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Effect of incorporating aluminum oxide nanoparticles on thermal conduction and flexural strength of acrylic resins

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ABSTRACT

Background: The mechanical and thermal properties of polymethyl methacrylate, as the most commonly used material for the fabrication of dental prostheses, should be improved due to its structural weaknesses. The present study aimed to compare the flexural strength and thermal conduction of two heat-cured and self-cured acrylic resins reinforced with aluminum oxide nanoparticles.

Materials and Methods: In this *in vitro* study, a total of 114 samples consisting of heat- and self-cured three subgroups (1% and 3% Al_2O_3 and the control) with 66 samples for the thermal conduction (n = 11) and 48 samples for the flexural strength (n = 8) tests were prepared. Flexural strength was assessed with a three-point bending test using a universal testing machine. One-way ANOVA was applied for data analysis, followed by *post hoc* Tukey paired group comparison tests ($P < 0.05$).

Results: An increase in the aluminum oxide nanoparticle percentage in acrylic resins increased the thermal conduction in heat-cured acrylic resin from 2.142 ± 0.0298 to 2.487 ± 0.0359 m (2)/sec and in self-cured acrylic resin from 2.0150 ± 0.02646 to 2.1475 ± 0.04031 m (2)/sec and decreased the flexural strength in heat-cured acrylic resin from 60.521 ± 8.9278 to 49.747 ± 4.4729 MPa and in self-cured acrylic resin from 37.573 ± 10.9237 to 35.569 ± 6.1531 MPa ($P < 0.05$).

Conclusion: The incorporation of aluminum oxide nanoparticles adversely affected acrylic resin flexural strength; however, it increased the thermal conduction.

Key Words: Aluminum oxide nanoparticles, flexural strength, nanoparticles, polymethyl methacrylate, thermal conductivity

INTRODUCTION

Modifications have been made in the polymers, including the coupling of copolymers with poly(styrene-butadiene) rubber to increase resistance to impact and overcome the physical and mechanical deficiencies of methacrylate resins.[1] Furthermore, triethoxyvinylsilane and oligomers in the polymer structure have been used to increase impact strength, flexural strength, and fatigue resistance, which has yielded positive results.[2]

In recent years, advances in nanotechnology science have resulted in efforts to improve the properties of these polymers by incorporating nanoparticles and synthesis of nanocomposite acrylic resins, which has led to some success; however, there are controversies

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in these studies. In a study by Ghaffari *et al*.,[3] the incorporation of nanoclay to heat-cured acrylic rein increased the thermal conduction; and decreased the flexural strength.

Hamedi *et al*.[4] reported that the use of silver nanoparticles resulted in an increase in thermal conduction and flexural strength of heat-cured acrylic resins; and decrease in tensile strength.

Aluminum oxide nanoparticles are one of the materials used to improve the properties of acrylic composite polymers. A study showed an improvement in the flexural strength and thermal conduction of heat-cured acrylic polymers after incorporating aluminum oxide nanoparticles into their structure.[5] In another study, incorporating 10% aluminum oxide into heat-cured polymethyl methacrylate improved the thermal conduction of denture bases; however, it did not change the acrylic resin's flexural strength.[6]

In addition, other studies on the properties of this material have shown that incorporation of this material into acrylic resin did not improve the physical and thermal properties of acrylic resins; however, it decreased acrylic resins polymerization shrinkage, increased its radiopacity, and prevented the growth of bacteria on the denture surface.[7,8]

Given the results of the studies above and the paucity of data on the effect of aluminum oxide nanoparticles on the flexural strength and thermal conduction of self-curd acrylic resins used in prosthetic procedures, the present study aimed to compare the mechanical and physical properties of autopolymerized resins reinforced with aluminum oxide nanoparticles with those of reinforced heat-polymerized acrylic resins. The hypothesis was that adding nano Al_2O_3 would increase flexural strength and thermal conduction compared to the control group (unreinforced acrylic resin specimens).

MATERIALS AND METHODS

In the present *in vitro* study with Code of Ethics IR. TBZMED. VCR. REC.1397.334, 114 samples in six groups (n = 8) were prepared for the flexural strength test and in six groups (n = 11) for the thermal conductivity test (57 samples for each of the autopolymerized or heat-polymerized acrylic resins).

