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Effect of Aluminum Oxide Addition on the Flexural Strength and Thermal Diffusivity of Heat-Polymerized Acrylic Resin

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Abstract

Purpose: This work was undertaken to investigate the effect of adding from 5% to 20% by weight aluminum oxide powder on the flexural strength and thermal diffusivity of heat-polymerized acrylic resin.

Materials and Methods: Seventy-five specimens of heat-polymerized acrylic resin were fabricated. The specimens were divided into five groups (n = 15) coded A to E. Group A was the control group (i.e., unmodified acrylic resin specimens). The specimens of the remaining four groups were reinforced with aluminum oxide (Al_2O_3) powder to achieve loadings of 5%, 10%, 15%, and 20% by weight. Specimens were stored in distilled water at 37˚C for 1 week before flexural strength testing to failure (5 mm/min crosshead speed) in a universal testing machine. Results were analyzed by one-way analysis of variance and post hoc Tukey paired group comparison tests ($p < 0.05$). Weibull analysis was used to calculate the Weibull modulus, characteristic strength, and the required stress for 1% and 5% probabilities of failure. Cylindrical test specimens (5 specimens/group) containing an embedded thermocouple were used to determine thermal diffusivity over a physiologic temperature range (0 to 70°C).

Results: The mean flexural strength values of the heat-polymerized acrylic resin were (in MPa) 99.45, 119.92, 121.19, 130.08, and 127.60 for groups A, B, C, D, and E, respectively. The flexural strength increased significantly after incorporation of 10% Al_2O_3. The mean thermal diffusivity values of the heat-polymerized acrylic resin (in m^2/sec) were 6.8, 7.2, 8.0, 8.5, and 9.3 for groups A, B, C, D, and E, respectively. Thermal diffusivities of the composites were found to be significantly higher than the unmodified acrylic resin. Thermal diffusivity was found to increase in proportion to the weight percentage of alumina filler, which suggested that the proper distribution of alumina powders through the insulating polymer matrix might form a pathway for heat conduction.

Conclusion: Al_2O_3 fillers have potential as added components in denture bases to provide increased flexural strength and thermal diffusivity. Increasing the flexural strength and heat transfer characteristics of the acrylic resin base material could lead to more patient satisfaction.

Acrylic resin and rubber-reinforced acrylic polymers represent approximately 95% of the denture base materials used in prosthodontics.[1] Acrylic resins have been successful as denture bases because of their ease of processing, low cost, light weight, and color matching ability;[2] however, acrylic resin denture base materials are low in strength, brittle, and low in thermal conductivity.[3,4] A variety of physical properties can be used to assess the strength of denture materials. The most common tests are impact strength, the ability of a material to resist a sudden high level force or "shock," flexural strength, and the force needed to deform the material to fracture or irreversible yield. Because of the risk of fracture if patients drop their dentures, high impact strength is a desirable property. Given the function of a denture base in a removable prosthesis, high flexural strength, flexural modulus, and a large yield point distance would help resist torsional forces in function, leading to a longer clinical service life for the prosthesis.

Although often overlooked, the heat transfer characteristics of the denture base material may be an important factor in determining patient satisfaction. For example, it has been established that food temperature significantly affects the perception of taste.[5-8]

Because the process of eating consists of frequent and abrupt changes in the temperature of food, thermal characteristics of the denture base material can become important factors affecting the gustatory response, for example, chemical perception of taste, smell, textural perception, and temperature. In a study by Kapur and Fischer,[9] thermal conductivity, or rather, the transient thermal conductivity (thermal diffusivity) of the denture base material was found to have an important effect on parotid secretions, which have been shown to be a good approximation of gustatory sensitivity.[10]

In edentulous patients wearing full dentures, the palate is partially covered by the denture base; consequently, the ability to sense transient temperature changes at the palate may be affected by the thermal characteristics of the denture base material. The acrylic polymer most commonly used for denture bases, polymethyl methacrylate (PMMA), has a thermal conductivity of approximately 0.2 W/min/°K,[3,11] which is approximately three orders of magnitude less than most metals. It is therefore not surprising that thermal conductivity has been one of the properties of acrylic resins most often associated with their replacement with metal as a denture base material (for example, gold and chromium cobalt alloys);[1,11-13] however, the use of metal as a denture base material has several disadvantages including increased weight of the denture, difficulty with tissue replacement in cases where substantial loss of bone has occurred, difficulty restoring denture borders within physiologic boundaries, difficulty with the relining process, esthetics, and high cost.[1]

