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ORIGINAL ARTICLE

Effect of Addition of Sapphire (Aluminium Oxide) or Silver Fillers on the Flexural Strength Thermal Diffusivity and Water Sorption of Heat Polymerized Acrylic Resins

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ABSTRACT

Purpose: This work was undertaken to investigate the effect of adding sapphire (aluminium oxide) or silver filler particles on the flexural strength, thermal diffusivity and water sorption of polymethylmethacrylate (PMMA) resin.

Materials and methods: A total of 90 samples were fabricated and divided into three groups (n = 30): Group I—PMMA (control), group II—PMMA with sapphire fillers and group III—PMMA with silver fillers. Each group samples were divided into three subgroups (n=10) according to the properties evaluated. Sub group I: Flexural strength samples (rectangular bars of 65 × 10 × 2.5 mm dimensions), sub group II: Thermal diffusivity samples (cylindrical shaped of 9 × 9 mm dimensions) and sub group III: Water sorption samples (disk shaped of dimensions 50 × 0.5 mm). Results were analyzed by one-way ANOVA and Bonferroni correction tests ($p < 0.05$).

Results: The mean flexural strength of group II (116.5 MPa) was significantly higher while that of group III (77.91 MPa) significantly lower than the control group (88.63 MPa). The mean thermal diffusivities of both group II (0.079 mm^2/sec) and III (0.123 mm^2/sec) were found to be significantly higher than the control group (0.062 mm^2/sec). Both group II (0.59 mg/cm^2) and group III (0.53 mg/cm^2) showed significantly less mean water sorption than the control group (0.65 mg/cm^2).

Conclusion: As compared to silver fillers, sapphire fillers are purported to be better fillers for the reinforcement of polymethylmethacrylate resin. This is because they have potential as added components in denture bases to provide increased flexural strength, thermal diffusivity and decreased water sorption.

Keywords: Polymethylmethacrylate (PMMA) resin, Sapphire filler, Silver filler, Flexural strength, Thermal diffusivity, Water sorption.

INTRODUCTION

Acrylic resin and rubber-reinforced acrylic polymers represent approximately 90 to 95% of the denture base materials used in prosthodontics today.[1] PMMA was introduced in 1937 by Dr Walter Wright and Vernon brothers.[2] The main attributes of acrylic resins that have made them so successful as denture bases include their ease of processing, low cost, light weight, and color matching ability. However, acrylic resin denture base materials are typically low in strength, brittle on impact, and low in thermal conductivity.[3,4]

Due to its low strength numerous approaches to strengthen the acrylic resin have been suggested. It has been approached either through chemical modifications to produce graft co-polymers of rubber methacrylate, referred to as high-impact resins[5] or by incorporating various metal forms[6] and several types of fibers, like carbon, glass, polyethylene and aramide to provide mechanical reinforcement to fracture prone areas.[7]

Although often overlooked, the heat transfer characteristics of the denture base resins may be an important factor in determining patient satisfaction.[8,9] Because the process of eating consists of frequent and abrupt changes in the temperature of food, thermal conductivity or rather, the transient thermal conductivity (thermal diffusivity) of the denture base becomes an important factor affecting the gustatory response, e.g. chemical perception of taste, smell, textural perception, and temperature.[10,11]

The thermal conductivity of PMMA is approximately 0.2 W/min/°K, 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 (e.g. gold and chromium cobalt alloys).[12,13] 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.[14] Because of the disadvantages associated with metallic denture base materials, exploring the development of acrylic-based materials with improved thermal diffusivity is of interest.

Water sorption of denture base material can also affect its physiochemical and mechanical properties.[15] PMMA though absorbs relatively moderate amounts of water when placed in an aqueous environment, nevertheless, this water exerts significant effects on the mechanical and dimensional properties of the polymer.[16]

In the literature, very few attempts have been made to develop acrylic resins that possess not only improved mechanical properties but also exhibit an overall improvement in physical properties, like thermal diffusivity (heat transfer characteristic and water sorption, without negatively affecting each other.