First, self-cured (SR Triplex Self, Ivoclar Vivadent, Germany) and heat-cured (SR Triplex hot, Ivoclar Vivadent, Germany) acrylic resin powders were mixed with 1 and 3 wt% aluminum oxide nanoparticles (Sigma Aldrich, Cas Number, USA) in an ultrasonic device (Hielscher Ultrasonic Gmbh, UP200H, Germany), and samples with standard sizes for each of the mechanical test were prepared according to the manufacturer's instructions for flasking. The manufacturer's instructions were followed to ensure proper manipulation, distribution of acrylic resin, and powder-to-liquid ratios. The self-cured samples were coded as follows for blinding the evaluating observer and the operator carrying out statistical analyses: group S0, without aluminum oxide nanoparticles; Group S1, with 1 wt% of aluminum oxide nanoparticles; and Group S3, with 3 wt% of aluminum oxide nanoparticles. In addition, the heat-cured samples were named as follows: group H0, without aluminum oxide nanoparticles; Group H1, with 1 wt% of aluminum oxide nanoparticles; and Group H3, with 3 wt% of aluminum oxide nanoparticles.

The shape of the samples was determined in terms of the standards for the evaluation of mechanical variables.

The samples were prepared in the form of cylinders with a standard size (ISO 8302) to evaluate thermal conduction. The cylindrical specimens had a length of 38 cm and a diameter of 25 cm. For the preparation of these samples, cast iron flasks with the same shape and internal dimensions as the standard sample were produced. To prepare the cylindrical samples, PMMA (poly methyl methacrylate) powder and monomer were mixed. The inside of the muffle was lubricated with Vaseline. PMMA paste was placed inside the muffle, and it was overfilled a bit. A piece of special cellophane paper was placed on the paste surface, and the muffle door was inserted in place and tightened by screws. The muffle and PMMA complex were placed under the pressure apparatus, with pressure equalling 1.5 pounds to prevent bubble or void formation within the samples and to establish a PMMA paste with the same interior surfaces as the muffle. Then, the muffle was heated. After cooling, the flask was opened and the sample was removed.

The samples were prepared in the form of rectangular cubes with a standard size (2 cm × 2 cm × 20 cm) (ISO 178) for the evaluation of flexural strength. The specimens of this shape were also flasked and heated in prepared muffles, similar to the cylindrical samples.

The samples were polished with Emery paper after preparation, and their sizes were evaluated with a

digital Vernier (Digital Caliper, Guanglu, Strikhlu, Germany) accurate to ± 0.01 μm Then, the standard tests were carried out in each group to determine the flexural strength and thermal conduction.

Holes were produced at the two ends of the cylindrical samples at a distance of 6.5 mm from each other and placed in the machine used to measure the thermal conduction coefficient (Cussons Thermal Conductivity Apparatus, England).

The machine's two thermocouple systems were inserted into the two holes in the samples. Water flowed from the two ends of the samples, and the temperature difference between the two ends was determined by the thermometers connected to the machine. Then, the thermal conduction coefficient (K) was determined using the following formula by considering the parameter available.

$$K = \frac{j \times M \times L \times (T_2 - T_1)}{A \times t \times (t_2 - t_3)}$$

where

J = the 0º mechanical equivalent of heat = 0.186 J/kcal

M = mass of water

L = the sample length

A = the surface area

T = the time of flow

T_2 = the temperature of the outflowing water

T_1 = the temperature of the inflowing water

t_2 = the temperature of the cold end

t_3 = the temperature of the warm end

The flexural strength was measured with the three-point flexural machine (Gotech Inc., Baton Rouge, LA, USA) at a rate of 2.4 mm/min. The test was carried out with the ASTMD 790-10 standard.

The means, medians, and modes were calculated in each group. The distribution curve was evaluated for the normality of the data. One-way ANOVA was used to analyze the differences between the groups, followed by *post hoc* Tukey tests, using SPSS17 software (Chicago, IL, USA). Statistical significance was set at $P < 0.05$.