Because of the disadvantages associated with metallic denture base materials, exploring the development of acrylic-based materials with improved thermal diffusivity is of interest. One approach to improving thermal diffusivity of denture base acrylic resins is to introduce a more thermally conducting phase within the insulating acrylic resin matrix, which creates a composite denture base material. Others have attempted to add particles of a conducting material, namely metal, to the powder or liquid resin and polymerize according to established procedures, increasing thermal conductivity modestly;[14] however, to significantly increase thermal conductivity with this approach requires high metal powder loading (>25%), which alters some of the beneficial characteristics of acrylic resins, such as increased density due to the presence of large amounts of metal, lower tensile strength and toughness, and diminished esthetic appearance due to the metal.

Aluminum oxide, commonly referred to as alumina, possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics. It can exist in several crystalline phases, which all revert to the most stable hexagonal alpha phase at elevated temperatures. This 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.[15]

In the literature, few attempts have been made to develop acrylic resins that possess increased mechanical properties and heat transfer characteristics. The aim of this study was to evaluate the effect of adding Al_2O_3 powder on the flexural strength and thermal diffusivity of heat-polymerized acrylic resin denture base.

Materials and methods

Specimen preparation for flexural testing

Teflon rectangles measuring 65 × 10 × 3 mm^3 (ISO 1567 Standard)[16] were invested in flasks with dental stone (Durguix, Type III, Protechno, Vilamalla, Girona, Spain). After the setting of the stone, the flasks were opened, and the Teflon rectangles were removed, leaving rectangular-shaped cavities in the stone, used as matrices for the fabrication of heat-polymerized acrylic resin beam specimens.

Seventy-five specimens of heat-polymerized acrylic resin were fabricated. The specimens were divided into five groups ($n = 15$) coded A to E. Group A was the control group (i.e., unmodified acrylic resin specimens). The specimens of the remaining four groups were reinforced with Al_2O_3 powder fillers (Junsei Chemical Co., Ltd., 4-4-16, Nihonbashi-Hoancho, Tokyo, Japan) to achieve loadings of 5%, 10%, 15%, and 20% by weight. The dental stone of the mold was lubricated with a thin layer of acrylic separating film (Isolmajor, Major, Moncalieri, Italy).

Aluminum oxide powder was added to the polymer of heat-polymerized acrylic resin (Diamond D, Keystone, Cherry Hill, NJ). The monomer and polymer of the heat-polymerized acrylic resin were proportioned, mixed, packed, and pressed into the mold following manufacturers' instructions. For specimens that contained Al_2O_3 powder, preliminary experiments were conducted to determine the best method of filler incorporation and revealed that simple mixing of Al_2O_3 powder fillers with resin powder and liquid monomer resulted in even distribution of filler within the polymer matrix.

After polymerization of the resin, the flasks were allowed to cool to room temperature before opening. The rectangular resin specimens were then deflasked. Flash and excess acrylic resin were removed by trimming the edges with tungsten steel burs using a handpiece at low speed, and with additional hand smoothing using 320-grit silicon carbide paper. All specimens were stored in distilled water at 37°C for 1 week before flexural strength testing.

Flexural strength testing

Prior to flexural strength testing, the width and thickness of each specimen were measured with a digital caliper (Fowler Tools & Instruments, Boston, MA) with measuring accuracy of ±0.1 mm. This procedure was necessary because after the trimming and polishing procedures the original size of each specimen was altered.

The flexural strengths of the specimens were determined using a 3-point bending testing device in a universal testing machine (Instron, Model TM 5565, Canton, MA). The device consisted 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, set to travel at 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:[16-18]

$$S = 3PI/2bd^2,$$

where S = flexural strength (N/mm^2), P = load at fracture (N), I = distance between the supporting wedges (mm), b = width of the specimen (mm), and d = thickness of the specimen (mm).

Specimen preparation for thermal diffusivity testing

Solid aluminum cylinders (10-mm diameter × 10-mm length) were invested in flasks with dental stone. Removal of the aluminum cylinders after hardening of the stone left cylindrical cavities used for molding the specimen cylinders. Five cylindrical specimens were fabricated for each group. The powder and liquid components of the resin were mixed into a pliable mass and then loaded into the stone molds and pressed at 200 psi to form the resin cylinders. Flasks were then briefly opened to expose the specimens, and each cylinder was carefully separated into equal halves with a razor blade. Type K thermocouples (0.127-mm wire diameter) were then inserted with the bead junction located at the center of the cylinder with the wires exiting one face of the cylinder. The two halves of the cylinder were then replaced, and the flask pressed a final time at 4000 psi. Specimens were cured in water for 8 hours at 72°C, followed by 1 hour at 100°C. After cooling to room temperature in water, specimens were removed from the molds and accurately measured with a micrometer to determine the radius (R) and length (L) of the cylinders.

