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ORIGINAL ARTICLE

Effects of aluminum oxide addition on the flexural strength, surface hardness, and roughness of heat-polymerized acrylic resin

Mahroo Vojdani [a], Rafat Bagheri [a,b], Amir Ali Reza Khaledi [a*]

[a] *Biomaterial Research Center, School of Dentistry, Shiraz University of Medical Sciences, Shiraz, Iran*
[b] *Melbourne Dental School, University of Melbourne, Melbourne, Australia*

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KEYWORDS
aluminum oxide;
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hardness;
roughness;
flexural strength

Abstract *Background/purpose:* Acrylic dentures frequently fracture during service due to their poor strength characteristics. The aim of this study was to evaluate the effects of adding 0.5–5 wt% aluminum oxide (Al_2O_3) powder on the flexural strength, surface hardness, and roughness of a conventional heat-polymerized acrylic resin.
Materials and methods: In total, 50 specimens were prepared for each test. Specimens were divided into five groups ($n = 10$) coded A to E. Group A was the control group (without adding Al_2O_3). Specimens in the other four groups (B–E) were reinforced with Al_2O_3 at loadings of 0.5, 1, 2.5 and 5 wt%. Flexural strength was assessed with a three-point bending test using a universal testing machine. Hardness testing was conducted using a Vickers hardness tester. A surface-roughness test was performed with a profilometer.
Results: Data analyses using analysis of variance and Tukey'shonest significant difference tests showed that adding 2.5 wt% Al_2O_3 significantly increased the flexural strength compared to the control group ($P = 0.000$). The Vickers hardness significantly increased ($P < 0.05$) after incorporation of 2.5 and 5 wt% Al_2O_3. No significant difference ($P > 0.05$) was detected in surface-roughness levels between the reinforced and control groups.
Conclusion: Reinforcement of the conventional heat-cured acrylic resin with 2.5 wt% Al_2O_3 powder significantly increased its flexural strength and hardness with no adverse effects on the surface roughness.

* Corresponding author. Biomaterial Research Center, School of Dentistry, Shiraz University of Medical Sciences, Shiraz 713451836, Iran.
E-mail address: khalediamiralireza@yahoo.com (A.A.R. Khaledi).

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Introduction

One of the most widely used materials in prosthetic dentistry is polymethyl methacrylate (PMMA). Since its introduction to dentistry, it has been successfully used for denture bases because of its ease of processing, low cost, light weight, and color-matching ability. However, acrylic resin denture base materials have poor strength, including low impact strength and low fatigue resistance.[1−6] A study by Johnston and colleagues[7] showed that 68% of acrylic resin dentures break within a few years after fabrication. Flexural fatigue occurs after separate flexing of a material, whereas impact fractures occur when force is applied extraorally.[8]

Many attempts have been made to enhance the strength of acrylic denture bases including the addition of metal wires and cast metal plates.[9−12] The primary problem with using metal wire is poor adhesion between the wire and resin, which leads to insignificant enhancement of mechanical properties. Although metal plates increase the strength, they may be expensive and prone to corrosion.[9−12] Modifications of the chemical structure, by adding crosslinking agents or copolymerization with rubber, result in significant increases in impact strength. However, stiffness, fatigue resistance, and transverse strength are reduced.[13−15] Mechanical reinforcement of acrylics has also been attempted through the inclusion of fibers and metal inserts.[1,6,16−18] Although the inclusion of the fibers produced encouraging results, this method has various problems including tissue irritation, increased production time, difficulties in handling, the need for precise orientation, and placement or bonding of the fibers within the resin.[19] In the case of metal inserts, failure due to stress concentration around the embedded inserts has been reported.[5,18,20]

The incorporation of ceramic particles in various dental materials has been studied and found to be biocompatible, and it also improves mechanical properties.[20−28] In addition, the white color of the ceramic powder is not expected to compromise aesthetic appearances.[20,23,29,30] However, reinforcement methods should not have adverse effects on the mechanical properties of denture materials. The roughness of acrylic resin surfaces is a critical property because surface irregularities increase the likelihood of microorganisms remaining on the denture surface after the prosthesis is cleaned.[31,32] Another property that can influence the surface characteristics of acrylic resins is the hardness, which indicates the ease of finishing a material and its resistance to in-service scratching during cleaning procedures.[33]

