Print ISSN: 2321-6379
Online ISSN: 2321-595X

Original Article

Comparison of Thermal Conductivity, Flexural Strength, and Surface Hardness of Alumina Incorporated and Conventional Heat-Activated Denture Base Resins

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Abstract

Introduction: Commonly used polymethyl methacrylate (PMMA) denture base material cannot be considered as ideal due to inferior thermal and mechanical properties.

Aim: The aim of the study was to evaluate and compare the thermal conductivity, flexural strength, and surface hardness of heat cure acrylic resin incorporated with 10 wt.% and 15 wt.% alumina and conventional denture base resin.

Materials and Methods: A total of 108 specimens were prepared. Specimens were divided into three main groups. Group A specimens were disk shaped (50 mm × 5 mm) and used for measuring thermal conductivity. Groups B and C specimens were rectangular shaped (65 mm × 10 mm × 3 mm) and were used for measuring flexural strength and surface hardness, respectively. Each group was further divided into three subgroups (1, 2, and 3) depending on the concentration, namely, PMMA without filler (control), PMMA + 10 wt.% of Al2O3, and PMMA + 15 wt.% of Al_2O_3 containing 12 samples each. Thermal conductivity was measured using a modified guarded hot plate apparatus. Flexural strength was assessed with a three-point bending test using a universal testing machine. Hardness testing was conducted using a Vickers Hardness Tester. The results were analyzed using one-way ANOVA followed by *post hoc* comparison by Tukey's method.

Results: Mean values of thermal conductivity were (in W/mK) 0.190, 0.231, and 0.275 for subgroups A1, A2, and A3, respectively. The mean flexural strength values were (in MPa) 56.62, 66.73, and 74.24 for subgroups B1, B2, and B3, respectively. Mean values of surface hardness was calculated to be (in HV) 15.17, 16.51, and 17.91 for subgroup C1, C2, and C3, respectively. There was statistically significant improvement in thermal conductivity, flexural strength, and surface hardness after incorporation of alumina and the increase was in proportion to the weight percentage of alumina filler.

Conclusion: Incorporation of alumina into heat cure denture base resin significantly improved the thermal conductivity, flexural strength, and surface hardness.

Key words: Aluminum oxide, Flexural strength, Polymethyl methacrylate denture base resin, Surface hardness, Thermal conductivity

INTRODUCTION

Loss of teeth is a matter of great concern to the majority of people. Although dental implants are increasingly used in the treatment of edentulous patients, in many cases, a conventional complete denture is still the treatment of choice due to medical or financial reasons.[1] An ideal denture base material for complete denture fabrication should have adequate mechanical and physical properties, besides biocompatibility, and esthetics.[2,3] Polymethyl methacrylate (PMMA) denture base material which has been introduced in 1937 by Dr. Walter Wright is considered to be the most popular denture base material till date.[4] It is mainly due to its advantages such as favorable working characteristics, acceptable physical and esthetic properties,

Access this article online

www.ijss-sn.com

Month of Submission : 03-2020
Month of Peer Review : 04-2020
Month of Acceptance : 05-2020
Month of Publishing : 05-2020

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ease of fabrication, and cost effectiveness.[5] However, it has certain limitations including low thermal conductivity, high thermal expansion coefficient, low elastic modulus, low impact strength, and low fatigue resistance.[6]

The thermal conductivity of PMMA is approximately 0.2 W/mK.[7] This rate is almost one-third of the coefficient of thermal conductivity of most metals. From the patient's perspective, the main problem with such a low thermal conductivity is their inability to sense transient temperature changes in the oral cavity. Lack of thermal conduction to underlying mucosa can also lead to reduction of its thickness and health.[8] With metal denture base, parotid secretion seems to increase as there is increase in the temperature of palatal soft tissues.[9] However, metal denture bases have some disadvantages including increased weight, difficulty with tissue replacement in cases where substantial loss of bone has occurred, difficulty in restoring denture borders within physiologic limits, difficulty with the relining process, esthetics, and high cost.[10] Because of these disadvantages, development of acrylic-based materials with improved thermal conductivity has always been a goal.