Thermal diffusivity (Δ) was measured by using a technique developed by Watts and Smith[19] to characterize the thermal properties of dental materials. This technique allows accurate and rapid determination of Δ by using specimens of cylindrical geometry that contain an embedded thermocouple for temperature measurement within the specimen. Specimen thermocouple leads were attached to a personal computer recorder for measurement of transient temperature changes at the center of the cylinder. All specimens were conditioned at room temperature in distilled water for 24 hours before measurement and were equilibrated in a $0 \pm 1°C$ (T_o) ice bath for 30 minutes before testing. The instrumented specimens were then immersed in a thermostated bath at $70 \pm 1°C$ (T_s) and the temperature at the center of the specimen (T) was recorded each second for up to 10 minutes. The resolution of all temperature measurements was 0.1°C. The temperature range used in this study (0 to 70°C) was chosen to correspond to the temperatures of food and drink likely to be ingested in a typical meal.[20]

Temperature versus time profiles (Fig 1) were obtained and temperatures converted to a normalized temperature (T_n) according to the equation:

$$T_n = (T - T_s)/(T_o - T_s). \qquad (1)$$

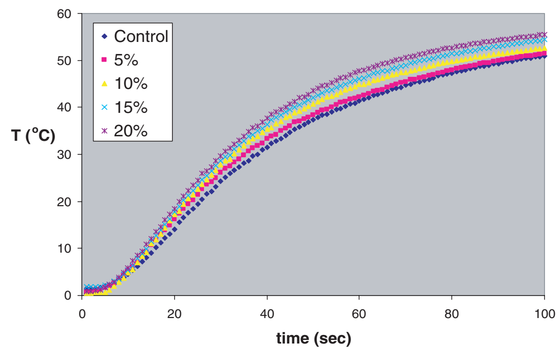

Figure 1 Representative temperature versus time profiles of groups A to E resin specimen cylinders.

As described in a previous study,[19] the slope (S) of a plot of ln T_n versus time was then used (Fig 2) to calculate thermal diffusivity (Δ) according to the following equation:

$$\Delta = -S/\{(5.7832/R^2) + (\pi^2/L^2)\}, \qquad (2)$$

where R and L are the radius and length of the cylinder, respectively. Each specimen (n = 5) was tested three times under identical conditions, with an equilibration at 0°C (30 minutes) between trials. Thermal diffusivity for each specimen was taken as the mean of three trials.

Statistical analysis

Flexural strength

Descriptive statistics, including mean, standard deviation, and minimum and maximum strength values were calculated for each of the experimental groups. One-way ANOVA was used to determine whether significant differences existed between the means of the various experimental groups. Tukey test was employed at the chosen level of probability ($p < 0.05$) to determine if the means were significantly different from each other. Weibull analysis was used to calculate the Weibull modulus,

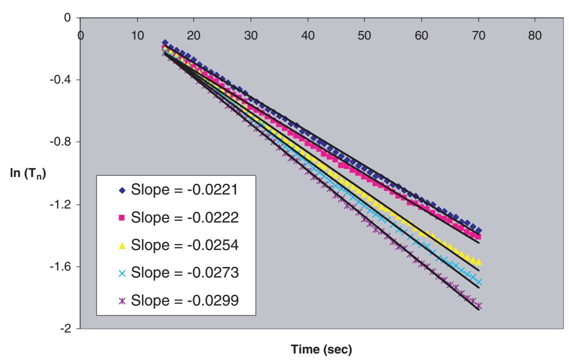

Figure 2 Representative plots of normalized temperature (T_n) versus time for groups A to E acrylic resin specimen cylinders. Linear least-squares fits for each trial are shown, slopes of which were used in equation 2 to calculate thermal diffusivity.

Table 1 Mean flexural bond strength (FS) for the groups tested (in MPa)

Group/Alumina% (weight)	FS	SD	Minimum	Maximum
A (control)	99.45[a]	8.21	88.76	113.28
B (5%)	119.92[b]	6.88	108.07	128.91
C (10%)	121.19[b]	4.34	114.58	126.30
D (15%)	130.08[b]	10.02	117.19	152.34
E (20%)	127.60[b]	9.00	114.58	140.63

*Groups with same superscript letter were statistically similar ($p > 0.05$).