Although it has been reported that untreated aluminum oxide (Al_2O_3) powder develops physical properties of high-impact acrylic resin,[20] there have been no investigations regarding the effect of Al_2O_3 powder on the mechanical properties of a conventional heat-cured acrylic resin. Therefore, we evaluated the effects of Al_2O_3 at five different concentrations on the flexural strength (FS), surface hardness, and roughness of a conventional heat-cured acrylic resin. The hypothesis was that adding Al_2O_3 would increase theFS, hardness and roughness compared to the control group (unreinforced acrylic resin specimens).

Materials and methods

Specimen preparation

A conventional heat-cured resin (Meliodent; Heraeus Kulzer, Newbury, Berks, UK) was used as a matrix component and Al_2O_3 powder with a medium grain size of 3 μm as a reinforcing agent (VITA Zahnfabrik, BadSackingen,Germany) (Table 1). For each test, 50 specimens were prepared; specimens were divided into five groups ($n = 10$) coded A to E. Group A was the control group (unmodified acrylic resin specimens). Specimens of the remaining four groups (B—E) were reinforced with Al_2O_3 powder to achieve respective loadings of 0.5, 1, 2.5 and 5 wt% (Fig. 1).According to studiesby Ellakwa et al[20] and Sehajpal et al,[18] for an even distribution of filler within the polymer matrix, Al_2O_3 powderwasmixed with resin powder and liquid monomer. The oxide powder and acrylic powder were thoroughly mixed using a mortar and pestle for initial mixing and blending, followed by hand tumbling in a plastic jar until a uniform color was achieved. The oxide—resin powder was mixed with monomer at a ratio of 2:1 by volume in a mixing jar with a tight-fitting lid.

FS testing

Specimens were fabricated using stone molds made by investing brass rectangles 65 mm ×10 mm ×3 mm.[20,24,34] After the stone had set, the rectangles were removed. The acrylic specimens were fabricated by packing the acrylic resin into the stone molds contained in denture flasks and curing them for 9 hours at 73.89 °C. The cycle was completed by boiling for an additional 30 minutes. The rectangular resin specimens were then deflasked. After removing flashes and trimming the edges, the specimens were ground with 320-grit silicon carbide paper to obtain a polished surface. Prior to FS testing, the dimensions of each specimen were measured with digital vernier calipers (Mitutoyo, Kawasaki, Japan). Specimens were stored in water at 37 °C for 7 days before FS testing. The FS was measured using a three-point bending test in a universal testing machine (Lloyds, LRX, Lloyds Instruments, Hampshire, UK) ata crosshead speed of 5 mm/min. The flexural strength was determined using the formula:

Table 1 Composition of the materials used in this study.

Material	Composition	Manufacturer
Meliodent	Powder: polymethyl methacrylate Liquid: methyl methacrylate, ethylene glycol dimethacrylate	Heraeus Kulzer Ltd., Newbury, Berks, UK
Alumina	Al_2O_3 powder (particle size 3 μm)	Vita Zahnfabrik, BadSackingen, Germany

