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Stereolithography: A new method for processing dental ceramics by additive computer-aided manufacturing

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A B S T R A C T

Objectives. The aim of this study was to compare the physical and mechanical properties of stereolithography (SLA)- manufactured alumina ceramics of different composition to those of subtractive- manufactured ceramics and to produce suitable dental crown frameworks. **Methods.** The physical and mechanical properties of a control and six experimental SLA ceramics prepared from slurries with small (S) and large (L) particles (0.46 ± 0.03 and $1.56 \pm 0.04 \mu\text{m}$, respectively) and three dry matter contents (70%, 75%, 80%) were evaluated by dynamic rheometry, hydrostatic weighing, three-point flexural strength measurements, and Weibull analyses, and by the micrometrics measurement of shrinkage ratio before and after the heat treatments.

Results. S75 was the only small particle slurry with a significantly higher viscosity than L70. The viscosity of the S80 slurry made it impossible to take rheological measurements. The viscosities of the S75 and S80 slurries caused deformations in the printed layers during SLA manufacturing and were excluded from further consideration. SLA samples with low dry matter content had significantly lower and densityflexural strengths. Only SLA samples with a large particle size and high dry matter content (L75 and L80) were similar in density and flexural strength to the subtractive- manufactured samples. The 95% confidence intervals of the Weibull modulus of the L80 ceramic were higher (no overlap fraction) than those of the L75 ceramic and were similar to the control (overlap fraction). The Weibull characteristics of L80 ceramic were higher than those of L75 ceramic and the control. SLA can be used to process suitable crown frameworks but shows results in anisotropic shrinkage. **Significance.** The high particle size and dry matter content of the L80 slurry allowed made it possible to produce a reliable ceramic by SLA manufacturing with an anisotropic shrinkage, and a density, and flexural strength similar to those of a subtractive-manufactured ceramic. SLA allowed made it possible to build up a dense 3D alumina crown framework

Keywords:

Dental crown
Stereolithography
Ceramics
CAD/CAM
Additive technology

1. Introduction

Subtractive computer-aided design (CAD) and computer-aided manufacturing (CAM) technologies have led to major improvements in dentistry [1]. They make it possible to produce reliable restorations with accurate dimensions [2] and to reduce manufacturing time [1]. However, these processes are limited by the waste of raw material (loss of unused portions of blocks and difficulty in recycling excess material) and heavy wear of milling tools. Microscopic cracks, which can weaken restorations, can also appear in objects due to the milling process [3].

Additive manufacturing processes avoid these limitations by building objects layer-by-layer. While such processes are already being used to manufacture metal and polymer prototypes [1], the shaping of ceramics for dental applications is still in its infancy [4,5]. Ceramics can be additively manufactured by polymerizing an inorganic binder in a ceramic powder bed (3D printing) [6], selective laser sintering (SLS) [7], ceramic slurry printing (direct inkjet printing) [8], or stereolithography (SLA) [9]. SLA produces scaffolds in calcium phosphate hydroxyapatite with the higher accuracy, surface quality, and mechanical properties [9] than the other processes by selectively curing a photosensitive ceramic slurry using an ultraviolet beam. Curing with ultraviolet light makes it possible to work under artificial light without causing polymerization during the slurry preparation process.

Alumina and zirconia ceramics, the gold standard for crown frameworks in polycrystalline ceramic, possess good mechanical resistance [10] and are thus the focus of SLA additive manufacturing research and development [9]. Even though additive-manufactured zirconia has the highest flexural strength [11], its high refractive index [12] limits curing depth compared to alumina. This parameter is of utmost importance for SLA manufacturing [9], making alumina a good material for optimizing additive processes for preliminary dental research.

Since the viscosity of a slurry influences the reliability of complex structures, this parameter must be adapted to the SLA-manufacturing process. The incorporation of ceramic particles in curable resins modifies their rheological behavior. Increases in viscosity depend on the nature, dry matter content, and particle size of the ceramic [13,14]. The dry matter content of slurries has to exceed 50% (v/v) to avoid deformation and crack formation during thermal treatments. However, the higher the dry matter content the higher the viscosity [9]. As such, a compromise between a high dry matter content and low viscosity that is compatible with SLA manufacturing must be found.

The photosensitive cured polymer used to assemble ceramic particles during SLA manufacturing is burnt out during debinding. This increases the porosity and reduces the mechanical strength of sintered objects [5], preventing their

use as crown frameworks, which require the highest density possible.