In addition, acrylic denture base materials have poor strength including low impact and fatigue resistance.[11] Fracture of acrylic denture bases primarily occurs due to impact or fatigue failure. Fatigue failure is caused by repeated flexure over a period of time.[12] Another property that can influence the surface characteristics of acrylic resins is the hardness, which indicates the ease of trimming and finishing of a material and its resistance to in service scratching during cleaning procedures.[13] Since patients practice a wide variety of methods for cleaning dentures other than recommended by the dentist it is essential to have a high surface hardness for denture base material to reduce the rate of denture abrasion.

Efforts to improve the physical and mechanical properties of PMMA have been made by different methods. These include chemical modification of PMMA and reinforcing with other materials. Addition of fillers and fibers is a commonly used method to improve its properties. These additives include Fibers (glass fiber, polyamide fiber, polyethylene and polypropylene fibers, and natural fibers), Fillers (Metal oxides: Alumina [Al_2O_3], zirconia [ZrO_2], titanium dioxide [TiO_2]; Noble metals: Silver [Ag], nanogold [Au], platinum [Pt], palladium [Pd]; Minerals: Hydroxyapatite fillers, silicon dioxide [SiO_2], silica-based filler; and Carbon family fillers: Nanocarbon, nanodiamonds), and Hybrid fiber reinforcement.[14]

As alternatives to metal powder fillers, thermally conducting ceramics may be useful for increasing the thermal conductivity while preserving many of the advantageous qualities of acrylic resins. Recent advances in the processing of ceramics have led to the development of thermally conducting ceramics, such as sapphire (single crystal form of Al_2O_3), silicon nitride (Si_3N_4), boron nitride, and aluminum nitride.

Aluminum oxide (Al_2O_3) commonly referred to as alumina possesses strong interatomic bonding, giving rise to its desirable material characteristics. Its high hardness, excellent dielectric properties, refractoriness, and good thermal properties make it the material of choice for a wide range of applications.[15] 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 compared to the metal powder incorporated denture base.[5] Thus, alumina possesses various favorable properties which may improve the physical and mechanical properties of PMMA.

In this context, the present study was conducted to evaluate and compare the thermal conductivity, flexural strength, and surface hardness of heat cure acrylic resin incorporated with 10 wt.% and 15 wt.% aluminum oxide microparticles and conventional denture base resin.

MATERIALS AND METHODS

Incorporation of Al_2O_3 Particles into PMMA Heat Cure Resin

Al_2O_3 particles (nanoshel, purity – 99.9%, and average particle size – 50–60 μm) were incorporated into the polymer of heat cure acrylic resin (DPI, The Bombay Burmah Trading Corporation Ltd., Mumbai) at two different concentrations, namely, 10% and 15% by weight. Appropriate amount of Al_2O_3 and acrylic resin polymer was weighed using a digital weighing balance and mixed together using a mortar and pestle. To ensure uniform distribution of Al_2O_3 in the polymer of heat cure acrylic resin "geometric dilution" method was employed for trituration.

Fabrication of Test Specimens

The properties evaluated in this study were thermal conductivity, flexural strength, and surface hardness. Thermal conductivity testing required disk shaped specimens measuring 50 mm diameter and 5 mm thickness. For testing flexural strength and surface hardness, rectangular blocks measuring 65 mm length 10 mm width and 3 mm thickness were required. Plexiglass molds of the above-mentioned dimensions were fabricated with high precision laser cutting machine. Wax patterns were prepared from plexiglass molds and were invested in denture flask in the conventional manner. The monomer and polymer

of the heat-polymerized acrylic resin were proportioned, mixed, packed, and pressed into the mold following manufacturer's instructions and processed. Dimensions of specimens were measured by a digital Vernier caliper with a measuring accuracy of ±0.1 mm. All specimens were stored in thermostatically controlled water bath at 37±1°C for 7 days, before testing.

Distribution of Specimens [Table 1]

Thermal conductivity testing

Thermal conductivity was tested using a modified guarded hot plate apparatus. The solid disk shaped specimens were placed between the two plates of the apparatus. A thin film of heat flux sensors was positioned on either side of the sample for the measurement of heat transmitted through the sample [Figure 1]. The thin film sensor had in built type T thermocouples. Both cold and hot plate were instrumented using type K thermocouples at the interface between the sample and the plates for the measurement of temperature on the surface of the sample. The hot plate was heated using a known power and the cold plate was cooled to a constant temperature using a recirculating chiller. The temperature on both sides of the sample was monitored using type K and type T thermocouples until the entire system reaches a steady state. The power to the hot plate was selected so as to create temperature gradient of 5–10°C across the thickness of the sample which in turn was ensured by the temperature measurement.