RESULTS

The results showed that the thermal conduction in control, heat-cured, and self-cured acrylic resin samples was significantly less than that in the samples containing 1 and 3 wt% of aluminum oxide nanoparticles ($P < 0.05$). In addition, the thermal conduction of the heat-cured and self-cured acrylic resin samples containing 1 wt% of aluminum oxide nanoparticles was significantly less than that of the samples containing 3 wt% of aluminum oxide nanoparticles ($P < 0.05$).

A comparison of the thermal conduction of heat-cured and self-cured acrylic resins reinforced with 1 and 3 wt% of aluminum oxide nanoparticles and the control group showed that at 1 and 3 wt% of aluminum oxide nanoparticles, the thermal conduction of heat-cured acrylic resin was significantly higher than that of the self-cured acrylic resin ($P < 0.05$) [Table 1].

The results showed that the flexural strength of the self-cured acrylic rein samples in the control group was significantly higher than that of the samples reinforced with 1 and 3 wt% of aluminum oxide nanoparticles ($P < 0.05$). The flexural strength of the samples containing 1 and 3 wt% of aluminum oxide nanoparticles was similar. The flexural strength of heat-cured acrylic resin samples in the control group was significantly higher than that of the samples containing 1 and 3 wt% of aluminum oxide nanoparticles ($P < 0.05$). The flexural strength of the heat-cured samples containing 3 wt% of aluminum oxide nanoparticles was significantly less than that of the samples containing 1 wt% of nanoparticles ($P < 0.05$).

Comparison of the flexural strength of heat-cured and self-cured acrylic resin samples reinforced with 1 and 3 wt% of aluminum oxide nanoparticles and the control samples showed that at 1 and 3% wt% of aluminum oxide nanoparticles, the flexural strength of heat-cured acrylic resin samples was significantly higher than that of self-cured acrylic resin samples ($P < 005$) [Table 2].

Figure 1 presents the electron microscope image of the fractured surfaces of the samples manufactured with different percentages of aluminum oxide nanoparticles.

Figure 2 presents the mapping images of the distribution of aluminum oxide nanoparticles with the samples containing different concentrations of nanoparticles.

Table 1: Comparison of thermal conduction (m^2/s) between two acrylic resins at two concentrations of nanoparticles

Groups	1%, mean±SD	*P*	Mean±SD		*P*
			3%	Control	
Heat-cured acrylic resins	2.142±0.0298	0.000*	2.487±0.0359	1.955±0.0465	0.000*
Self-cured acrylic resins	2.0150±0.02646	0.000*	2.1475±0.04031	1.7750±0.03416	0.000*
P	0.001*		0.000*		

*Significance *P*<0.05. Control: Without Al_2O_3; 1%: 1 wt% Al_2O_3; 3%: 3 wt% Al2O3. SD: Standard deviation

Table 2: Comparison of the flexural strength (MPa) of two acrylic resins at two concentrations of nanoparticles

Groups	1%, mean±SD	*P*	Mean±SD		*P*
			3%	Control	
Heat-cured acrylic resins	60.521±8.9278	0.045*	49.747±4.4729	76.5552±7.34728	0.009*
Self-cured acrylic resins	37.573±10.9237	0.047*	35.569±6.1531	50.967±4.9462	0.038*
P	0.032*		0.020*		

*Significance *P*<0.05. Control: Without Al_2O_3; 1%: 1 wt% Al_2O_3; 3%: 3 wt% Al_2O_3. SD: Standard deviation