In addition, crown frameworks require high flexural strength to ensure the durability of restored teeth that are subjected to masticatory strains [15]. The 3-point flexural strength of these materials should be at least 300 MPa (ISO 6872:2008) if they are to be employed for clinical uses. Ceramics used for dental restorations must also be very reliable. Structural reliability is generally based on two Weibull distribution parameters: the Weibull modulus and the Weibull characteristic strength [15,16]. The Weibull modulus allows lower range strength values whose distribution is typical of ceramic materials (do not follow Gaussian distribution) to be used [17]. A high Weibull modulus indicates a high degree of homogeneity and smaller variations in flexural strength. Both parameters have a major impact on the clinical success of ceramic restorations [15].

Most reports in the literature dealing with ceramic SLA manufacturing focus on the impact of slurry composition on polymerization [9,13] and the optimization of photo-initiator, diluent, and polymerizable monomer concentrations in ceramic slurries. However, no data is available with regard to the impact of variations in particle size and dry matter content on the flexural strength and Weibull characteristics of SLA-manufactured dense ceramics. While SLA-manufactured temporary crowns are commercially available [1], there are no reports in the literature on the feasibility of using SLA-manufactured crown frameworks composed of dense ceramic.

The aim of the present study was thus to compare the physical and mechanical properties of SLA-manufactured alumina ceramics of different composition (dry matter content, particle size) and viscosity to those of subtractive-manufactured ceramics, and to demonstrate the feasibility of producing suitable dental crown frameworks by the SLA process.

The four null hypotheses tested were: (1) variations in the composition of the ceramic slurry used in the SLA-manufacturing process do not influence viscosity; (2) there is no difference in density between SLA-manufactured and subtractive-manufactured alumina ceramics; (3) there is no difference in flexural strength between SLA-manufactured and subtractive-manufactured alumina ceramics with high Weibull characteristics; (4) there is no difference in shrinkage between SLA-manufactured and subtractive-manufactured alumina ceramics; and (5) it is possible to produce dental crown frameworks by SLA manufacturing.

2. Materials and methods

2.1. Specimen preparation

2.1.1. Experimental groups

Slurries were prepared using small (S) (CT3000SG; Almatis, PA, USA) and large (L) (CT1200SG; Almatis, PA, USA) particle size

Table 1 - Materials used in the different experimental groups with their particle sizes and dry matter content.

Group	CAD type	Dry phase						Photosensitive resin					
		Composition	Material	Manufacturer	Batch no.	D50 particle size (μm)	Mean (sd)	Material	Manufacturer	Batch no.	Dry matter content (% weight)		
1.80	SLA	Al ₂ O ₃	CT1200SG	Almatis	1446533255A	1.58 (0.03)	C1-Alumina	CryoBeryl Software	01	80			
1.75	SLA	Al ₂ O ₃	CT1200SG	Almatis	1446533255A	1.58 (0.03)	C1-Alumina	CryoBeryl Software	01	75			
1.70	SLA	Al ₂ O ₃	CT1200SG	Almatis	1446533255A	1.58 (0.03)	C1-Alumina	CryoBeryl Software	01	70			
S80	SLA	Al ₂ O ₃	CT3000SG	Almatis	200412442	0.46 (0.04)	C1-Alumina	CryoBeryl Software	01	80			
S75	SLA	Al ₂ O ₃	CT3000SG	Almatis	200412442	0.46 (0.04)	C1-Alumina	CryoBeryl Software	01	75			
S70	SLA	Al ₂ O ₃	CT3000SG	Almatis	200412442	0.46 (0.04)	C1-Alumina	CryoBeryl Software	01	70			
Cont	Sub.	Al ₂ O ₃	In-Ceram AL	Vita Zahnfabrik	32510								

(Table 1) alumina powders. The powders were dried for 1 h in an oven (Beschickung loading model 100-800; Memmert, Germany) at 90 °C and were stored in hermetically sealed containers (TROP-500-010; Labbox, Spain) at room temperature. Particle sizes were measured ($n=3/\text{group}$) by laser scattering (Malvern 2000; Mastersizer, UK) (Table 1). Six experimental groups were formed based on particle size and dry matter content (S80, S75, S70, L80, L75, L70). A photosensitive ($\lambda = 350\text{--}400 \text{ nm}$) acrylic resin (C1-alumina; CryoBeryl Software, France) was placed in jars (01.462.0220; Retsch, Germany) using graduated pipettes (pip-0806-250; Labbox, Spain) for the SLA manufacturing of the slurries. The powders were weighed (PS8000/X; Radwag, Poland) (linearity $\pm 0.03 \text{ g}$) and were incorporated into the liquid. The slurries were poured into agate jars containing zirconia milling balls and were homogenized at 150 rpm by planetary milling for 30 min (PM100; Retsch, Germany). The absence of media contamination during the milling process was confirmed by inductively coupled plasma-optical emission spectrometry (720-ES, Varian Inc., PA, USA).