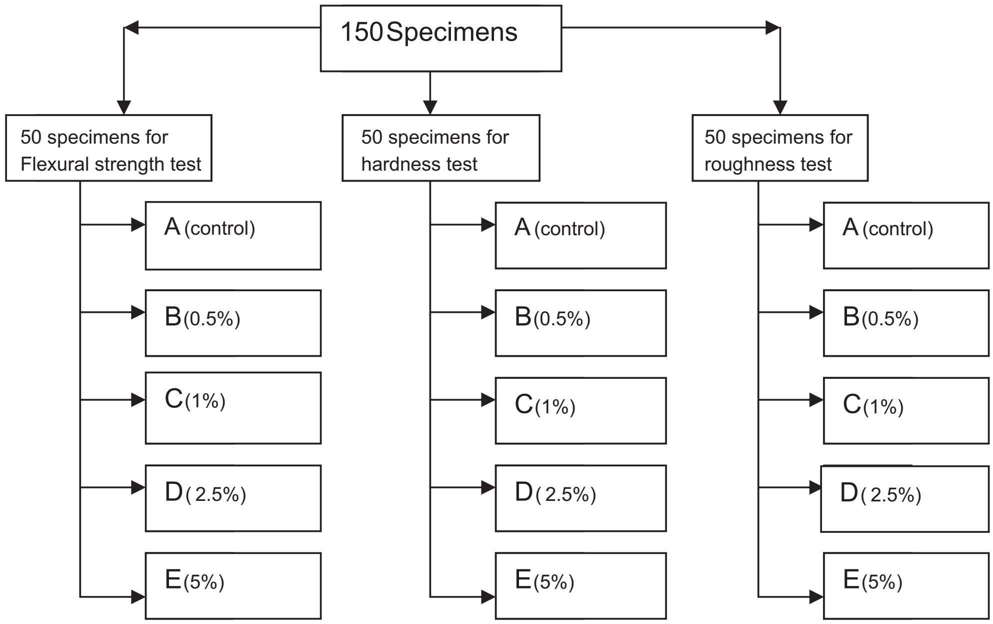

Figure 1 Classification of test specimens according to the concentration of Al_2O_3.

$$S=\frac{3Pl}{2bd^2}$$

[formula];

where S is the FS (MPa), P is the load at fracture (N), l is the distance between the supporting wedges (50 mm), b is the width of the specimen (mm), and d is the thickness of the specimen (mm).

Vickers hardness and surface roughness

Specimens were produced in molds prepared by the investment of brass dies (12 mm $\times$12 mm $\times$3 mm) within a flask.[35] The liquid/powder ratio of the polymer dough was mixed as mentioned earlier in specimen preparation. It was then inserted into the molds and packed. Specimens were polymerized using a long polymerization cycle followed by boiling for 30 minutes. After polymerization, specimens were visually inspected to have a smooth surface without voids or porosity. Specimens were manually wet-polished using a circular motion with a sequence of 600-grit, 800-grit, 1000-gritand 1200-grit silicon carbide papers. Each specimen was washed between each different-sized grit paper using an ultrasonic bath. In this manner, 50 specimens were prepared for the hardness test and 50 for the roughness evaluation.

To determine Vickers values, a load of 30 g was applied for 30seconds to specimens using a digital hardness tester (Otto Wolpert, Werke, Ludwigshafen, Germany). Each specimen was subjected to three indentations (one at the center and two at the border), and the average value was calculated for each group.

Surface roughness (Ra) of the acrylic specimens was measured using a profilometer (Surfcorder SE 1700, Kosaka, Japan) with a 0.01-μm resolution calibrated to a specimen length of 0.8 mm, 2.4 mm percussion of measure, and 0.5 mm/s. Three readings were made for each specimen, and the mean value was calculated.

Preparation of specimens for scanning electron microscopy

Using a randomized method, one sample from each group was coated with gold for imaging by scanning electron microscopy (SEM) (Stereoscan S-360, Cambridge, UK).

Data analysis

Descriptive statistics were carried out for each of the three tests. One-way analysis of variance was used to determine inter-group differences. *Posthoc* Tukey'shonest significant difference test was used to assess if the means significantly differed from those of the control group. Data were analyzed at a significance level of 0.05.

Results

The mean, standard deviation, and minimum and maximum values for FS,the Vickers hardness number, and roughness values are presented in Tables 2, 3 and 4, respectively.

One-way analysis of variance showed a significant difference between mean values of FS and surface hardness ($P = 0.000$). No significant difference was detected for surface roughness ($P > 0.05$).

Statistical analysis using the *posthoc* Tukey's honest significant differences test revealed that although 0.5% and 1% Al_2O_3 addition reinforced the acrylic resin specimens, this reinforcement was not significant compared to the control group. The FS significantly increased after incorporating 2.5% Al_2O_3 ($P = 0.000$), whereas for acrylic resin specimens containing 5% Al_2O_3, the FS significantly

Table 2 Mean flexural strength (MPa), standard deviation, minimum and maximum values.