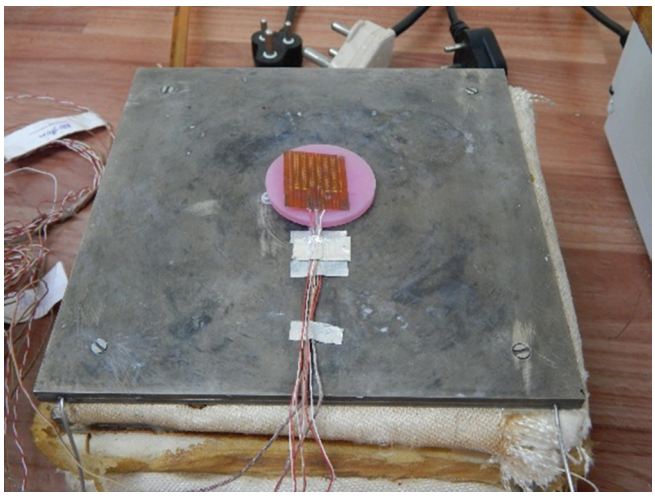


Figure 1: Sample with instrumentation

The steady state temperatures, the thickness of the sample and the heat transfer rate (measured using thin film heat flux sensors) were used to calculate thermal conductivity as follows.

$$k = q\frac{dx}{dT}$$

k = Thermal conductivity, W/m-K.
q = Heat flux, W/m^2.
dx = Thickness of the sample, m.
$dT = T_{cold} - T_{hot}$ = Temperature gradient across the sample, °C.

Flexural Strength Testing

The flexural strengths of the specimens were evaluated according to the ISO 1567, 1999, for denture base resins, by three-point bending test using universal testing machine (INSTRON). The rectangular specimens were inserted in relative points on the testing machine such that the span length was 50 mm. 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, engages the center of the upper surface of the specimen [Figure 2]. Specimens were loaded until fracture occurred.

Flexural strength was calculated using the following equation.

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

S: Flexural strength.
P: load at fracture.
I: distance between supporting wedges.
b: width of specimen.
d: thickness of specimen.

Table 1: Distribution of specimens

Group	Measured property	Shape of specimens	Subgroup	Description	Number of specimens
Group A	Thermal conductivity	Disc (50×5)	A1	PMMA (control)	12
			A2	PMMA + 10 wt.% AL_2O_3	12
			A3	PMMA + 15 wt.% AL_2O_3	12
Group B	Flexural strength	Rectangular (65×10×3)	B1	PMMA (Control)	12
			B2	PMMA + 10 wt.% AL_2O_3	12
			B3	PMMA + 15 wt.% AL_2O_3	12
Group C	Surface hardness	Rectangular (65×10×3)	C1	PMMA (Control)	12
			C2	PMMA + 10 wt.% AL_2O_3	12
			C3	PMMA + 15 wt.% AL_2O_3	12
Total					108

PMMA: Polymethyl methacrylate

Surface Hardness Testing

A Vickers Hardness Tester (HMV SCHIMADZO, model – HMV 2T ADW) was used to evaluate the surface hardness. To determine Vickers values, a load of 25 gram-force (gf) was applied for 15 s to specimens using a Vickers Hardness Tester [Figure 3]. Each specimen was subjected to three indentations (one at the center and two at the border), and the average hardness (HV) value was calculated.

RESULTS

Data were analyzed using computer software, Statistical Package for the Social Sciences (SPSS) version 16.0. Data were expressed in its mean and standard deviation. Analysis of variance (one-way ANOVA) was performed as parametric test to compare different groups. To facilitate multiple comparisons between groups, Tukey's method was employed as a *post hoc* test along with ANOVA. For all statistical evaluation, a two-tailed probability of value, <0.05 was considered significant.

Graphical representation of the mean values of thermal conductivity, flexural strength, and surface hardness is shown in Graphs 1-3, respectively.