The parallelepipedic samples ($1.3 \times 4 \times 22 \text{ mm}$, oversized by the shrinkage ratios given in Table 3, ISO 6872:2008) with different densities ($n=10/\text{group}$) and flexural strengths ($n=15/\text{group}$) were designed using CAD software (Catia; Dassault Systèmes, France) (Fig. 1). The 3D objects were supported by pillars (end diameter: 0.2 mm, base diameter: 0.7 mm, height: 2 mm) during the design phase and were sliced (width = Z-direction, layer thickness = 50 μm) using Creation Workshop software (Datatree3D, TX, USA). The data were transferred to SLA software (CryoCeram; CryoBeryl Software, France) and equipment (CryoCeram Printer; CryoBeryl Software, France) for additive manufacturing (curing energy = 10 mW cm⁻²; resolution = 50 μm). The SLA process used integral SLA methods that make it possible to polymerize the entire surface of a layer at once using a dynamic mask of digital micro-mirror devices. Pillars were hand-sectioned following the SLA-manufacturing phase, and the microstructures of the samples were assessed by scanning electron microscopy (S-3500N; Hitachi, Japan) (magnification $\times 90$, secondary electron imaging). The samples were thermally debound (0.1 °C/min, 450 °C, 2-h dwell time) in an oven (LHT; Carbolite, UK) before sintering (15 °C/min, 1690 °C, 3-h dwell time) at atmospheric pressure in a furnace (HT16/17; Nabertherm, Germany).

2.1.2. Control group

Samples prepared using the subtractive method were used as controls (Cont). In-Ceram Al blocks (Vita Zahnfabrik, Germany) were serially cut into sticks using a Setocom-15 cutting machine (Struers Ltd., UK) equipped with a 0.8-mm-thick abrasive cut-off wheel. The samples were sintered using the manufacturer's standard procedure (17 °C/min, 1530 °C, 2-h dwell time) in a Zycromat sintering furnace (Vita Zahnfabrik, Germany).

After sintering, the sample sizes manufactured by the SLA and subtractive methods were determined using an analog micrometer (Mitutoyo; Japan) (precision $\pm 0.01 \text{ mm}$). Based on the ISO 6872:2008 standard, the standard deviations of specimen dimensions should not exceed 0.20 mm in thickness and width, with minimal values of 1.20 and 4.00 mm, respectively. In the present study, the sample dimensions were

Table 2 – Physical and mechanical properties of dental alumina ceramics manufactured using the stereolithography (L80, L75, L70, S70) and subtractive methods (Cont): viscosity, density, 3-point flexural strength (σ), and Weibull characteristics (σ_0 and m).

Sample name	Viscosity at 2 s ⁻¹ retention in mPa s Mean (sd)	Density in % Mean (sd)	σ in MPa Mean (sd)	σ_0 in MPa[CI range 95%]	m [CI range 95%]
L80	113 (5.54) ^{ab}	98.1 (0.4) ^{cd}	367.9 (52.4) ^e	396 [385.7–409.5] ^g	10.2 [7.9–12.5] ^{jk}
L75	77.11 (3.59) ^{ab}	98.2 (0.8) ^c	363.7 (74.6) ^e	392.5 [379.9–412] ^g	05.5 [4.5–6.4] ^l
L70	65.73 (4.94) ^a	97 (0.7) ^d	273.8 (41.9) ^f	293.1 [281.5–301.9] ^h	07.5 [6–9.1] ^{kl}
S80	Na	–	–	–	–
S75	218.9 (12.78) ^b	–	–	–	–
S70	137.7 (12.68) ^{ab}	96.9 (0.8) ^d	271.7 (44.5) ^f	273.9 [269.6–280;3] ⁱ	11.7 [9.8–13.5] ^j
Cont	–	99.4 (0.5) ^c	350.4 (49.5) ^e	371.5 [360–386.5] ^g	08.3 [6.4–10.1] ^{jk}
p-Value	0.0091	0.0009	<0.0001		

sd: standard deviation; σ : 3-point flexural strength; m : Weibull modulus; σ_0 : Weibull characteristic strength. In the viscosity, density and σ columns, there were no statistical differences between groups with the same superscript in. In the σ_0 and m columns, there was an overlap fraction of confidence intervals between groups with the same superscript letter.

Table 3 – Shrinkage after the heat treatment of alumina ceramics manufactured using the stereolithography (L80, L75, L70, S70) and subtractive methods (Cont) in terms of thickness, width, and length. Statistical analyses by column and by line are separated by a comma.