Table 3 Mean thermal diffusivity (m^2/sec) values for the groups tested

Group/Alumina% (weight)	Mean	SD
A (control)	6.8[a]	3.3
B (5%)	7.2[b]	1.5
C (10%)	8.0[c]	2.7
D (15%)	8.5[d]	1.7
E (20%)	9.3[e]	6.6

*Groups with same superscript letter were statistically similar ($p > 0.05$).

characteristic strength, and the required stress for 5% and 95% probabilities of failure.

Thermal diffusivity

Descriptive statistics, including mean, standard deviation, and minimum and maximum thermal diffusivity values were calculated for each of the experimental groups. One-way ANOVA followed by post hoc Tukey test was used to assess statistical differences in Δ among the experimental composites and the control.

Results

The mean flexural strength, standard deviation, and minimum and maximum stress values for each experimental group are presented in Table 1. One-way ANOVA revealed a statistically significant difference between the mean values ($p < 0.001$). Tukey test showed that there was a statistically significant difference ($p < 0.05$) between the tested groups and the control group. There was no significant difference ($p > 0.05$) between the tested groups.

Results from the Weibull analysis are displayed in Table 2. The Weibull modulus ranged from 11.82 for group A (control) to 26.95 for group C (10% alumina). The stress levels required for 5% and 95% probabilities of failure are shown for each group (Table 2).

Flexural strength results of the present study revealed that 5% alumina addition (group B) was responsible for a 20.5% increase in the flexural strength (Table 1). Alumina addition (15% by weight) was responsible for a 30% increase in flexural strength of groups D and E in comparison to the unreinforced control.

The mean thermal diffusivity and standard deviations for each experimental group are tabulated in Table 3. Thermal diffusivity results revealed that 5% alumina addition was responsible for a 5% increase in thermal diffusivity of group B. Alumina addition from 15% to 20% (by weight) was responsible for a 25% and 30% increase in thermal diffusivity of groups D and E in comparison to the unreinforced control.

Table 2 Weibull analysis of flexural bond strength (in MPa)

Group	Weibull Modulus	Strength for 5% Probability of Failure	Strength for 95% Probability of Failure
A	11.82	80.44	113.47
B	17.01	103.63	131.62
C	26.95	110.46	128.45
D	12.44	106.32	147.43
E	13.72	107.93	145.17

The results of the one-way ANOVA revealed a statistically significant ($p < 0.001$) difference between the mean values of the tested groups. Tukey test showed that there was a statistically significant difference ($p < 0.05$) in thermal diffusivity between group A (control group) and the remaining reinforced groups.

Discussion

The main objectives of the current study are closely related to each other and generally affect the durability of heat-polymerized denture base materials. To improve the thermal properties of a new denture base, it is important not to affect its mechanical properties. Zappini et al[21] noted that most studies evaluating the strength of denture base resins rely mainly on impact data, and this may not be the best test to predict clinical function. Impact tests are influenced by loading conditions and specimen geometry, such as the dimensions of the specimen and the presence and configuration of notches. The authors also suggested that a more reasonable test would be a fatigue test; however, the time and number of specimens required for fatigue testing and the fracture toughness test might be more practical. In the current study, flexural strength was tested to get an understanding of how denture base resins hold up under function. The material selected for the current study (diamond) was reported by its manufacturer to have high impact strength. Addition of alumina (group D) significantly improved ($p < 0.05$) the flexural strength of the polymer tested, and this may be attributed to the proper distribution and bonding of the filler within the matrix.

Thermal diffusivity, Δ, is a material property related to thermal conductivity (k) which measures the rate at which a body with nonuniform temperature reaches equilibrium. It is already known that there is a close relation between thermal conductivity and diffusivity. Thermal conductivity measurements are taken under steady state temperature conditions and therefore may not accurately reflect the ability of a material to respond to transient temperature changes (namely, thermal diffusivity), such as those present in the oral cavity during food and liquid intake. Thermal diffusivity of denture bases affects the ability of these materials to transmit thermal changes from the oral cavity to the underlying tissues of the palate. These changes may affect patients' general satisfaction with dentures. Thermal diffusivity of the denture base can be defined as a measure of transient heat flow. Therefore, it is the most relevant parameter when considering the patient's experience with intraoral temperature changes. In the literature, few attempts have been made to develop acrylic resins that possess increased heat transfer characteristics.

