents were prepared by two mixing methods, having long and short mixing durations. In order to evaluate the printability of inks, the rheological behavior of suspensions was investigated, and printing parameters such as curing time and layer thickness were changed. The ceramic-resin suspensions were prepared via 24-hour ball-milling and 10,000 rpm mechanical homogenizing. The suspension containing 60 wt% solid content and prepared by mechanical homogenizing showed the best stability with 8% sedimentation within 4 days and the lowest viscosity of 1.37 Pa·s at shear rates of 30 s⁻¹, exhibiting a suitable viscosity for DLP printing. Therefore, a mechanical homogenizer can be a promising and quick method for mixing by providing simultaneously appropriate rheology and printability.

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1. INTRODUCTION

Conventional shaping methods of advanced ceramics, especially for fabricating complex shaped components, have always been challenging. The development of Additive Manufacturing as a layered fabricating technique has overcome some of the difficulties and now is recommending an alternative way of shaping ceramics by providing both unique structures and acceptable dimensional accuracy [1–3].

In Ceramic Stereolithography, which is a photopolymerization-based technique, ceramic particles are dispersed in the photosensitive resin, and the ceramic part is manufactured layer by layer by the exposure of UV light (either laser or projector) [4–6].

In preparing a printable ink for the stereolithography process, loading a photocurable resin with high ceramic

solid content is essential to lower the shrinkage in sintering and provide a high density with desirable mechanical properties. Meanwhile, increasing the solid content in resin makes the suspension thicker by losing its auto-leveling property and increasing the light scattering effect due to refractive index contrast between the resin and the ceramic powder. As a result, the printing procedure becomes more difficult. This property is one of the pivotal points required in Stereolithography, especially in bottom-up Digital Light Processing (DLP) to spread thin layers. Therefore, preparing a highly ceramic-loaded resin with eligible rheological properties is essential [5, 7–11].

Besides the auto-leveling property, it is necessary to create sufficient adhesion between the newly cured layer and the build plate to dominate the vacuum force and prevent the sample from being removed in bottom-up

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DLP. Instead, there is no vacuum force in the top-down DLP approach, which makes the printing process smoother; nevertheless, the bottom-up approach is more common due to its higher resolution, faster printing process, and less material waste [12]. Also, the mentioned problems can be minimized by careful selection of the resin components and dispersants or by changing the particle size distribution of the ceramic powder and the suitable mixing method to control the light scattering effect [13, 14]. Besides selecting the proper mixing method, the preparation time can also be an essential factor to distinguish the 3D printing technique from the conventional shaping methods.

For manufacturing dense ceramic parts, printing ceramic-resin inks with a high amount of ceramic powder might develop a better sintering quality. The desirable amount of ceramic powder in resin should be at least 40 vol% to obtain a fully dense ceramic part after debinding and sintering [10, 15]. However, the viscosity is highly affected by the ceramic particles. Resins with higher solid content would increase the viscosity and deteriorate the self-leveling property [16]. As reported, for ceramic-resin photocurable suspensions, shear-thinning behavior and viscosity of 3-5 Pa·s at 30 s⁻¹ shear rate are preferable [14, 15].

This study presents the Alumina-resin inks' preparation for bottom-up DLP printing by employing two mixing methods having a significant difference in preparation time. The effect of mixing methods and solid contents on rheological properties, stability to sedimentation and accordingly printability was investigated.

2. MATERIALS AND METHODS

2. 1. Raw Materials and Ink Preparation

Ceramic-loaded photocurable resins were prepared using α -Alumina powder (d90=5 μ m and 99.99% purity), soda-lime glass frit, etc., methacrylate-based photosensitive resin, containing certain amounts of defoamer, dispersant, and photoinitiator (Maan Polymer). Firstly, the Alumina powder and the glass frit were fast milled for 10 minutes at a weight ratio of 7 to 3 and sieved through a 270 mesh screen. Then, according to Table 1, the powder mixture was added to the resin monomer in a stepwise manner and two different mixing methods were applied for the suspensions; i.e. high-speed mechanical homogenizer (total time of 10 minutes in 10,000 rpm speed) and ball-milling (24 hours using Alumina balls).