Sample name	Thickness shrinkage (%) Mean (sd)	Width shrinkage (%) Mean (sd)	Length shrinkage (%) Mean (sd)	p-Value	Overall shrinkage (%) Mean (sd)
L80	14.0 (1.8) ^{Aa}	19.1 (0.7) ^{Bd}	17.2 (0.3) ^{Ch}	<0.0001	16.9 (2.3) ^{ln}
L75	19.6 (0.7) ^{Db}	23.1 (1.1) ^{Ee}	19.3 (1.4) ^{Di}	0.0002	20.6 (2.1) ^{lm}
L70	23.0 (1.1) ^{Fc}	28.2 (1.5) ^{Gf}	22.9 (0.7) ^{Fj}	<0.0001	24.5 (2.6) ^m
S70	21.2 (1.9) ^{Hbc}	27.2 (0.5) ^{If}	22.7 (0.6) ^{Hj}	<0.0001	24.0 (2.6) ^m
Cont	15.6 (1.6) ^a	14.7 (1.2) ^{Jg}	14.6 (0.4) ^{jk}	0.3925	15.0 (1.2) ⁿ
p-Value	<0.0001	<0.0001	<0.0001		<0.0001

sd: standard deviation; There were no statistical differences between groups with the same superscript.

1.35 ± 0.14 mm in thickness, and 3.89 ± 0.09 mm in width and passed the D'Agostino Pearson normality test, indicating that they had good dimensional homogeneity.

2.2. Dynamic viscosities of the slurries

The dynamic viscosities of three slurries from each group were evaluated by dynamic rheometry using parallel plates (cone-plate 2°/55 mm) at 25 °C (Haake, France). The slurries were placed on the lower plate of the rheometer, and the instant-

taneous viscosities (Pas) were measured at a constant shear rate of 2 s⁻¹.

2.3. Densities of the sintered ceramic samples

The densities of the ceramic samples were determined by hydrostatic weighing on a high-precision balance (ALT-310-4 AM; Kern, Germany) using the Archimedes principle under water (3 vacuum cycles, n = 10/group).

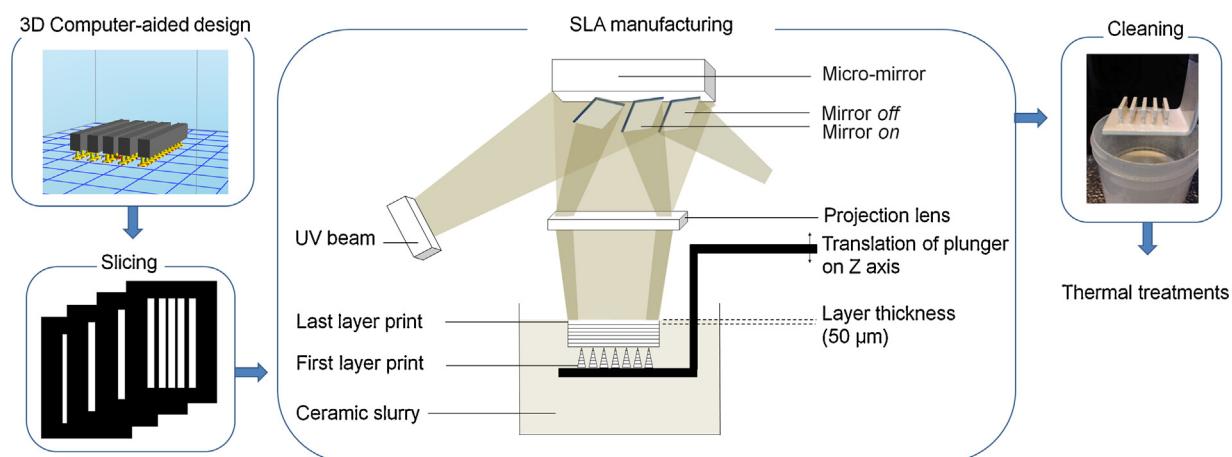


Fig. 1 – Stereolithography manufacturing process.