Metallic powders have added to conventional denture base resins by various investigators.[14] Addition of silver, aluminum, or copper powder increased the thermal conductivity by as much as 4.5 times that of unmodified acrylic resin; however, the volume fraction of metal filler required to achieve that increase (25% metal by volume) significantly decreased the tensile strength of the acrylic resin polymer by as much as 35%. Explanations given for this reduction in strength included a decrease in the cross-section of load-bearing polymer matrix, stress concentration because of filler particles, change in the modulus of elasticity of resin and mode of crack propagation through it because of fillers, void formation from entrapped air and moisture, and incomplete wetting of the fillers by resin. Many attempts, however, to strengthen acrylic resin in this way failed because stress concentrations occur around embedded materials, and the net effect of embedding fibers or metals is actually to weaken the polymer. This is often due to poor adhesion between the acrylic resin matrix and the fiber metal inserts.

As alternatives to metal powder fillers, thermally conducting ceramics may be useful for increasing the thermal diffusivity while preserving many of the advantageous qualities of acrylic resins. Recent advances in the processing of ceramics have made thermally conducting ceramics, such as sapphire (single crystal form of Al_2O_3), silicon nitride (Si_3N_4), boron nitride (BN), and aluminum nitride (AlN) widely available. Interestingly, many of these ceramics have thermal conductivities approaching or even exceeding that of some metals. For example, the thermal conductivity of sapphire (42 W/min/°K at 20°C) approaches that of cobalt (Co) (100 W/min/°K) and chromium (Cr) (94 W/min/°K), two metals commonly used in denture base alloys.[1] In the current study, it was interesting to find that the addition of Al_2O_3 powder not only improved the flexural strength, but also the thermal diffusivity of the denture base.

The reason for the use of ceramic filler as opposed to metal filler is the lower filler density [the density of sapphire (3.99 g/cm^3) is considerably less than that of Co (8.9) and Cr (7.1)], thus the light weight of acrylic resin denture bases is retained. Furthermore, these ceramic powders have the advantage of being white, and therefore are less likely to alter the finished appearance of the denture base material than are metal powders. The results of the current study are consistent with those of Messersmith et al,[22] who reported that thermal diffusivity of denture base acrylic resin was increased by the addition of thermally conducting sapphire whiskers; however, they did not measure the mechanical properties.

In the current study, the method reported by Watts and Smith[19] was used with slight modification by capturing the data through a personal computer. This modified method even allowed the study of thermal diffusion at different times. Theoretical and experimental data exist to suggest that filler particle shape plays an important role in determining the thermal conductivity of a polymer composite.[9,10]

Specifically, it has been shown that elongated (high aspect ratio) particles of thermoconducting filler embedded in a polymer matrix are more efficient at increasing thermal conductivity than equiaxed (low aspect ratio) particles, such as spheres. The reason for this behavior is believed to be due to the ability of high aspect ratio particles to form continuous pathways for the conduction of heat through the insulating matrix;[9] however, in the current study, using sphere particles of alumina improved both thermal diffusivity and flexural properties significantly. The reason for these results may be attributed to proper distribution of alumina spheres within denture base powders.

Flaws and microcracks may develop during the processing of denture bases. The influence of these flaws and defects on strength measurement can cause large variation in strength data. Weibull modulus is used to describe the variation in the strength results. The lower the value of Weibull modulus, the greater the variability of the strength data, which in turn points to more flaws and defects of the material and unreliability.[23] Weibull modulus values are also affected by the method adopted to finish the specimens and test environment because of possible influence on residual flaw sizes and subcritical crack growth. During processing of the specimens, flaws might have developed. In this study, group C showed a higher Weibull modulus than those of groups B, D, and E. As the mean strength values of these groups were not significantly affected, this indicated that only a few specimens among those tested were affected.

Further research is needed to quantify the filler distribution in the polymer matrix. It may be suggested that the alumina powders were well distributed within the acrylic resin matrix. The fine nature and even distribution of these powders as was clear during processing possibly form a continuous pathway through the insulating polymer matrix. Thus, it is likely that the high aspect ratio of the alumina powders facilitated the formation of a pathway for heat conduction through the acrylic resins to result in increased thermal diffusivity. Further research is also needed to evaluate the effect of aging on these new reinforced denture base materials before clinical application.

Conclusions

From the results of this study, it could be concluded that incorporating Al_2O_3 powder from 5% to 20% by weight into conventional heat-polymerized denture base resin results in an increase in both flexural strength and thermal diffusivity. Increasing the flexural strength and thermal diffusivity of the acrylic resin base material could lead to more patient satisfaction.

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