The aim of the present study was to know the effect on the physical, mechanical and thermal properties of PMMA denture base material by the incorporation of sapphire (aluminium oxide) fillers and metal fillers (silver). In the present study, both types of fillers were chosen as reinforcers because they are purported to not only influence the strength of the resin but also provide thermally conducting pathways within the insulating acrylic resin matrix. So, an overall enhancement of the physical, thermal as well as mechanical properties of acrylic resin can be expected.

MATERIALS AND METHODS

Commercial acrylic resin denture base powder and liquid (Trevalon powder and liquid, Dentsply India Pvt Ltd) was used to act as a control (with no fillers added). Filler particles: Sapphire and silver (Qualigens India, Ltd) fillers were used in 25% ratios by the weight of acrylic resins.

Incorporation of Fillers

Unmodified acrylic resin control samples were processed according to the manufacturer's specifications. For samples that contained fillers, sapphire (ceramic) and silver fillers were added separately to PMMA polymer and mixed manually by hand in a mortar and pestle. To this uniform mixture of fillers and PMMA powder, monomer was later added.

Distribution of Samples

A total of 90 samples were made and divided into three groups (n=30): Group 1 (G I)—PMMA with no reinforcement, group II (G II)—PMMA with sapphire fillers and Group III (G III)—PMMA with silver fillers. Each group had 30 samples which were further divided into three subgroups (n = 10) on the basis of the type of property which was evaluated. Sub group I (g I)—flexural strength samples (rectangular bars), sub group II (g II)—thermal diffusivity samples (cylinders) and sub group III (g III)—water sorption samples (disks) (Table 1). The dimensions of each sample were measured with a digital calliper with an accuracy of ± 0.1 mm.

Specimen Preparation for Testing of Flexural Strength

Thirty samples of heat-polymerized acrylic resin were fabricated. Rectangular strips with dimensions 65 × 10 × 2.5 mm were fabricated in accordance with ADA specification No. 12 for denture base polymers.[17] A two piece custom-made brass metal mold with three rectangular grooves of size (65 × 10 × 2.5 mm) was made for preparing acrylic bar specimens (Fig. 1).

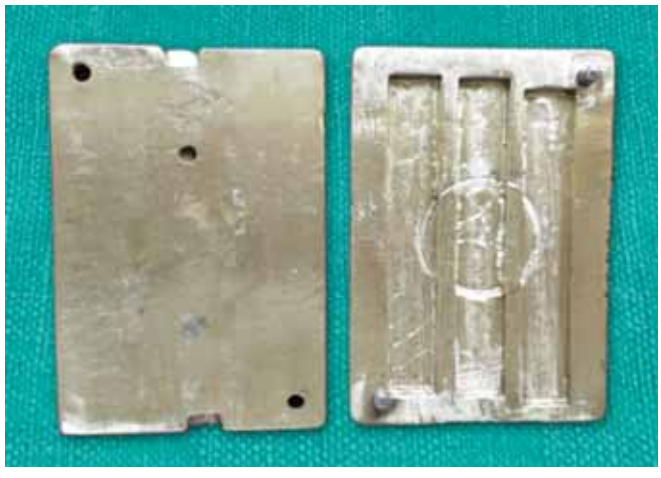

Fig. 1: Brass mold for flexural strength samples

Flexural Strength Testing

The flexural strengths of the samples were determined using a three-point bending testing device in a universal testing machine (Zwick Z010) (Fig. 2). The device consisted of a loading wedge and a pair of adjustable supporting wedges placed 50 mm apart. The specimens were centerd on the device in such a way that

Table 1: Distribution of samples

Groups	*Sub-groups*			*Total samples*
	Flexural strength (g I)	*Thermal diffusivity (g II)*	*Disks (g III)*	
Group I (G I) *PMMA with no reinforcement*	10	10	10	30
Group II (G II) *PMMA with sapphire fillers*	10	10	10	30
Group III (G III) *PMMA with silver fillers*	10	10	10	30
Total samples	30	30	30	90

PMMA: Polymethylmethacrylate

Fig. 2: Sample testing on Zwick-Z010 Universal testing machine

the loading wedge, set to travel at a crosshead speed of 5 mm/min, engaged the center of the upper surface of the samples. Specimens were loaded until fracture occurred. Flexural strength was calculated using the following equation: $S = 3PI/2bd^2$, where S = flexural strength (N/mm^2), P = load at fracture (N), I = distance between the supporting wedges (mm), b = width of the sample (mm) and d = thickness of the sample (mm).