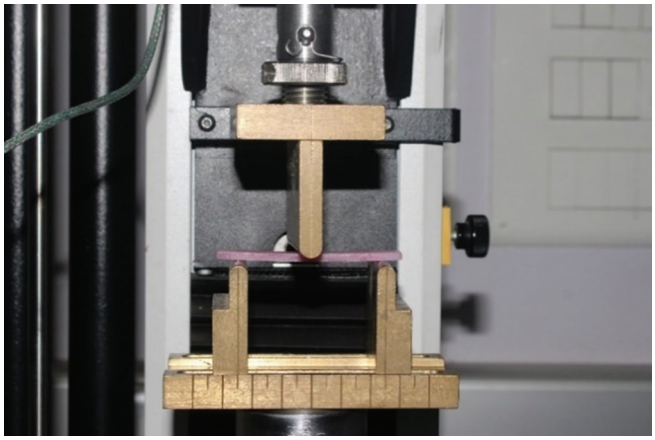


Figure 2: Specimen being tested for flexural strength

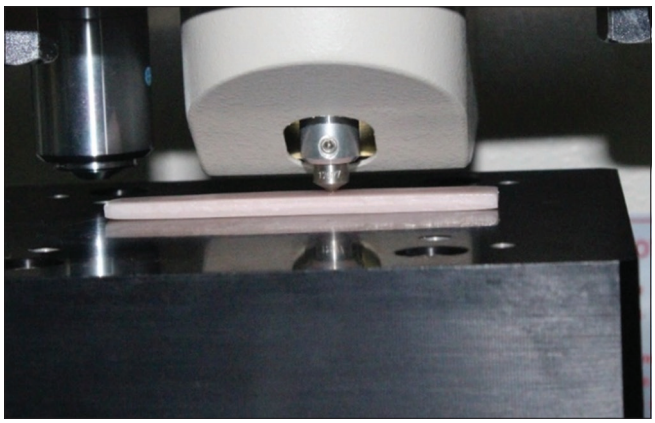
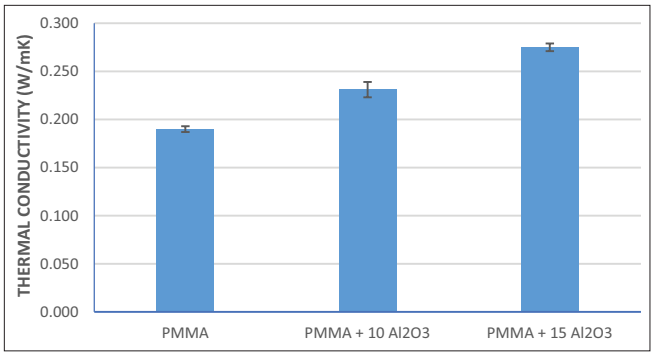


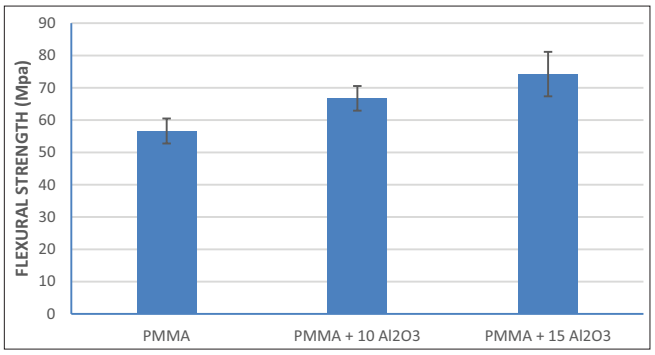
Figure 3: Specimen being tested for surface hardness

Mean and standard deviation values of the thermal conductivity of subgroup A1, A2, and A3 are presented in Table 2. As per this study, maximum mean thermal conductivity was given by PMMA modified by adding 15 wt.% alumina. One-way ANOVA showed a statistically significant difference between mean values [Table 3]. Multiple comparisons with *post hoc* by Tukey's method revealed that comparison between subgroups (A1 and A2), (A1 and A3), and (A2 and A3) were statistically significant as $P < 0.001$ [Table 4].

