

Reinforcement of Different % Weights of Aluminum Oxide on the Flexural Strength of Heat-Polymerized Acrylic Resins

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Abstract

Acrylic resin denture base fracture is a primary clinical mode failure after denture delivery due to high impact forces or repetitive stress rate. The fracture can result from flexural fatigue or degradation of the base material. Therefore, it is necessary to increase the strength of denture base resins for its longevity. The purpose of this study was to evaluate the effect of 2.5 and 10 weight % silane-treated alumina oxide reinforcement on the flexural strength of heat-polymerized acrylic resins. Thirty rectangular (65x10x3 mm.³) (ISO standard 1567) heat-polymerized acrylic resins were fabricated (n: 10). The control group was an intact heat-polymerized acrylic resin group. The two experimental groups were reinforced with 2.5 and 10 weight % silane-treated aluminum oxide particles (50-70 µm.). The specimens were tested for flexural strength using a three-point bending test. One-way ANOVA and Tukey Honestly Significant Difference (HSD) were used for statistical analysis. Flexural strength of 10 weight % of silane-treated aluminum oxide reinforced groups (99.27 MPa) did not increase significantly compared with the control (99.25 MPa) (p-value<0.005). In conclusion, changing the percentage by weight of silane-treated alumina eniforced affected the flexural strength of heat-polymerized acrylic resins, as the flexural strength increased significantly when 10 weight % silane-treated alumina fillers were added.

Keywords: Aluminum oxide, alumina, Nano-alumina particles, Reinforcement, Denture base, Flexural strength, Heat cure acrylic resin

1. Introduction

Polymethyl methacrylate (PMMA) is the most commonly used material to fabricate denture base (Craig et al., 2003). The popularity of PMMA is mainly due to its superior properties such as biocompatibility, ease of processing, stability, low cost, and esthetic properties. However, several inferior physical and mechanical properties have prevented PMMA from becoming an ideal denture base material. Its low thermal conductivity, high coefficient of thermal expansion, relatively low modulus of elasticity, and brittleness made the material more susceptible to clinical failure (Craig R. G., 2001). One of the desirable properties of denture base material is high impact stress due to the risk of fracture that could happen if the patient drops their dentures. To achieve a longer clinical denture life, increasing the flexural strength may help resist torsional forces, which should be found in the function of acrylic resin denture base (Arora et al., 2015; Carroll et al., 1984).

Several methods have been introduced to reinforce the material used for denture base acrylic resin, such as chemical modification to prepare high impact resin and mechanical reinforcement with glass fibers, sapphire whiskers, aramid fibers, carbon fibers, metal wires, and plates, metal powder fillers, nylon polyethylene fibers and zirconia (Arora et al., 2015; Carroll et al., 1984). Glass fiber addition affected to higher impact strength and flexural strength of PMMA (Moreno et al., 2012; Sang-Hui et al., 2012). Several metal fillers like tin oxide, silver powder, and zirconium oxide have been used to improve the physical properties of acrylic resins (Woelfel, 1971). Titanium, zirconia, and nano-zirconia reinforcement also contributed to an increase in flexural and impact strength of PMMA (Asar et al., 2013; Safi, 2014; Zhang et al., 2011). A recent study showed that nano-zirconia may improve transverse strength of repaired acrylic denture base (Gad et al, 2016). However, the use of metal powder filler has caused the dentures to be

[250]

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unaesthetic. Ideally, the metal powder can be incorporated into the resin and increased strength without affecting other properties.