2. 2. DLP 3D Printing In stereolithography, there is a vat containing photocurable resin which is exposed to UV light. Then it is polymerized layer by layer according to a computer-aided design (CAD) file, which is sliced into 2D layers. The 3D part is fabricated on a build plate

TABLE 1. Alumina-resin inks characteristics

Label	Powder		Mixing method
	wt%	vol%	
B50	50	23	Ball-mill
H50	50	23	Mechanical homogenizer
B60	60	31	Ball-mill
H60	60	31	Mechanical homogenizer

moving along the Z-axis and solidifying the photocurable liquid in each layer. There are two types of light sources used in stereolithography, i.e., laser beam and projection. In projection-based stereolithography or Digital Light Processing (DLP), the layer is polymerized by the exposure of the UV projection light at once. The light source can be positioned either at the top or bottom of the vat [17]. In this work, the samples were printed by a bottom-up approach. The stereolithography process was performed via Parsa 3D bottom-up DLP Printer, using Vivitek 4000 Lumen projector. The models were printed according to the optimized parameters of layer thickness, curing time, and amount of the photoinitiator, as shown in Table 2. The schematic of DLP printing is shown in Figure 1.

2. 3. Sintering Sintering of the samples was carried out at a temperature of 1400 °C. First, for resin burn-out, samples were heated at rate of 5 °C/min up to 400 °C maintained for 1 hour, and then heated up to sintering temperature at rate of 10 °C/min, with 1.5 hours soaking time.

TABLE 2. DLP printing parameters

Sample	Curing time	Layer thickness	Photoinitiator
H50	2.3 s	30 μ m	3.33%
B50			
H60	2.3 s	30 μ m	4%
B60			

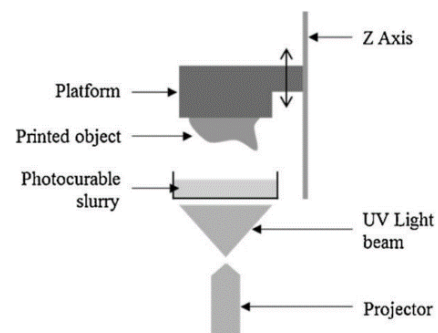


Figure 1. Schematic of bottom-up DLP printing [18]

2. 4. Characterization The rheological properties were tested at room temperature using a rotational rheometer (MCR301, Anton Parr, Austria), varying the shear rate from 0.1 to 1000 s^{-1} . For measuring the stability of the suspensions at room temperature, the prepared inks were kept 4 days in the dark place, considering the total initial suspension height as H and the settled suspension as H_0 .

3. RESULTS AND DISCUSSION

3. 1. Ink Properties The rheological behavior and stability of the printable inks with 50 and 60 wt% solid contents are shown in Figure 2. The viscosity of resins increased by introducing ceramic powder, from 0.15 Pa·s in pure resin to of 0.57 and 1.75 Pa·s in the shear rate of 30 s^{-1} for 50% and 60 wt%, respectively. All samples' viscosity was within the accepted range for pouring in a 3D printer tank [19].

The Alumina-glass inks' viscosity is reduced with the shear rate increase, exhibiting a non-Newtonian, shear-thinning behavior, a desirable rheological property for the stereolithography process [14, 20, 21].

In stereolithography 3D printing, the resin is cleaned up from the VAT surface by a moving paddle between the layer printing intervals. The resin's shear-thinning behavior is directly related to self-leveling properties, where the resin must rapidly fill the empty spaces after paddle sweep [22, 23].

As shown in Figure 2, all the samples had a high viscosity in low shear rates, and the samples with more ceramic powders (B60 and H60), showing higher viscosity. The viscosity of the samples decreases by increasing the shear rate and reaching a plateau, significantly for the lower powder samples (B50 and H50), as previously seen in other reports. The more stable viscosity at a higher shear rate, the better is self-leveling properties and, consequently, the better printing process. It can be concluded that high solid content resins show less stable viscosity at high shear rates [24].