Specimen Preparation for Testing of Thermal Diffusivity

Solid metal cylinders (9 mm diameter × 9 mm length) were invested in brass flasks using dental stone. After hardening of the stone, the metal cylinders were removed which left cylindrical cavities used for molding the samples. The type K thermocouple wire (0.127 mm diameter) with bead junction was inserted into the cylindrical samples at the dough stage, i.e. before curing (Figs 3A to C). Before incorporation, the thermocouple wire was calibrated in order to standardize the thermocouple to get accurate temperature reading. All samples were conditioned at room temperature in distilled water for 24 hours before measurement. The temperature range of 0 to 70ºC was chosen in this study to correspond to the temperatures of food and drink likely to be ingested in a typical meal.

Thermal Diffusivity Testing

Thermal diffusivity was measured by using a technique developed by Watts and Smith[18] to characterize the thermal properties of dental materials. Sample with thermocouple leads were attached to a personal computer recorder for the measurement of transient temperature changes at the center of

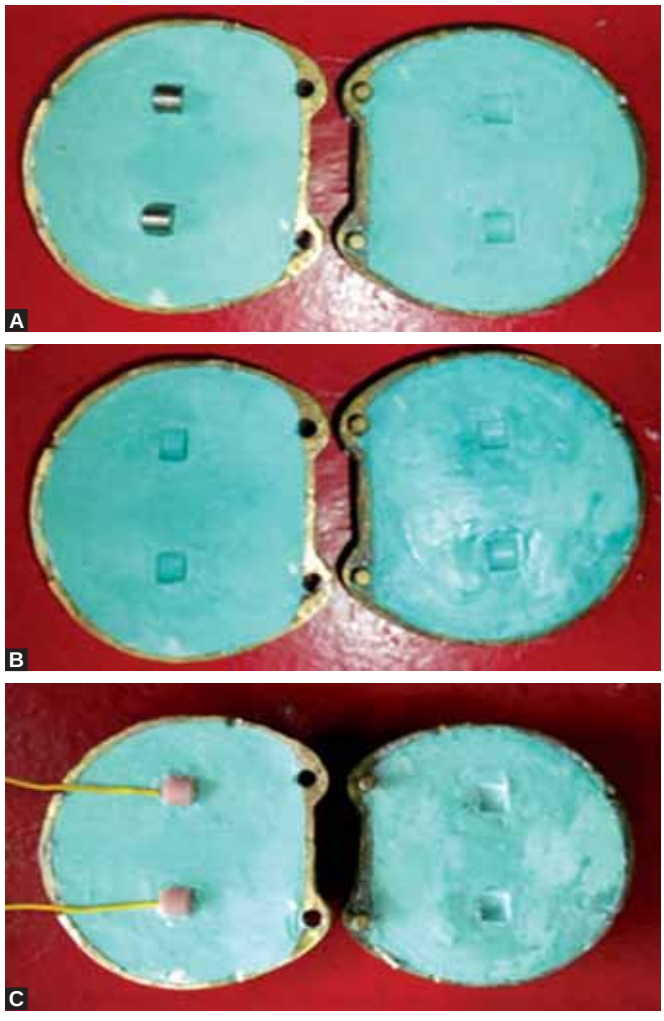

Fig. 3A to C: Cylindrical stone molds and incorporation of thermocouple wire: (A) Investing of solid metal cylinders, (B) cylindrical cavities in dental stone, (C) incorporation of thermocouple wire and packing of resin

the cylindrical samples. During testing, initially the samples were conditioned in distilled water for 24 hours at room temperature and were equilibrated in a ice bath at 0 ± 1ºC (T_o) for 30 minutes. They were then immersed in a thermostated water bath at 70 ± 1ºC (T_s). This was done in order to note the transient temperature changes within the specimen from 0 to 70ºC by using the thermal diffusivity set-up. The components of this set-up were: thermostated water bath, thermocouple module, temperature control module, DC voltage stabilizer and a personal computer (Fig. 4).