One of the materials that are commonly found in dental settings is aluminum oxide. Aluminum oxide, commonly referred to as alumina, possesses strong ionic interatomic bonding, giving rise to various desirable material characteristics. It can exist in several crystalline phases, which all revert to the most stable hexagonal alpha phase at elevated temperatures. It is the phase of particular interest for structural applications. Alpha phase alumina is the strongest and stiffest of the oxide ceramics. Its high hardness, excellent dielectric properties, refractoriness, and good thermal properties make it the material of choice for a wide range of applications in dentistry (Arora et al., 2015; Hamad, 2017). Antimicrobial properties are also another important property that nanoparticles should possess. Aluminum oxide nanoparticles have a wide range of applications in the industry and are also known to possess antimicrobial properties in the dental aspect. Alumina showed a mild bacterial growth-inhibitory effect, but only at very high concentrations (Abdulkareem & Hatim, 2015). Another research showed that the antimicrobial properties of nanoparticles containing formulations were increased (Acosta-Torres et al., 2011). The addition of 5 weight % aluminum oxide nanoparticles to acrylic resin also improved the thermal properties and transverse strength of acrylic resin, and at the same time decreased its water sorption and solubility. On the other hand, there was an increase in surface roughness of acrylic resin, yet the surface roughness did not significantly change even though the concentration of aluminum oxide nanoparticles was increased (Kul et al., 2016).

Treating aluminum oxide particles with a coupling agent resulted in an improvement of the flexural strength of acrylic resin (Kul et al., 2016; Yaadav et al, 2012). Moreover, silane-treated aluminum particles significantly increased the compressive, tensile, and flexural strength and the wear resistance of reinforced resin denture base (Chaijareenont et al., 2012; Yadav et al., 2012). Therefore, the application of silane on alumina surface prior to being reinforced in heat cure acrylic resins was used in the study.

Therefore, this research aims to investigate the effect of changing percentage by weight of aluminum oxide particle reinforcement on the flexural strength (MPa) of heat-polymerized acrylic resin. If the reinforcement of aluminum oxide particles increases the flexural strength of heat-polymerized acrylic resin, it may be used to facilitate the clinical success of denture base fabrication.

2. Objectives

The objective of the study is to determine the effect of changing percentage by weight of aluminum oxide particle reinforcement on the flexural strength (MPa) of heat-polymerized acrylic resin. The null hypothesis states that there is no significant difference between the flexural strengths of heat-polymerized acrylic resin that resulted from different volumes of filler reinforced. The alternate hypothesis states that there is a significant difference between the flexural strengths of heat-polymerized acrylic resin that resulted from different volumes of heat-polymerized acrylic resin that resulted from difference between the flexural strengths of heat-polymerized acrylic resin that resulted from different volumes of filler reinforced.

3. Materials and Methods

All PMMA specimens $(65*10*3 \text{ mm}^3)$ (ISO standard 1567) were fabricated from heat polymerization (Meliodent Heat Cure). All specimens were prepared using a heavy-body condensation silicone mold made of rectangular custom-made acrylic blocks. Pink wax was melted and placed into the silicone mold, where glass slides were used to control the thickness of the rectangular wax pieces. The rectangular wax pieces were invested in a metal flask with dental stone. After setting of dental stone, the flasks were placed in a scalding unit for wax boil-out (100°C, 5 minutes), leaving the rectangular-shaped mold cavity in the dental stone, which was used as a matrix for the fabrication of specimens. A separating medium was applied to the stone mold.

Then, aluminum oxide particles (50-70 μ m.) (Kepler international co., LTD Thailand) were preweighed into 2.5 wt % and 10 wt % of heat-polymerized acrylic resin particles using an electronic weighing machine. Then, 0.1% silane coupling agent MPS (3-methacryloxy propyl trimethoxysilane) was measured using a micropipette (10-100 μ l size, SCILAB) according to Arkle's equation:

[251]

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> amount of silane (g) = $\frac{\text{amount of filler (g) x surface area (m²/g)}}{\text{minimum coating area of}}$