Groups	Mean ± SD	Minimum	Maximum	P value
A (control)[a]	85.10 ± 2.34	81	89	
B (0.5%)[b]	86.22 ± 1.71	85	90	0.85
C (1%)[c]	85.20 ± 1.44	82	87	1.00
D (2.5%)[d]	90.51 ± 3.36	84	95	0.000*
E (5%)[e]	80.15 ± 3.17	76	86	0.001*

* Significance $P < 0.05$.
SD = standard deviation.
[a] without Al_2O_3.
[b] 0.5 wt% Al_2O_3.
[c] 1 wt% Al_2O_3.
[d] 2.5 wt% Al_2O_3.
[e] 5 wt% Al_2O_3.

decreased compared to that of the unreinforced acrylic resin ($P = 0.001$).

Increased mean hardness values were observed after reinforcing all acrylic resin specimens with Al_2O_3 powder. Yet, only specimens reinforced with 2.5 and 5 wt% Al_2O_3 showed a significant increase in hardness compared to the control group ($P < 0.05$). Incorporation of Al_2O_3 powder in the heat-cured acrylic resin produced a slight increase in the surface roughness of all reinforced groups, but not to a significant level ($P > 0.05$). A radiographic examination revealed that the addition of Al_2O_3 powder at concentrations of 0.5–5 wt% produced no appreciable differences in the radio-opacity of the acrylic resin specimens.

Figs 2, 3 and 4 respectively give results of the SEM microstructural examinations of the control, 2.5% Al_2O_3 and 5% Al_2O_3 specimens. The results of the SEM study showed that, at 2.5% loading, the oxides in the matrix of the resin were widely and evenly distributed. The addition of 5 wt% Al_2O_3 caused many voids to form within the resin specimens.

Discussion

We principally aimed to assess possible improvements in the mechanical properties of PMMA, in particular, the FS,

Table 3 Mean Vickers hardness number (kg/mm^2), standard deviation, minimum and maximum values.

Groups	Mean ± SD	Minimum	Maximum	P value
A (control)[a]	14.33 ± 1.33	12.20	16.20	
B (0.5%)[b]	15.11 ± 1.55	13.00	17.50	0.73
C (1%)[c]	15.58 ± 1.14	14.00	17.50	0.30
D (2.5%)[d]	16.46 ± 1.26	14.10	18.00	0.01*
E (5%)[e]	17.57 ± 1.75	14.80	20.60	0.000*

* Significance $P < 0.05$.
SD = standard deviation.
[a] without Al_2O_3.
[b] 0.5 wt% Al_2O_3.
[c] 1 wt% Al_2O_3.
[d] 2.5 wt% Al_2O_3.
[e] 5 wt% Al_2O_3.

Table 4 Mean roughness (μm), standard deviation, minimum and maximum values.

Groups	Mean ± SD	Minimum	Maximum	P value
A (control)[a]	0.41 ± 0.13	0.25	0.60	
B (0.5%)[b]	0.43 ± 0.10	0.31	0.60	0.99
C (1%)[c]	0.46 ± 0.12	0.20	0.62	0.93
D (2.5%)[d]	0.45 ± 0.15	0.25	0.72	0.96
E (5%)[e]	0.51 ± 0.16	0.35	0.85	0.52

[a] without Al_2O_3.
[b] 0.5 wt% Al_2O_3.
[c] 1 wt% Al_2O_3.
[d] 2.5 wt% Al_2O_3.
[e] 5 wt% Al_2O_3.

surface hardness, and roughness, through incorporating untreated Al_2O_3 particles.

There are three ways to improve the mechanical properties of PMMA: replacing PMMA with an alternative material; chemically modifying it; and reinforcing the PMMA with other materials.[5,6] Some of the resin materials from such developments exhibit an excellent balance of impact resistance and flexural properties. However, the processes of etching, preparing, positioning, and impregnation of fibers may be impractical for dental offices. Currently, the most popular material, as an alternative to conventional PMMA is a rubber-modified acrylic polymer. By contrast, this material has relatively poor FS, and long-term failure due to fatigue has occured.[5] Moreover, the high costs of these materials restrict their widespread use.[19]

Adding treated or untreated ceramic particles to improve the physical properties of acrylic resin bases is a controversial matter, and there are no conclusive results about the priority of each of the above particles.[20,25,28] However, the present study showed that addition of 2.5 wt% of untreated Al_2O_3 to a conventional heat-cured resin improved the mechanical properties of PMMA without essential additional processing steps. Therefore, the fabrication of dentures by this method is not time-consuming, which would encourage its routine use in dental laboratories due to its low cost and ease of handling and processing. If a cost-effective material with enhanced