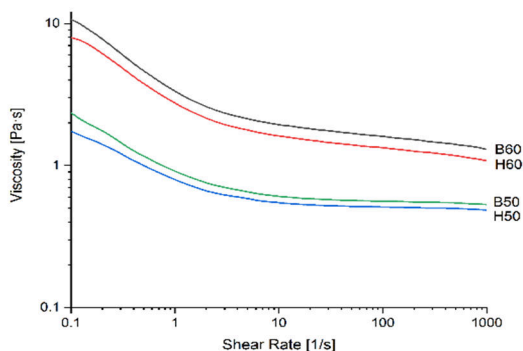


Figure 2. Viscosity variations of the suspensions

Despite the two different preparing methods, it is observed that the inks indicate relatively similar rheological behaviors and viscosity values of the same orders. In other words, mixing processes have not significantly affected the rheological behavior. The ball-milled samples have relatively higher viscosities (in all shear rates) compared to mechanically homogenized samples. In general, DLP ceramic slurries' viscosity needs to be less than 20 Pa·s within the shear rate of 10-100 s^{-1} [25]. B60 ink, as the most viscous ink, exhibits a viscosity of about 1.75 Pa·s at 30 s^{-1} shear rate, which, as reported, is still suitable for the stereolithography process [14, 15, 24].

The lower viscosity in mechanical homogenized inks and increase in flowability can be attributed to the agglomerates' break up and more dispersion of the particles in the resin [15]. As a result, H60 ink has a viscosity of 1.37 Pa·s at shear rates 30 s^{-1} which is lower than B60 ink. On the other hand, although ball-milling is well-known as an effective method in preparing ceramic slurries, there is a time limitation to use it for slurry preparation. Even though ball-milling duration is not investigated as a parameter in this research, it was believed that long-time milling and overmixing of ceramic loaded resins could interrupt dispersion by damaging the structure of resin and dispersants. In other words, overmixing has increased the viscosity by reducing particle size. Particle size plays a vital role in viscosity. When the particles become finer, and the surface area is increased, more dispersant may be needed to cover all the particles' surface. So, the viscosity of the suspension will increase, as reported previously [26, 27]. Moreover, it can be said that damaging the dispersant within the resin and losing its functionality would be another possible reason for viscosity-increasing [28].

The stability and viscosity have the same critical role in the performance of ceramics slurries for the DLP process. As it is mentioned previously, low viscosity slurries were obtained by using lower solid load and larger particle sizes [22]. However, this results in sedimentation enhancement. Sedimentation of slurries during the printing process leads to discontinuity and inhomogeneity [5, 28, 29]. The sedimentation rate test is normally used to evaluate the stability of suspensions. Figure 3 shows the sedimentation of the suspensions within 3 days. As shown in Figure 3 and discussed earlier, the suspensions containing 60 wt% solid (B60 and H60), were significantly more stable than the suspensions with lower solid content. Also, it was observed that the sedimentation rate of the inks having 50 wt% solid had increased considerably after 36 hours. Generally, H60 is the most stable suspension with 8% sedimentation over 4 days. Therefore, as expected, in a 2-hour printing duration or more, the ink stayed homogeneous, and settlement was negligible.

The mixing method effect on slurries' stability is quite different in low and high ceramic loading samples. As shown in Figure 3, in lower solid content samples, the ball-milled sample (B50) is more stable than H50. As discussed, ball-milling has resulted in finer particles and relatively higher viscosity, making the settlement rate of the particles lower, according to Stock's law [26, 27]. Therefore, the B50 sample has settled slower than the H50 sample. Whereas in the samples with higher solid inks, the effect of the mixing method was less significant. By increasing the mixing or sedimentation test time, this difference would be more effective, which was not investigated in this study. Therefore, by comparing the two mixing methods and considering that for ceramic manufacturing, high-solid content suspensions are required. It seems that high-speed mechanical homogenizing is a better mixing option.

3. 2. 3D Printing In the bottom-up method, the cured layer must be detached from the VAT in each step of layer printing, following by recoating fresh ceramic slurry to be prepared for the next layer printing [12]. However, in printing the suspensions, the main issue was to make the first layer stick to the build plate. Besides the detachment force that is induced for each cured layer, the layer adhesion can be enhanced by adjusting the curing time, layer thickness, and the amount of the photoinitiator [30]. These parameters influence the curing depth, a crucial parameter determining the accuracy of the printing [31].