Recording of Data

Using the data acquisition software the temperature at the center of the specimen (T) was recorded each second for upto 10 minutes. The resolution of all temperature measurements was 0.1ºC. Temperature versus time data obtained was plotted graphically. These recorded temperatures were then converted to normalized temperatures (T_n) according to the equation:

$$T_n = (T - T_s)/(T_o - T_s)$$

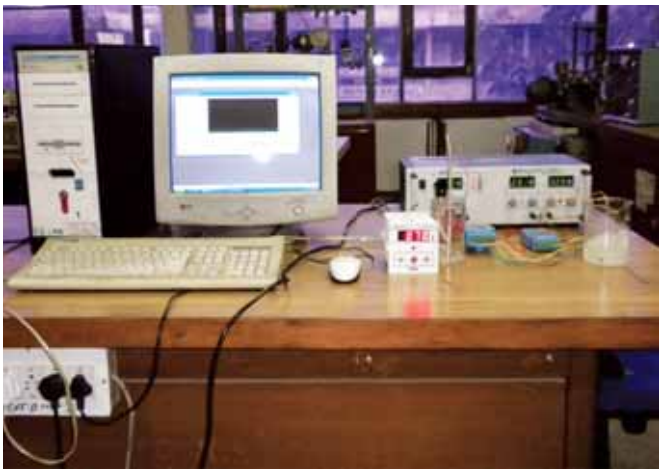

Fig. 4: Thermal diffusivity set-up

As described in a previous study,[18] the slope (S) of a plot of $\log_e T_n$ (i.e. $l_n T_n$) versus time was then used to calculate thermal diffusivity according to the following equation: Thermal diffusivity = $-S/\{(5.7832/R^2) + (\pi^2/L^2)\}$, where R and L were the radius and length of the cylinder respectively.

Specimen Preparation for Testing of Water Sorption

Each disk was prepared with a diameter of 50 mm and thickness of 0.5 mm according to ADA Specification no. 12 for denture base polymers.[17] A two piece custom made brass metal mold with a disk shaped slot of dimension (50 × 0.5 mm) was used for preparing the acrylic disk specimens (Fig. 5).

Water Sorption Testing

All disk shaped test specimens were dried in a desiccator containing dry silica gel at 37 ± 2°C for 24 hours, removed to a similar desiccator at room temperature for one hour, and then weighed with a precision of 0.1 mg in a weighing balance .This cycle was repeated until the weight loss of each disk was not more than 0.5 mg in a 24-hour period. The disks were immersed in distilled water at 37 ± 1°C for seven days, after which the disks were removed from water, wiped with a clean, dry hand towel until free from visible moisture, waved in air for 15 seconds and weighed one minute after removal from water. Water sorption for each disk was calculated using the formula:

Sorption (mg/cm^2) = [mass after immersion (mg) – conditioned mass (mg)]/surface area.

Statistical Analysis

Descriptive statistics was used to analyze the data. One-way ANOVA was used to analyze the significance of results within the control group and the experimental groups. Bonferroni post hoc was used for comparison of values among different groups.

RESULTS

Flexural Strength

The highest mean flexural strength was found in group II (116.5 ± 7.067 MPa) and the lowest mean flexural strength was found in group III (77.91 ± 3.784 MPa), which was lower than the control specimens (group I) (88.63 ± 5.217 MPa) tested. Intergroup comparison showed a significant ($p < 0.0001$) increase in flexural strength on addition of sapphire (Al_2O_3) fillers to PMMA resin as compared to the control group, whereas a significant ($p < 0.001$) decrease on addition of silver fillers to PMMA resin was noted (Table 2 and Fig. 6).