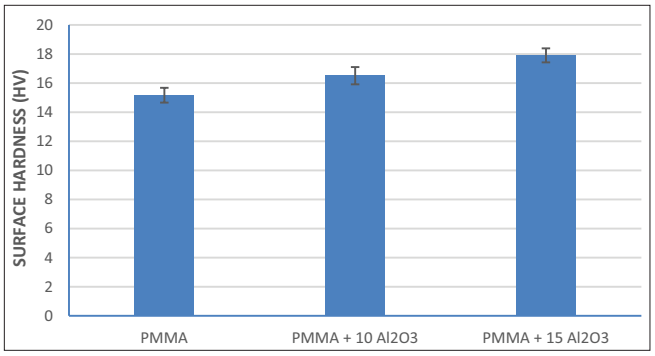
Mean and standard deviation values of flexural strength of subgroup B1, B2, and B3 are presented in Table 5. Maximum flexural strength values were given by PMMA modified by adding 15 wt.% alumina and the least values were given by unmodified PMMA. One-way ANOVA showed a statistically



Graph 1: Comparison of mean values of thermal conductivity



Graph 2: Comparison of mean values of flexural strength



Graph 3: Comparison of mean values of surface hardness

Table 2: Mean and standard deviation of the thermal conductivity of three subgroups

Subgroup	*n*	Thermal conductivity (W/mK)	
		Mean	Standard deviation
A1	12	0.190	0.003
A2	12	0.231	0.008
A3	12	0.275	0.004
Total	36	0.232	0.036

Table 3: Comparison of the thermal conductivity among three subgroups using one-way ANOVA

ANOVA	Sum of squares	Degrees of freedom	Mean Square	F	*P*
Between groups	0.043	2	0.022	776.305	<0.001
Within groups	0.001	33	0		
Total	0.044	35			

Table 4: Inter group comparison of thermal conductivity among the three subgroups using Tukey *post hoc* test

Inter group comparison	Mean difference	Standard error	*P*
A1 versus A2	.0414	0.002	<0.001
A1 versus A3	.0850	0.002	<0.001
A2 versus A3	.0435	0.002	<0.001

Table 5: Mean and standard deviation of flexural strength of three subgroups

Subgroup	*n*	Flexural strength (MPa)	
		Mean	Standard deviation
B1	12	56.62	3.86
B2	12	66.73	3.81
B3	12	74.24	6.88
Total	36	65.86	8.82

Table 6: Comparison of flexural strength among three subgroups using one-way ANOVA

ANOVA	Sum of squares	Degrees of freedom	Mean square	F	*P*
Between groups	1877.277	2	938.639	36.703	<0.001
Within groups	843.928	33	25.574		
Total	2721.206	35			

Table 7: Inter group comparison of flexural strength among the three subgroups using Tukey *post hoc* test

Inter group comparison	Mean difference	Standard error	*P*
B1 versus B2	10.1083	2.0645	<0.001
B1 versus B3	17.6250	2.0645	<0.001
B2 versus B3	7.5167	2.0645	0.003

Table 8: Mean and standard deviation of the surface hardness of three subgroups

Subgroup	*n*	Surface hardness (HV)	
		Mean	Standard deviation
C1	12	15.17	0.51
C2	12	16.51	0.59
C3	12	17.91	0.48
Total	36	16.53	1.25

Table 9: Comparison of surface hardness among three subgroups using one-way ANOVA

ANOVA	Sum of squares	Degrees of freedom	Mean square	F	*P*
Between Groups	45.107	2	22.554	80.522	<0.001
Within Groups	9.243	33	0.28		
Total	54.35	35			

significant difference between mean values [Table 6]. *Post hoc* comparison of flexural strength values between the subgroups B1, B2, and B3 was performed using Tukey's method [Table 7]. The comparison between B1 and B2 showed a mean difference of 10.1083 and that change is statistically significant with $P < 0.001$. The comparison between B1 and B3 showed a mean difference of 17.6250 which is also statistically significant with $P < 0.001$. The comparison between B2 and B3 showed a mean difference of 7.5167 and that change is also statistically significant with $P = 0.003$.

Mean and standard deviation values of surface hardness of subgroup C1, C2, and C3 are given in Table 8. Maximum values of surface hardness were given by PMMA modified by adding 15 wt.% alumina. One-way ANOVA showed a statistically significant difference between mean values [Table 9]. Multiple comparisons with *post hoc* by Tukey's method revealed that comparison between subgroups (C1 and C2), (C1 and C3), and (C2 and C3) was statistically significant as $P < 0.001$ [Table 10].

DISCUSSION

PMMA has been the material of choice for the fabrication of dentures since 1930s because of its advantages such as favorable working characteristics, acceptable physical and esthetic properties, ease of fabrication, and cost effectiveness.[5] However, it cannot be considered as an ideal denture base material due to its inferior thermal and mechanical properties.