In Figure 4, an example of a sample printed from a suspension containing 20 wt% solid and prepared by mechanical homogenizing is shown. It can be seen that

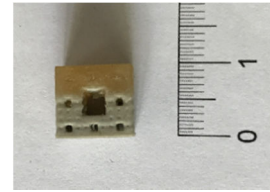


Figure 4. Two-phased printed sample from suspension containing 20 wt% solid content

the suspension instability has resulted in phase separation in suspension and a two-phased sample. The ceramic particles have settled too quickly before the sample is printed entirely, which highlights the significant effect of the solid loading on the suspension stability.

H50 was successfully printed by adjusting the printing condition as curing time of 2.3 s, layer thickness of 30 μm , and 3.33% of photoinitiator. Eventually, as the viscosity of H50 and B50 was quite the same, both were printable with a similar condition. But the switching in mixing method of 50 wt% inks from mechanical homogenizing to ball-milling resulted in drastic changes in the quality and accuracy of printing, as is shown in Figure 5. Small holes of the printing model did not form, the size of larger holes decreased, and the sample's edges were extended. This could be attributed to light scattering, which is intensified as a result of ball-milling, delivering more radiation to the sideways. Previously, the sedimentation and viscosity results showed that ball-milling was more effective in deagglomeration and production of finer particles in ceramic slurry.

By increasing the ceramic content to 60 wt%, as expected, the curing energy was not enough for the previous set of 50 wt% inks. The low adhesion was assumed to be a result of the high curing time, causing the first layer to stick to the silicon vat instead of the build plate. However, decreasing the curing time and then the layer thickness to 15 μm showed that the penetration depth was not high enough to cure a single layer completely. In other words, in curing each layer, the resin monomers did not get the necessary activation energy for polymerization due to the high light scattering effect, which caused the resin to be more stable to light exposure [8, 32]. Although a sample containing ~60 wt% ceramic powder was printable with 15 μm layer thickness, however; considering the long printing duration, this condition was not reasonably practical.

Therefore, for modifying the reactivity and reducing the activation energy needed for polymerization in 60 wt% suspensions, the photoinitiator amount in resin was doubled, reaching the ratio of 4 to 100 per weight [33]. Adding more photoinitiator helped increase the layer thickness up to 30 μm , without any sensible changes in the viscosity. According to Hinczewski et al. [9], the photoinitiator does not influence the viscosity of the resin monomer. On the contrary, increasing the photoinitiator

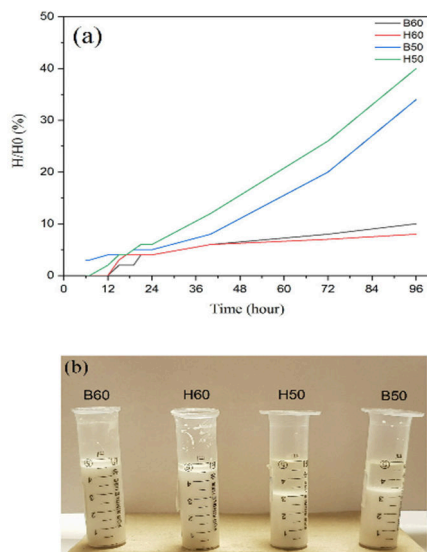


Figure 3. Stability of Alumina-glass suspensions prepared by two different mixing methods: a) Stability test after 4 days b) Sedimentation over time (showing H/H₀ value vs time)

amount led to new problems related to the light stability of the resin. The resin got highly sensitive to the environmental light during the preparation and printing so that the prepared ink was used to polymerize quickly overnight, even though it was kept in the dark place.

As shown in Figure 5, suspension propagated around the printed samples (both 50 and 60 wt%), and the printing accuracy has declined drastically. Propagation might happen due to high light scattering, which causes the reduction of the cure depth. In other words, ball-milling has intensified the scattering, delivering more radiation to the sideways [17, 31]. Probably, ball-milling produced finer particles or broke up agglomerates, resulting in more light scattering. Additionally, long ball-milling time damaged the dispersant in the resin, affecting the inter-particle spaces. Both finer particles and damaged dispersants declined the photopolymerization process [28, 33].