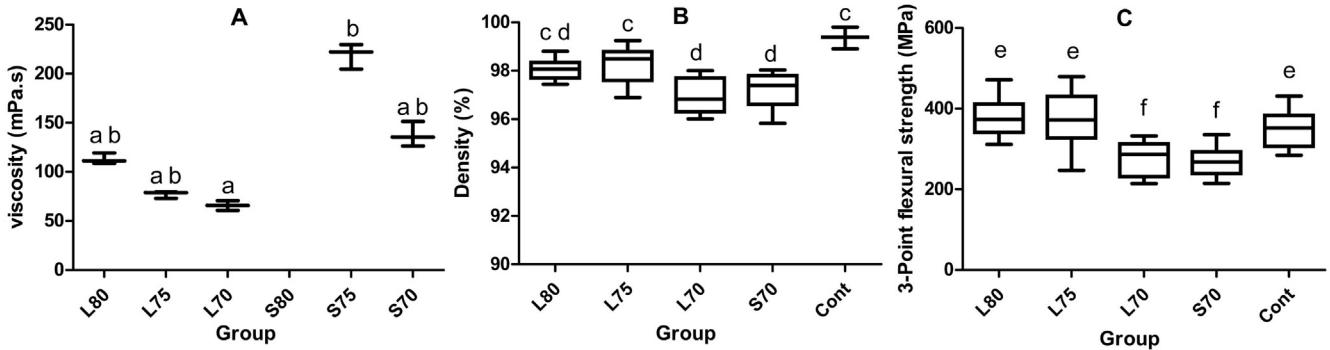


Fig. 2 – Boxplot (box: mean, Q1, Q3; whiskers: 10–90 percentile) of viscosity (A), density (B), and 3-point flexural strength (C) of alumina ceramics manufactured by the stereolithographic (L80, L75, L70, S70) and subtractive methods (Cont). There is no statistical difference between the groups with the same letter.

2.4. Three-point flexural strengths of the sintered ceramic samples

The samples ($n=15/\text{group}$) used to determine the 3-point flexural strength were evaluated using a bending machine (DY30; Adamel Lhomargy, France) equipped with a 100 N cell (Adamel Lhomargy, France) set to a maximum force of 1 kN. A progressive central vertical load was applied at a constant speed of 0.5 mm/min (ISO 6872:2008 standard). The distance between the lower supports was 20 mm. The flexural strength in MPa (σ) was calculated using following equation (Eq. (1)):

$$\sigma = (3 \times F \times L) / (2 \times W \times H^2) \quad (1)$$

where F is the maximum force to failure (N), L is the distance between the lower supports (mm), W is the sample width (mm), and H is the sample height (mm).

2.5. Weibull characteristics

The structural reliability of ceramics is commonly assessed by a Weibull distribution analysis using flexural strength data for two parameters. The Weibull modulus (m) and the Weibull characteristic strength (σ_0) were calculated using the following equation (Eq. (2)):

$$P_f = 1 - \exp \left[- \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (2)$$

where P_f is the probability of failure between 0 and 1, σ is the flexural strength in MPa, and σ_0 is the characteristic strength in MPa.

2.6. Shrinkage

The shrinkage of the samples was assessed by anisotropic retraction (thickness, width, length). The sample ($n=10/\text{group}$) sizes (d) were measured before and after the thermal treatment step using an analog micrometer

(Mitutoyo, Kawasaki, Japan). The shrinkage (S) ratios in each direction were calculated as follows (Eq. (3)):

$$S (\%) = \left[1 - \frac{d_{\text{after sintering}}}{d_{\text{before sintering}}} \right] \times 100 \quad (3)$$

2.7. Statistical analysis

The normality of the distribution of the data was verified using the Kolmogorov-Smirnov test ($\alpha=0.05$). The viscosity, density, and shrinkage values were analyzed using the non-parametric one-way Kruskal-Wallis test and Dunn's post-hoc test ($\alpha=0.05$). Three-point flexural strength was analyzed for statistical significance using a one-way parametric test and Tukey's multiple comparison post-test ($\alpha=0.05$). The statistical analyses were performed using GraphPad 5 software (Prism, CA, USA).

2.8. Feasibility of SLA-manufactured dental crown frameworks

A dental crown framework (maxillary premolar) was fabricated for an extracted human premolar (ethical committee approval no. DC-2008-642) that was prepared for an all-ceramic crown restoration. The optical impression was assessed using a Ceramill® Map400 scanner (Amann Girrbach, Germany). The crown framework was designed using Ceramill® Mind design software (Amann Girrbach, Germany). It was 35% oversized to compensate for shrinkage during sintering and to facilitate framework insertion and was SLA manufactured using the L80 slurry. Photographs were taken after sintering using a Nikon D3100 camera (Japan).

3. Results

The results of the physical (viscosity, density, shrinkage) and mechanical (flexural strength, Weibull modulus, Weibull characteristic strength) measurements are summarized in Tables 2 and 3. Figs. 2 and 3 shows box plots of the results to facilitate comparisons.