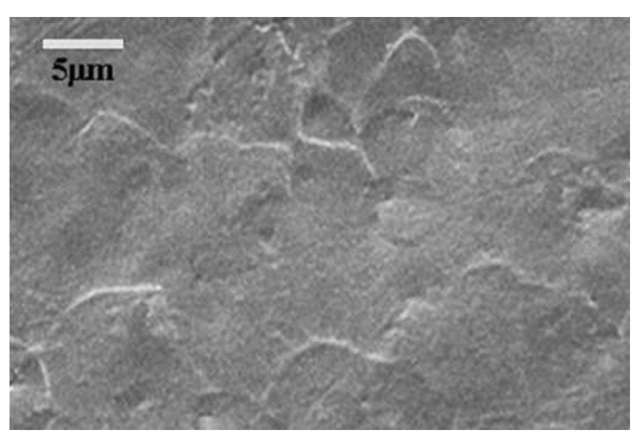

Figure 2 Scanning electron microscopy of acrylic resin without Al_2O_3.

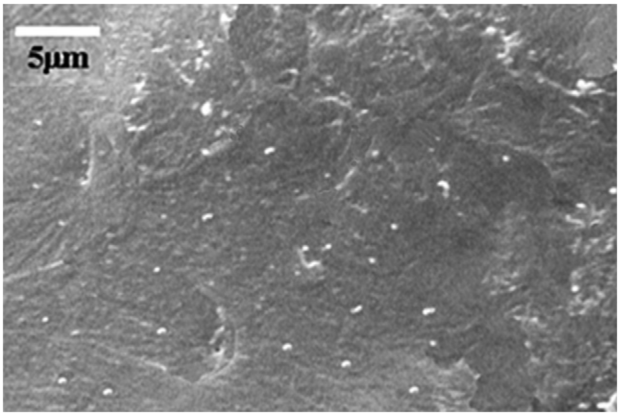

Figure 3 Scanning electron microscopy of acrylic resin reinforced with 2.5 wt% Al_2O_3.

mechanical properties were readily available, the health service expenditures of countries would be significantly reduced.[5]

Incorporating ceramic fillers in various dental materials has been studied, and it was biocompatible and improved mechanical properties.[20–29] Ellakwa and colleagues have reported that reinforcing high-impact acrylic resin (Diamond D) with untreated Al_2O_3 powder at concentrations of 5–20 wt% resulted in increases in both the FS and thermal diffusivity of this high-impact acrylic resin.[20] In our pilot study, Al_2O_3 was mixed with a conventional heat-cured acrylic resin to achieve loadings of 5, 10, 15 and 20 wt%. It was observed that the FS significantly decreased after incorporating the filler in proportion to the weight percentage of Al_2O_3 filler. Therefore, lower weight percentages of the filler (up to 5%) were selected for use in this study.

Results of this study showed that FS significantly increased after incorporating 2.5 wt% Al_2O_3. Addition of 2.5 wt% $Al_2O_3$was responsible for a 6.36% increase in FS. This increase in FS can be explained on the basis of transformation toughening. Al_2O_3 exists in several crystalline phases, and all filler particles revert to the most stable hexagonal alpha phase at elevated temperatures. This is the phase of particular interest for structural applications.[20,36] When sufficient stress develops and microcracks begin to propagate, the transformation phenomenon occurs, which depletes energy for crack propagation.[24] Therefore, proper distribution of the filler within the matrix can stop or deflect cracks.[25] The SEM evaluation of fracture cross-sections of specimens reinforced with 2.5 wt% Al_2O_3 showed a wide, even distribution of oxide particles within the resin matrix (Fig. 3). It seems that adding an appropriate amount of $Al_20_3$to PMMA did not significantly decrease the cross-section of the polymer matrix or cause void formation. The addition of 5 wt% Al_2O_3 powder caused a 5.82% decrease in FS. It appears that addition of 5–20 wt% Al_2O_3 significantly reduced FS compared to the control group. Possible explanations for this reduction in strength could be: a decrease in the cross-section of the load-bearing polymer matrix; stress concentration because of too many filler particles; changes in the modulus of elasticity of the resin and mode of crack propagation through the specimen due to an increased amount of fillers; void formation from entrapped air and moisture; incomplete wetting of the fillers by the resin; and the fact that Al_2O_3acts as an interfering factor in the integrity of the polymer matrix.[5,18,20] SEM examination of the samples revealed that, at a 5% loading, many voids had formed within the acrylic resin matrix (Fig. 4). Such defects can catalyze the failure process and might be an area in which crack propagation is initiated.