Silane was used to coat the preweighed aluminum oxide particles using a micro brush and waited to dry for 1 minute. Then, mixing of preweighed aluminum oxide particles with resin polymer powder (50 mg.) was done by using a magnetic stirrer to achieve an equal distribution of particles and a uniform color (Sehajpal & Sood, 1986). Packing and processing of specimens were done according to the manufacturer's instructions. The upper and lower flasks were closed and maintained under 200 Ibs of compression for 30 minutes. The flasks were removed from the hydraulic press and cooled over the bench for 150 minutes. The curing procedure was processed by placing the flasks in the water bath at 71°C for 9 hours (Crag & Ward, 1997). Flasks were allowed to cool to room temperature overnight before opening and then deflasked. The specimens were removed from the mold, finished, and polished with 320-grit silicon carbide paper (Carbimet; Buehler, Lake Bluff, III) using a polishing machine. The specimens were later stored in distilled water at 37°C for 7 days before flexural strength testing (Vojdani et al., 2012).



Figure 1 Polished silane-treated 10 weight % aluminum oxide (particle size 50-70 µm.) reinforced heat-polymerized acrylic resin specimen (size 65*10*3 mm³)

Before flexural strength testing, the thickness, width, and length of each specimen were examined for accuracy by using a digital micrometer (minimum reading: 0.001 mm, Digimatic Micrometer Mitutoyo Corp., Kanagawa, Japan) (Figure 2). Hence, the midpoint in the length of each specimen was determined and marked. The flexural strength of the specimens was determined by using a three-point bending testing device in a universal testing machine (EZ test, Shimadzu, Japan) (Figure 3). The device is composed of a loading wedge and a pair of adjustable supporting wedges placed 50 mm apart. The specimens were centered on the device in such a way that the loading wedge, and set a crosshead speed of 5 mm/min, engaged the center of the upper surface of the specimens. Specimens were loaded until fracture occurred. Flexural strength was calculated using the following equation:

 $S = 3PI/2bd^2$

where S is the flexural strength (N/mm²), P is the fracture load (N), I is the distance between the supporting wedges (mm), b is the specimen width (mm), and d is the specimen thickness (mm).

[252]





Figure 2 Digimatic Micrometer



Figure 3 EZ test Shimadzu, Japan

4. Results and Discussion

4.1 Results

As shown in Table 1, in investigating the effect of percentage by weight of aluminum oxide particle reinforcement on the flexural strength of heat-polymerized acrylic resin, the alternate hypothesis was accepted. There was a significant difference between the flexural strengths of heat-polymerized acrylic resin that resulted from different volumes of filler reinforced. The mean flexural strength of the 10% aluminum oxide particle reinforced group was 11.194 MPa and 10.766 MPa higher than that of the control and 2.5% aluminum oxide particle reinforced groups, respectively, which appeared to be statistically significant (p < 0.05). Although the mean flexural strength of the 2.5% aluminum oxide particle reinforced group, the difference was not statistically significant (p > 0.05). As shown in Figure 4, as the percentage by weight of aluminum oxide particle increases from 2.5%, 5%, to 10%, the flexural strength of heat-polymerized acrylic resin specimen increases. Having said that, the only percentage by weight of aluminum oxide reinforcement that resulted in a significant increase in flexural strength was 10 weight %, whereas that of 2.5 and 5 weight % did not produce a significant difference.



Table 1 The effect of percentage by weight of aluminum oxide particle reinforcement on the flexural strength of heatpolymerized acrylic resin

% weight of aluminum oxide particle [I]	Mean flexural strength (MPa)	Standard deviation	Mean difference (p-value) [J]		
			Control	2.5%	10%
Control	99.25 2	3.993		-0.883 (1.000)	-11.194 (.008*)
2.5%	99.26 9	6.335	.0176 (1.000)		-11.177 (.008*)
10%	110.4 46	9.509	11.194 (.008*)	11.177 (.008*)	

ANOVA analysis and Post-hoc Tukey test were used for comparison of mean flexural strength (MPa) of all groups. Mean difference was calculated from [I]-[J]