The only significant difference in terms of viscosity was observed between the S75 ($218.9 \pm 12.78 \text{ mPa.s}$) and L70

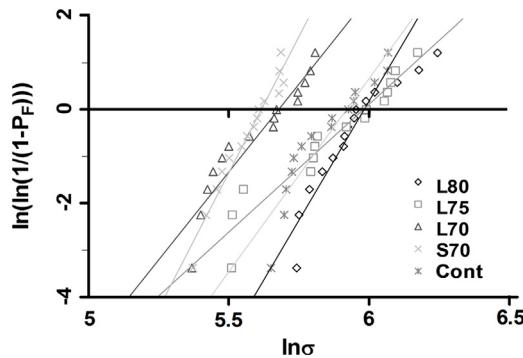


Fig. 3 – Weibull plot comparing the strength distribution of alumina ceramics manufactured by the stereolithographic (L80, L75, L70, S70) and subtractive methods (Cont).

(65.73 ± 4.94 mPa s) slurries ($p < 0.05$). The excessive viscosity of the S80 slurry made it impossible to take rheological measurements and impeded the SLA-manufacturing process. In addition, as can be seen in Fig. 4, the high viscosity of the S75 slurry resulted in macroscopic and microscopic deformations of the printed layers during the SLA-manufacturing process (Fig. 4C,D). This problem did not occur with the S70, L70, L75, and L80 slurries (Fig. 4A,B). Based on these results, the first null hypothesis that the effect of variations in ceramic slurry composition on the SLA-manufacturing process does not influence viscosity was rejected.

The viscosities of the S75 and S80 slurries were too high for the SLA-manufacturing process. They were thus excluded from further consideration.

The densities of the subtractive-manufactured samples ($99.4 \pm 0.5\%$) were higher than those of the SLA-manufactured L70 and S70 samples ($p < 0.05$). Even though there was no difference in density between the control of the subtractive-manufactured samples and the SLA-manufactured L80 ($98.1 \pm 0.4\%$) and L75 ($98.2 \pm 0.8\%$) samples ($p > 0.05$), the second null hypothesis that there is no difference in density

between SLA-manufactured and subtractive-manufactured alumina ceramics was rejected.

The flexural strengths of the L70 (273.8 ± 41.9 MPa) and S70 (271.7 ± 44.5 MPa) SLA-manufactured samples were lower than those of the L80 (367.9 ± 52.4 MPa), L75 (363.7 ± 74.6 MPa), and control (350.4 ± 49.5 MPa) samples ($p < 0.05$). The samples all had a Weibull modulus ranging from 5 to 15. Based on this, the third null hypothesis that there is no difference in flexural strength between SLA-manufactured and subtractive-manufactured alumina ceramics with high Weibull characteristics was rejected.

Table 3 shows that the overall shrinkage of the ceramics was lower for the control samples than for the SLA-manufactured ceramics ($p < 0.05$) but that the difference was not significant for the L80 group ($p > 0.05$). The results were similar when each direction of shrinkage was considered separately ($p < 0.05$). An increase in dry matter content decreased the shrinkage in each direction ($p < 0.05$). No significant difference was found between the S70 and L70 groups in terms of particle size ($p > 0.05$). The subtractive-manufactured samples displayed homogeneous shrinkage in all spatial planes ($p > 0.05$). However, the SLA-manufactured samples displayed more shrinkage in width than in length and thickness ($p < 0.05$). Only the L80 group displayed more shrinkage in length than in thickness ($p < 0.05$). Based on this, the fourth null hypothesis that there is no difference in shrinkage between SLA-manufactured and subtractive-manufactured alumina ceramics was rejected.

The ability to shape crown frameworks by additive manufacturing was tested in order to determine whether it was suitable for manufacturing objects for clinical applications. Fig. 5 shows an SLA-manufactured crown framework inserted on an all-ceramic crown preparation (L80 slurry) (Fig. 5C). The crown framework could be inserted on the all-ceramic crown preparation, but excessive oversizing generated a large marginal gap. The residual pillars and marginal gaps can still be seen (Fig. 5A). Based on this, the hypothesis that it is