The thermal conductivity of PMMA is approximately 3 times lower than that of metals.[15] Various methods have been considered so far for increasing the thermal conductivity of denture base to provide the maximum

Table 10: Inter group comparison of surface hardness among the three subgroups using Tukey *post hoc* test

Inter group comparison	Mean difference	Standard error	*P*
C1 versus C2	1.34167	0.216	<0.001
C1 versus C3	2.74167	0.216	<0.001
C2 versus C3	1.40000	0.216	<0.001

gustatory response. One method is the replacement of acrylic denture base with metallic base. However, the use of metal as a denture base material has several disadvantages including increased weight of the denture, difficulty with the relining process, esthetics, and high cost.[15] Even though metallic denture bases have better thermal conductivity they are not widely used because of their relatively high cost. Another approach for improving thermal conductivity of denture base resin is to incorporate a material having better thermal conductivity. In addition to improvement in thermal conductivity, they also influence some of the mechanical properties.

Ellakwa *et al.*[15] investigated the effect of adding different proportions of aluminum oxide powder on the flexural strength and thermal diffusivity of heat-polymerized acrylic resin. Thermal diffusivity was found to increase in proportion to the weight percentage of alumina filler while the flexural strength shows significant increase up to incorporation of 15% followed by a reduction with 20%. Messersmith *et al.*[5] reported that thermal diffusivity of denture base resin was increased by the addition of thermally conducting sapphire whiskers at concentrations of 9.35 and 15%.

As alternatives to metal powder fillers, thermally conducting ceramics such as alumina may be useful for increasing the thermal conductivity while preserving many of the advantageous qualities of acrylic resins.[15] Most of the studies showed significant improvement in thermal conductivity with the incorporation of alumina. Along with thermal conductivity, alumina incorporation also resulted in significant improvement of flexural strength. However, the optimum concentration of reinforcement varied among authors.

Aluminum oxide (Al_2O_3), commonly referred to as alumina, possesses strong ionic interatomic bonding resulting in its desirable material characteristics. It can exist in several crystalline phases, which all revert to the most stable hexagonal alpha phase at elevated temperatures. 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] The biocompatibility of aluminum has been shown by Kawahara *et al.*[16] The ceramic filler as opposed to metal filler has lower filler density. Thus, the weight of acrylic resin denture bases does not increase significantly. In addition, the ceramic powder being white is less likely to alter the final appearance of the denture base when compared to the metal powder incorporated denture base.[15]

The present study was conducted by the incorporation of alumina into the denture base resin at two different concentrations that matches with the majority of the previous studies. One of the popular commercially available denture base resins (DPI, The Bombay Burmah Trading Corporation Ltd., Mumbai) was modified by incorporating 10 wt.% and 15 wt.% of alumina microparticles. Geometric dilution was employed for the uniform distribution of alumina in the PMMA polymer. Along with thermal conductivity, flexural strength and surface hardness were also assessed in this study as these two parameters are important for an extended clinical service life of the prosthesis.

As endorsed by other authors, this investigation has also showed that addition of alumina increased the thermal conductivity of PMMA and this increase was proportional to the concentration of filler. PMMA modified with 15wt.% of alumina showed the highest mean thermal conductivity of 0.275±0.004W/mK. These results were in agreement with the studies by Ellakwa *et al.*[15] The improvement in thermal conductivity on addition of alumina can be attributed to the formation of thermally conducting pathways within the polymer matrix. This may be due to overlapping of thermal conductive particles inside the polymer matrix to bridge the insulating effect of PMMA matrix. The increase in the amount of fillers make the particles approximate and overlap each other forming conductive pathways and permit transition of heat from one side of the specimen to its opposite side thus increasing thermal conductivity.[17]

In addition to the increased temperature and taste perception, the increase in thermal conductivity could also minimize the development of porosities in the denture base resin. Since PMMA is a poor thermal conductor, the heat generated in thick segments of the denture base during polymerization cannot be dissipated. When heating is poorly controlled, the peak temperature of this resin can rise well above the boiling point of monomer and can cause boiling of unreacted monomer thereby causing porosity within the processed denture base. With the incorporation of alumina into PMMA, thermal conductivity is improved allowing the temperature to dissipate thereby reducing porosity.