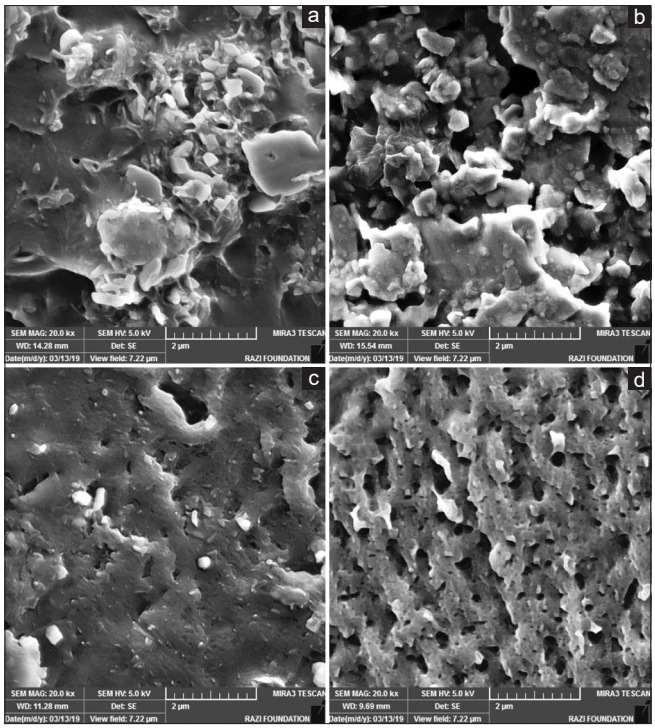

Figure 1: The electron microscope images of the samples. (a) 1 wt% heat-cured sample; (b) 3 wt% heat-cured sample; (c) 1 wt% self-cured sample; (d) 3 wt% self-cured simple.

Evaluation of the images above shows a more homogeneous distribution at lower concentrations. An increase in the concentration of the reinforcing agents led to the loss of homogeneous distribution, increasing the tendency for agglomeration, which is an inherent property of nanoparticles. Porosities are abundant around the nanoparticles, and the size and amount of these porosities increase with an increase in the concentration of nanoparticles.

DISCUSSION

In the present study, the null hypothesis regarding the effect of adding aluminum oxide nanoparticles into acrylic resins on flextural strength was rejected, but with regard to thermal conduction, it was confirmed.

The incorporation of aluminum oxide nanoparticles into heat- and self-cured acrylic resins improved their thermal conduction. The thermal conduction in the heat-cured group was significantly higher than that in the self-cured group. In both acrylic resins, an increase in the weight percentage of aluminum oxide nanoparticles increased the thermal conduction significantly ($P = 0.000$).

An improvement in the thermal conduction property might be attributed to the proper distribution of aluminum oxide nanoparticles within the samples and the creation of thermal conduction pathways within the resins.[5]

Consistent with the present study, the incorporation of different fillers (microfillers and nanofillers) into the resin matrix has improved the thermal characteristics of these resin polymers. However, the type and concentration of fillers and their distribution within the resin matrix affect the improvements of these properties in different studies.[3-6,9-11]

All the studies above have confirmed the results of the present study. The only difference is that higher concentrations (>10%) of the reinforcing agents were used in them. Lack of definite results in lower concentrations of nanoparticles was the main drawback and addressed completely in the present study.

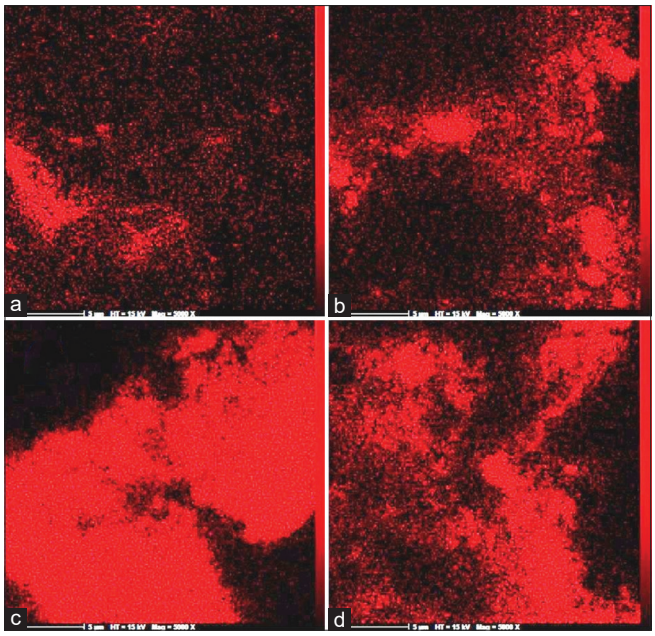

Figure 2: Mapping images of the samples. (a) 1 wt% heat-cured sample; (b) 3 wt% heat-cured sample; (c) 1 wt% self-cured sample; (d) 3 wt% self-cured sample.