Thermal Diffusivity

The highest mean thermal diffusivity was found in group II (0.123 ± 0.014 mm^2/sec) followed by group III (0.079 ± 0.006 mm^2/sec) and least thermal diffusivity were found in the control specimens (0.062 ± 0.012 mm^2/sec). Therefore, the maximum thermal diffusivity was seen after the addition of sapphire fillers to PMMA resin and minimum values were seen in the control group, i.e. group I. Intergroup comparisons showed statistically significant differences for group I with II ($p < 0.0001$), group II with III ($p < 0.0001$) and group I with III ($p < 0.001$) (Table 2 and Fig. 7).

Fig. 5: Brass mold for water sorption samples

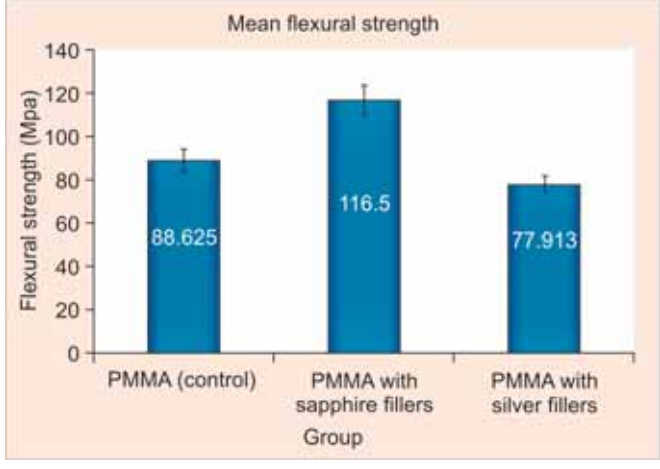

Fig. 6: Mean and standard deviation values of flexural strength of the three groups

Table 2: Intergroup comparison of flexural strength, thermal diffusivity and water sorption for the three groups

Sub groups	*Mean ± standard deviation*			*p-value*		
	Group I PMMA (control)	*Group II PMMA with sapphire fillers*	*Group III PMMA with silver fillers*	*Group I vs II*	*Group I vs III*	*Group II vs III*
Flexural strength (MPa)	88.63 ± 5.217	116.5 ± 7.067	77.91 ± 3.784	(0.0001)*	(0.001)*	(0.0001)*
Thermal diffusivity (mm^2/sec)	0.062 ± 0.012	0.123 ± 0.014	0.079 ± 0.006	(0.0001)*	(0.001)*	(0.0001)*
Water sorption (mg/cm^2)	0.65 ± 0.018	0.59 ± 0.052	0.53 ± 0.038	(0.006)*	(0.0001)*	(0.003)*

*Significant ($p < 0.05$), PMMA: Polymethylmethacrylate

Water Sorption

Both the experimental groups showed decreased water sorption as compared to the control group specimens (0.65 ± 0.018 mg/cm^2). For PMMA resin reinforced with sapphire fillers the water sorption was 0.59 ± 0.052 mg/cm^2 and for PMMA reinforced with silver fillers it was even lower (0.53 ± 0.038 mg/cm^2). Intergroup comparisons were found to be significant on comparison of group I with III ($p < 0.0001$), group I with II ($p < 0.006$) and group II with III ($p < 0.03$) (Table 2 and Fig. 8).

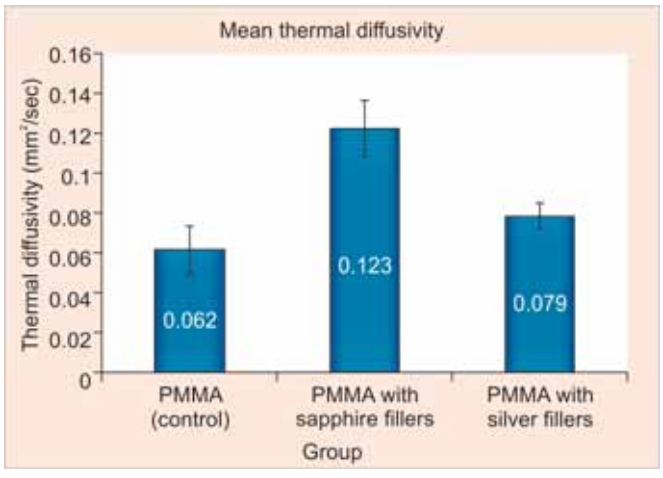

Fig. 7: Mean and standard deviation values of thermal diffusivities of the three groups