* p-value less than 0.05 was considered to be statistically significant

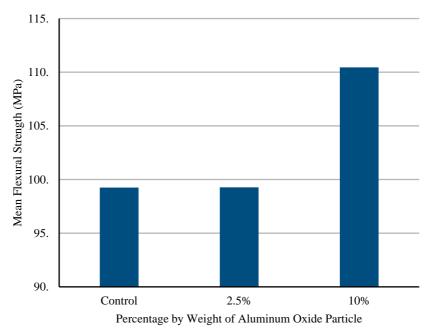


Figure 4 The effect of percentage by weight of aluminum oxide particle reinforcement on the flexural strength of heat-polymerized acrylic resin

4.2 Discussion

The present study investigates the effect of 2.5 and 10 weight % silane-treated aluminum oxide particles reinforcement on the flexural strength of heat-polymerized acrylic resin denture base. Zappini et al. (2003), showed that evaluating the strength of acrylic resins mostly depended on impact strength. However, flexural strength tests are better than impact strength tests to predict clinical function (Woelfel, 1971).

The result of the study demonstrated that flexural strength of heat-polymerized acrylic resin increased after the incorporation of 10 weight % silane treated alumina particles. Similarly, Chaijareenont P.

[254]

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et al., presented that 10 weight % treated particle with silane coupling agent 18-23 μ m particles led to a 23.86% increase in flexural strength (Chaijareenont et al., 2012). Besides, Ellakwa et al. (2008) reported that 10, and 15 weight % aluminum fillers that were added in denture bases provided an increase in flexural strength. Moreover, Vojdani M, et al. (2012) also revealed that reinforcement of the conventional heat-cured acrylic resin with Al₂O₃ powder significantly increased its flexural strength. In the present study, aluminum oxide particles that are 50-70 μ m in diameter and irregular in shape were used, as this particular size and shape of alumina particles are oftentimes used in dental settings for the process of sandblasting prosthetic materials. Therefore, they are easy to access and applicable in real clinical practices.

Although the increase in weight % of alumina results in an increase in flexural strength of acrylic, the increased chances of the void formation may occur. In the present study, aluminum oxide particles were pretreated with silane, which significantly improved the bond strength of alumina particles to PMMA. Therefore, the chances of void formation were reduced, and flexural strength significantly improved in higher weight % reinforced groups, specifically 10 weight % alumina reinforced groups (Vojdani et al., 2012).

The increase in flexural strength can be explained via the phenomenon of transformation toughening. Al_2O_3 is found in many crystalline phases, and all filler particles revert to the most stable hexagonal alpha phase at elevated temperatures. Its structural application is interesting (Ellakwa et al., 2008). The transformation phenomenon happens and reduces crack propagation when enough stress develops and micro cracks begin to propagate (Ayad et al., 2008). As a consequence, proper distribution of the filler within the matrix can stop or deflect cracks (Ellakwa et al., 2008).

All in all, the results obtained from this experiment can be beneficial in clinical practices. Seeing that 50-70 μ m aluminum oxide particles are used in the process of sandblasting for material surface treatment, they can be easily found in dental settings. The use of such aluminum oxide particles to reinforce PMMA in the process of denture base fabrication can improve the flexural strength of denture bases only when added at an adequate amount, which is 10 weight%. Increasing the flexural strength of denture bases may result in increased longevity of the prostheses. Having said that, the physical properties that may be affected by aluminum oxide reinforcement are brittleness and surface roughness, which are factors that can affect brittleness, plaque accumulation, and patient comfort. Therefore, these physical properties should be tested in further investigations before the application of aluminum oxide reinforcement.

5. Conclusion

From the research's results, it was concluded that the reinforcement with aluminum oxide particles on acrylic resin denture base improved flexural strength of the denture base when it was used in an adequate amount (10 wt %). Enhancing flexural strength could promote the long-term clinical success of the prosthesis and patient satisfaction.

However, as aforementioned, further research is required to examine the effect of aging on these reinforced denture base materials before clinical application. Besides, thermal conductivity, impact strength, and surface roughness of alumina reinforced acrylic resin should be considered.

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[255]

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[256]

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- 30 APRIL 2021
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[257]