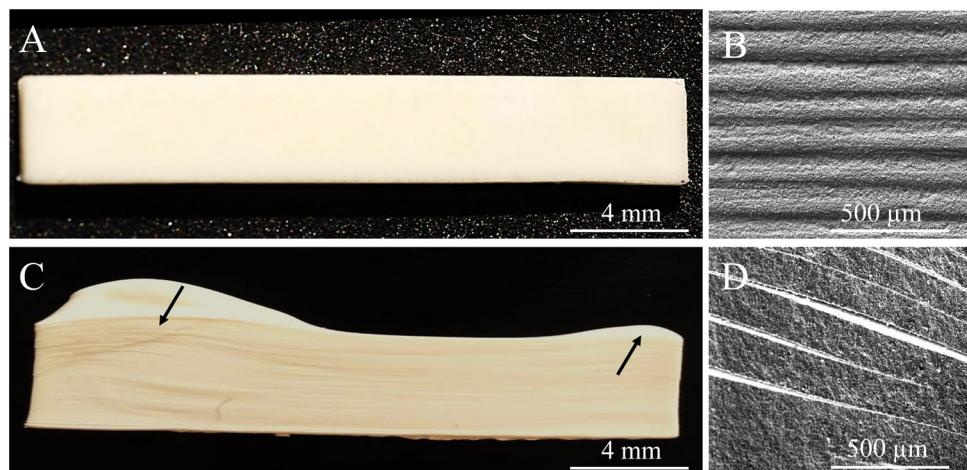


Fig. 4 – Representative photograph (A) and scanning electron micrograph (B) (magnification: $\times 90$) of a rectangular SLA-manufactured alumina sample made from the S70 slurry. The photograph and micrograph are also representative of the L70, L75, and L80 slurries. Representative photograph (C) and scanning electron micrograph (D) of a rectangular S75 sample (arrows = macroscopic deformations).

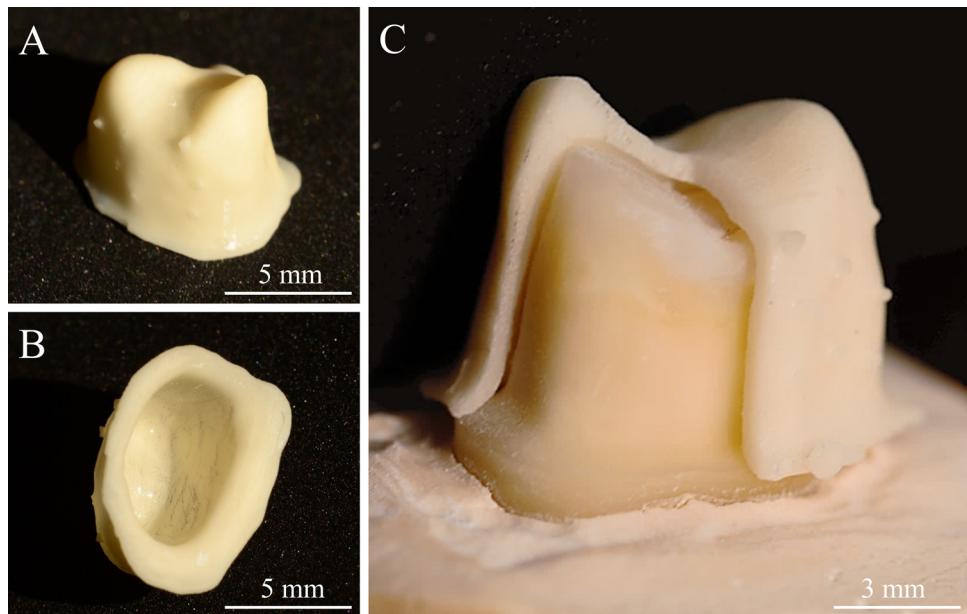


Fig. 5 – Photographs of an SLA-manufactured alumina dental crown framework: external (A) and internal (B) views. Crown framework in place on an all-ceramic crown preparation (C).

possible to produce dental crown frameworks by SLA manufacturing was confirmed.

4. Discussion

Subtractive methods are the gold standard for manufacturing dental ceramics by CAM [1,2]. Additive methods have certain advantages over subtractive methods in that they result in less waste, less stress on objects, and less instrument wear. However, little information is available in the literature on additive methods, especially with respect to the high mechanical strength needed for dental applications. SLA manufacturing shows promise [18], but the two methods have never been compared. The goal of the present study was to compare the physical and mechanical properties of SLA-manufactured alumina ceramics of different compositions (dry matter content, particle size) and adapted viscosity to those of subtractive-manufactured ceramics in order to produce suitable dental crown frameworks.

We showed that very viscous slurries are not compatible with the SLA-manufacturing process (acceptability window: 137.7 ± 12.68 mPa s to 218.9 ± 12.78 mPa s). While the viscosities of alumina slurries are well known for other ceramic shaping processes [19], this parameter has never been evaluated in terms of particle size and dry matter content for SLA manufacturing. Our previous results and those of others have shown that small particle sizes and high dry matter content increase viscosity [9,13]. Moreover, because it was impossible to increase the dry matter content of slurries composed of small particles, the viscosities of the L80 and S70 slurries were the most suitable for the SLA-manufacturing process (Fig. 4). In addition, resin composition can influence the rheological behavior of the slurries, which is why further studies on resin composition must be conducted [13].