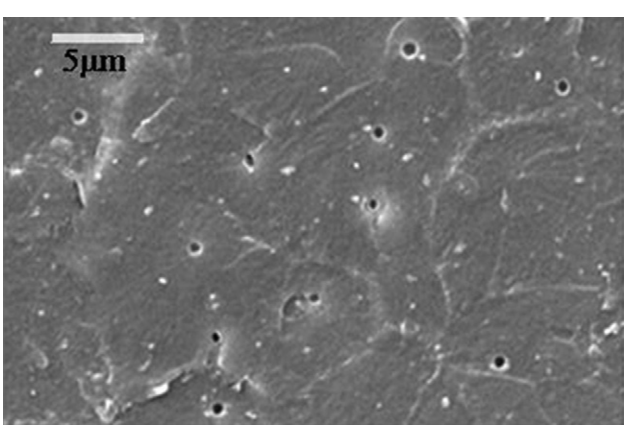

Figure 4 Scanning electron microscopy of acrylic resin reinforced with 5 wt% Al_2O_3.

We showed that the hardness increased in proportion to the weight percentage of the Al_2O_3 filler. The hardness significantly increased after incorporating 2.5 and 5 wt% Al_2O_3. This finding is in agreement with previous investigators,[22,28] who have concluded that reinforcing dental restorative resins and acrylic resin with ceramic particles can produce some improvements in the surface hardness. This increase in hardness may have been due to inherent characteristics of the Al_2O_3 particles. Al_2O_3 possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics, that is, hardness and strength. The most stable hexagonal alpha phase Al_2O_3 is the strongest and stiffest of the oxide ceramics. Therefore, it is expected that when Al_2O_3 particles disperse in a matrix, they increase its hardness and strength. Its high hardness, excellent dielectric properties, refractoriness, and good thermal properties make it the material of choice for a wide range of applications.[20,36] Furthermore, the white color of ceramic fillers is not expected to affect adversely the aesthetic appearance of denture base resins, as stated by others.[20,23,29,30]

We evaluated the effect of Al_2O_3 addition on the surface roughness of the acrylic resin material. The surface roughness of denture material is important, because it affects the oral health of tissues in direct contact with the dentures.[31,32] The surface roughness threshold for acrylic resin is 0.2 μm, below which no significant decrease in bacterial colonization occurs.[37] Dramatic colonization would be expected to occur on surfaces with a roughness value of 2.2 μm.[38] The surface roughness of polished acrylic resin varies between 0.03 μm and 0.75 μm. However, an important factor in the clinical performance of a material is the way it responds to hygiene procedures.[39] In agreement with the study of Saad-Eldeen et al,[26] the results of our study showed that incorporating Al_2O_3 at four different concentrations did not adversely affect the roughness of the denture base resin.

Radiographic evaluation showed that reinforcement with Al_2O_3 made no appreciable difference to the radio-opacity, because Al_2O_3 has a relatively low atomic weight.[20] Although the radio-opacity did not increase, the light weight of the acrylic resin denture base was retained.

Further research is needed to examine other physical and mechanical properties of PMMA reinforced with untreated ceramic particles. The effect of aging on these reinforced denture base materials also needs to be evaluated further before clinical application.

Within the limitations of this *in vitro* study, we concluded that the FS of a conventional heat-cured acrylic resin significantly increased when reinforced with 2.5 wt% Al_2O_3 powder; the surface hardness significantly increased after incorporating 2.5 and 5 wt% Al_2O_3; and the surface roughness of the Al_2O_3-reinforced acrylic resin did not significantly differ from that of the unreinforced denture resin.

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