Eventually, it was found that for the bottom-up approach, H60 suspension is the most convenient ink to print Alumina-glass ceramic parts. By considering the properties resulted for ball-milling, it is prepared quickly, the viscosity is low enough, and no sedimentation occurs during the printing process. Moreover, using Maan polymer resin, it was not possible to increase the solid ceramic content by more than 60 wt% due to high light scattering and difficulties in first layer adhesion to the build plate.

In order to consolidate printed parts, sintering was carried out in 1400 °C. By increasing the solid content, the shrinkage and relative density of samples changed. Typically, Alumina ceramics is reaching maximum density in the range of 1500 to 1700 °C [34, 35], but by adding the glass as a sintering aid, the full dense alumina-glass bodies fabricated at 1400 °C.

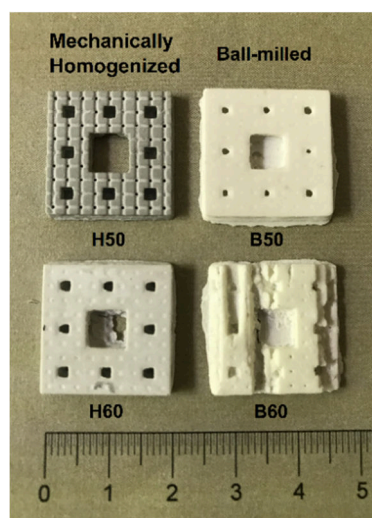


Figure 5. Printed samples via bottom-up approach; samples printed from suspensions H50, B50, H60 and B60

4. CONCLUSIONS

In this work, employing two different mixing methods for the DLP 3D printing showed that the effectiveness of mixing techniques mainly varies when the amount of the solid loading is changed. Ball-milling created higher viscosities and stability for the suspensions containing 50 wt% solid content. In comparison, high-speed mechanical homogenizing was found to be more suitable for preparing the suspensions with 60 wt% solid content. However, the suspensions have shown shear-thinning behavior and viscosities in the accepted range for DLP printing for both mixing methods.

5. ACKNOWLEDGMENTS

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Persian Abstract

چکیده

ساخت افزایشی سرامیک‌ها با استفاده از استریولیتوگرافی را می‌توان به عنوان روشی مطمئن برای ساخت قطعات با شکل‌های پیچیده در نظر گرفت، چرا که اغلب ساخت برخی از قطعات خاص با روش‌های شکل‌دهی متداول سرامیک‌ها مشکل است. در روش استریولیتوگرافی، قطعات سرامیکی با استفاده از یک سوسپانسیون رزین حساس به نور و ذرات سرامیکی چاپ می‌شوند. در این پژوهش، قابلیت چاپ جوهرهای آلومینا-شیشه در درصدهای مختلف ماده جامد و با استفاده از دو روش اختلاط مورد بررسی قرار گرفته است. جهت بررسی قابلیت چاپ، خواص رئولوژیکی سوسپانسیون‌ها و همچنین شرایط چاپ مانند زمان تابش نور و ضخامت لایه به عنوان متغیر در نظر گرفته شدند. سوسپانسیون‌های رزین-آلومینا، توسط بالمیل به مدت ۲۴ ساعت و هموژنایزر مکانیکی با سرعت ۱۰۰۰۰ دور بر دقیقه تهیه شدند. سوسپانسیون حاوی ۶۰ درصد وزنی پودر و آماده شده توسط هموژنایزر مکانیکی، بهترین پایداری با حدود ۸ درصد ته‌نشینی در طی ۴ روز از خود نشان داد. بعلاوه، گرانروی سوسپانسیون‌های تهیه شده با هموژنایزر مکانیکی در مقایسه با بالمیل، کمترین و با مقدار $1/37 \text{ Pa}\cdot\text{s}$ در نرخ برشی 30 s^{-1} اندازه‌گیری شدند که مطابق تحقیقات انجام شده برای روش استریولیتوگرافی گرانروی مناسبی تلقی می‌شود. در نتیجه، هموژنایزر مکانیکی، به عنوان یک روش اختلاط سریع برای تهیه سوسپانسیون‌های فرایند چاپ سه‌بعدی استریولیتوگرافی می‌تواند به کار رود.