In terms of densified ceramics, there was a significant difference in density between the subtractive-manufactured and the SLA-manufactured alumina ceramics with a 70% (w/w) dry matter content. However, the densities of the L80 and L75 slurries were similar to those of the subtractive-manufactured ceramics. We thus hypothesized that there are no variations in the densities of SLA-manufactured alumina ceramics with a dry matter content exceeding 75% and that these values are similar to subtractive-manufactured alumina ceramics. Indeed, Braga et al. [20] suggested that the higher shrinkage of low dry matter content slurries causes more crack formation in ceramics. Other parameters such as residual monomers [21] and unsuitable light curing strategies [22] can also cause cracks. However, these results must be tempered by the fact that the absence of significant differences between the L80, L75, and control slurries may have been due to the high standard deviation of the SLA-manufactured samples.

The flexural strength values followed the same trend as the density values. The SLA-manufactured alumina ceramics with a dry matter content equal to or exceeding 75% (w/w) as well as the controls complied with the ISO6872:2008 standard and were statistically different from ceramics with a 70% (w/w) dry matter content. These results suggested that there is a correlation between density and flexural strength [5]. The ceramic manufactured from the L80 slurry was the only sample that displayed a statistically significant difference between these two parameters. The results from the L70 and S70 ceramics indicated that particle size has no impact on flexural strength, which is in agreement with the study by Seidel et al. [23], who reported that variations in small grain size do not modify the flexural strengths of ceramics manufactured from fine alumina particles.

The Weibull moduli of all the samples were between 5 and 15, meeting dental ceramic requirements [15]. Our results also showed that the SLA-manufactured S70 and L80 ceramics had

the best Weibull moduli, with no confidence interval overlap with L75, and thus had the greatest structural reliability [17]. These results were similar to those of Schwentenwein & Homa [24], who reported an 11.2 Weibull modulus for SLA-manufactured alumina. A decrease in particle size improved the structural reliability of the S70 ceramic. However, a lower particle size required a low dry matter content to obtain a viscosity suitable for the SLA-manufacturing process, which decreased the flexural strength of the ceramics, making the L80 slurry more suitable for the SLA-manufacturing process. However, these conclusions have to be confirmed by complementary studies with a greater sample size, which would reduce the overlap fractions of the Weibull moduli confidence intervals.

Although the high dry matter content samples displayed the lowest shrinkage after the heat treatment, particle size did not influence shrinkage. As such, the highest dry matter content should be chosen to reduce the risk of deformation during shrinkage and to decrease material stress that can lead to the appearance of cracks [20]. The SLA-manufactured ceramics did not display isotropic shrinkage, unlike the subtractive-manufactured samples. In addition, there is less anisotropic shrinkage of additive-shaped ceramics in the plane of the printed layer [25]. Given this, further development of the SLA-manufacturing process requires that differential shrinkage be anticipated by providing adequate oversizing in order to improve the marginal adaptation of prostheses.

We were able to reproduce framework shapes from optical impression data using the SLA-manufacturing process. However, a number of parameters affected the process, leading to substantial marginal gaps. In general, although an oversize of 35% makes it feasible to manufacture complex morphologies, differential shrinkage leads to the deformation of the final structure. Further refinement of the SLA-manufacturing process is thus required, including a differential over sizing model that could make it possible to reduce marginal gaps between crown frameworks and crown preparations for clinical applications (Fig. 5C). In addition, the pillars on the upper surface of the crown frameworks had to be removed manually prior to the sintering step. Special care had to be taken to avoid placing them on critical areas such as the margin or under the prosthesis. For clinical applications, such crown frameworks need to be veneered.

The present study provided promising results for manufacturing dense 3D alumina crown frameworks by SLA.

5. Conclusion

The viscosity of the slurries influenced the SLA-manufacturing process. Compared to subtractive-manufactured ceramics, only the SLA-manufactured L80 slurry, with its high particle size ($d_{50} = 1.58 \pm 0.03$) and dry matter content (80% in weight), gave a reliable material with the anisotropic shrinkage, high density, flexural strength, and Weibull characteristics suitable for SLA manufacturing.

Our results showed that it is possible to build up a dense 3D alumina crown framework with a controlled shape using the SLA-manufacturing process. However, further studies on the marginal adaptation and shrinkage of alumina crown

frameworks will be required in order to take the direction of anisotropic shrinkage into consideration.

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