This study also compared the flexural strength of unmodified resin and alumina incorporated resin. High flexural strength is crucial for denture wearing success, as alveolar resorption is a gradual and irregular process that leaves tissue-borne prostheses unevenly supported. Such dentures flex in the mouth around the midline during function and repeated occlusal loadings during mastication lead to the fatigue fracture.[12] There was also a significant improvement in flexural strength on addition of alumina into PMMA resin. This increase in flexural strength was proportional to the concentration of alumina particles. PMMA reinforced with 15 wt.% showed highest mean flexural strength (74.24 MPa).

These results were in agreement with the studies conducted by Ellakwa *et al.*[15] and Saritha *et al.*[18] Improved flexural strength could be attributed to uniform distribution of the filler particles within the matrix and transformation toughening. When sufficient stress develops and micro-cracks begin to propagate, the transformation phenomenon occurs, which depletes the energy for crack propagation. Therefore, proper distribution of the filler within the matrix can stop or deflect cracks.[17] However, certain studies have also proposed a reduction in flexural strength on incorporation of alumina at higher concentrations. Ellakwa *et al.*[15] showed that by increasing the filler loading to 20 wt.% there was substantial reduction in the flexural strength. Vojdani *et al.*[11] found that addition of 5wt% Al_2O_3 decreased mean flexural strength significantly. This reduction in flexural strength on increase in filler concentration could be attributed to decrease in cross section of 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; incomplete wetting of the fillers by the resin; and the fact that alumina acts as an interfering factor in the integrity of the polymer matrix.[19]

The decrease in flexural strength of PMMA with increased filler content could be overcome by treating the filler with coupling agent to ensure better wettability of the filler. Coupling agents provide good interfacial bond between the filler and resin matrix. These may provide an intermediate layer at the filler matrix interface. This layer may change the pattern and reduce magnification of stress.[8] In the present study, an improvement in flexural strength was noted with increasing the filler loading even without the use of silane coupling. This could be attributed to the uniform distribution of filler particles within the resin matrix.

Since aluminum oxide also possesses high hardness due to the high ionic interatomic bonding, the possibility of improvement in surface hardness through the incorporation of alumina was also investigated. There was a significant increase in the surface hardness of alumina incorporated PMMA. This finding is in agreement with the previous investigators, 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 the inherent characteristics of alumina. The most stable hexagonal alpha phase of Al_2O_3 is the strongest and stiffest of the oxide ceramics. Therefore, it is expected when Al_2O_3 particles are dispersed in a matrix, they increase its hardness and strength.[15]

The white color of alumina is not expected to adversely affect the esthetic appearance of denture base resin; however, the translucency was adversely affected as concentration of alumina increased. The decrease in translucency obtained in this study may be attributed to differences in the optical properties of the alumina and acrylic resin and its distributions within the resin matrix. Hence, further studies are required to find out the optimum concentration of alumina that would not adversely affect the optical property of acrylic denture base resin.

Aluminum oxide particles used in this study were microparticles rather than the highly expensive nanoparticles. Furthermore, favorable mechanical and thermal properties have been achieved with untreated alumina particles in this study without additional procedure of silanization. The study also has certain limitations. The results of the study were limited to two concentrations of the alumina (10 and 15 wt.%). Further research is needed to quantify the optimum filler distribution in the polymer matrix. Changes in only three properties (thermal conductivity, flexural strength, and surface hardness) were evaluated in this study. Elaborate research is needed to examine other physical and mechanical properties. The effect of aging on these reinforced denture base materials also needs to be evaluated. As this was an *in vitro* study, a direct correlation to the clinical situation could not be established. Long-term clinical studies and studies based on patient satisfaction also need to be conducted before prescribing alumina incorporated PMMA for denture fabrication.

CONCLUSION

Within the limits of the study, the following conclusions were drawn:

1. Incorporation of alumina significantly improved the thermal conductivity, flexural strength, and surface hardness of heat cure denture base resin
2. Magnitude of increase in these properties was proportional to the concentration of alumina incorporated.

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How to cite this article: Aparna PM, Kumar KH, Ravichandran R, Nair VV, Zeenath H, Manjuran NG. Comparison of Thermal Conductivity, Flexural Strength, and Surface Hardness of Alumina Incorporated and Conventional Heat-Activated Denture Base Resins. Int J Sci Stud 2020;8(2):64-71.

Source of Support: Nil, **Conflicts of Interest:** None declared.