In the present study, the flexural strength of both self- and heat-cured acrylic resins in the reinforced groups was significantly less than that in the control groups, and the decrease in the self-cured group was more significant. There was a correlation between this decrease in the mechanical properties with an increase in the concentration of the reinforcing agent, i.e., an increase in the concentration of aluminum oxide nanoparticles decreased the flexural strength in both self-cured and heat-cured acrylic resins.

Yadav *et al*., consistent with the results of the present study, showed that the incorporation of 5% aluminum oxide into heat-cured polymethyl methacrylate resulted in a decrease in the flexural strength of these resins, which was attributed to the microcracks at the reinforcing agent–acrylic resin interface.[12] In another study, the incorporation of 10% aluminum oxide into heat-cured polymethyl methacrylate did not change its flexural strength.[6] On the other hand, a study showed that the incorporation of aluminum oxide nanoparticles into self-cured and heat-cured acrylic resins resulted in a more significant increase in the flexural strength of self-cured than that of heat-cured acrylic resins.[13]

Pentapati *et al*. also showed that the reinforcement of the conventional heat-cured acrylic resin and high impact heat-cured acrylic resin with 15% aluminum oxide powder significantly increased its flexural strength with no adverse effect.[14]

In a study by Ellakwa *et al*., the incorporation of aluminum oxide nanoparticles into heat-cured acrylic resin polymers improved their flexural strength. An increase in the volumetric percentage of aluminum oxide nanoparticles from 5% to 20% (5%, 10%, 15%, and 20%) resulted in an increase in flexural strength.[5] The same result obtained in the study by Monika showed that the increasing of nanoparticles from 8% to 13% resulted in more flexural strength;[15] aluminum oxide reinforcement even showed better results in repairing the heat-polymerized acrylic resin.[16]

Besides, a study by Arora *et al*. showed that the incorporation of silver and aluminum oxide particles at 25 wt% into heat-cured acrylic resins improved the flexural strength compared to the control group.[11]

Considering the results of the studies above, it appears that the incorporation of high concentrations of aluminum oxide nanoparticles increases the flexural strength, which is due to the formation of scaffolds within the resin material that increase the resistance of the samples to fracture when a force is applied.

The degree of conversion and residual monomer are among the factors that could affect the physical and thermal properties of polymers, and any alteration in polymer structure would result in different ratios of degree of conversion. Hamouda *et al*., in a study, showed that aluminum oxide powder reinforcement was slightly increased the degree of conversion of the heat-cured acrylic resin and reduced the released monomer.[17]

However, in the present study, the incorporation of aluminum oxide nanoparticles at 1 and 3 wt% resulted in a decrease in this mechanical property, which was attributed to the lack of formation of scaffolds within the resin due to the inadequate concentration of the additive and the formation of porosities around the aluminum oxide nanoparticles within the acrylic resin in electron microscopy studies. The simultaneous use of high and low percentages of aluminum oxide and other nanoparticles is suggested in future studies. And besides, because of the high positive correlation between the degree of conversion and flexural strength, and negative correlation between the degree of conversion and monomer release in polymers, it is suggested that the effect of these two main determining factors in mechanical properties of acrylic resins should be evaluated more exactingly.

In this study, the incorporation of aluminum oxide nanoparticles into acrylic resin was studied only on

flextural strength and thermal conduction. The authors suggest future studies on adding other nanoparticles into acrylic resins and evaluating other physical properties. And also, the other processing methods of acrylic resins such as injection molding or microwave would be assessed.

CONCLUSION

1. Incorporation of 1 and 3 wt% of aluminum oxide nanoparticles increased the thermal conduction of acrylic resin samples
2. An increase in the concentration of aluminum oxide nanoparticles was associated with increased thermal induction
3. The effect of incorporating nanoparticle powder to heat-cured acrylic resins was higher than that in cold-cured acrylic resins
4. These nanoparticles decrease the flexural strength of acrylic resin samples compared to the control group
5. Decreased flexural strength was more prominent in higher percentages of nanoparticles
6. Decreased flexural strength was higher in self-cured acrylic resins than heat-cured acrylic resins
7. It is preferable to use nanoparticles in heat-cured acrylic resins than in cold-cured acrylic resins.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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