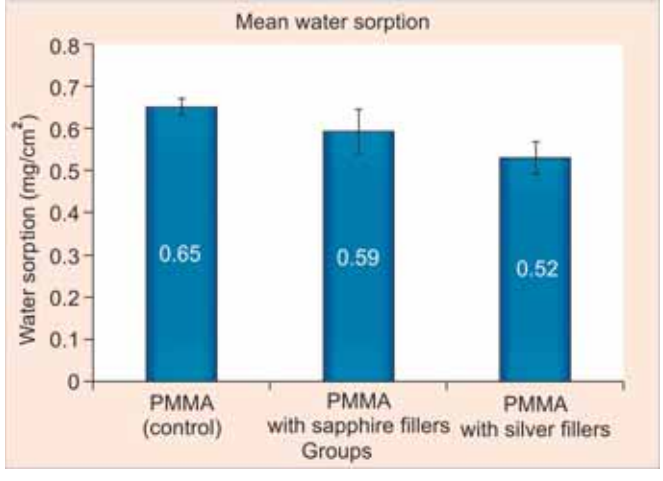

Fig. 8: Mean and standard deviation values of water sorption of the three groups

DISCUSSION

Numerous studies have been reported in dental literature pertaining to the reinforcement of polymethylmethacrylate with various types of fibers and fillers in order to improve its mechanical properties. Reinforcement has been achieved through inclusion of polyaramid fibers,[19,20] carbon fibers,[19] glass fibers,[19-22] ultra high modulus polyethylene fibers,[19,22] chopped PMMA fibers,[23] metal powders[24,25] and sapphire whiskers.[10,26,27] But, relatively few studies are present in literature where emphasis of research is also widened to include improvement in other physical properties, like thermal conductivity/diffusivity and water absorption with the same reinforcements.

It is, however, important to remember that the methodologies that improve the mechanical properties of a denture base material should not affect its physical and thermal properties. Therefore, in the present study, fillers, such as sapphire (aluminium oxide) and silver, were chosen as reinforcers, because they not only influence the strength of the resin but also provide thermally conducting pathways within the insulating acrylic resin matrix and possibly affect the water sorption. So, an overall enhancement of the physical, thermal as well as mechanical properties of acrylic resin can be expected.

Evaluation of Flexural Strength

Despite the material's popularity, fracture of the acrylic denture base has been a major problem. The mechanical strength of PMMA resin is not sufficient to maintain the longevity of dentures, in terms of their duration of service. A variety of physical properties can be used to assess the strength of denture materials. Zappini et al[28] noted that most studies evaluating the strength of denture base resins rely mainly on impact data and this may not simulate the clinical condition. Flexural strength tests are in fact better than impact strength tests to predict clinical function. Arnold et al[29] have reported that the ultimate flexure strength of a material reflects its potential to resist catastrophic failure under a flexural load.

In the current study, flexural strength was assessed to get an understanding of how denture base resins hold up under function, i.e. clinically. Addition of sapphire fillers significantly

improved the flexural strength of PMMA and this may be attributed to the uniform distribution of the filler within the matrix. Similar results have been shown by Ellakwa et al[10] who have demonstrated an increase in the mean flexural strength of PMMA resin reinforced with different concentrations (5-25%) of sapphire fillers. Grant and Greener[27] have also previously demonstrated that even in the absence of coupling agent, incorporating sapphire whiskers into heat-cured denture base resin substantially increased the flexural strength compared with unmodified heat-cured resin.

However, it was found that the addition of silver fillers significantly decreased the flexural strength of the acrylic resin polymer. Explanations for this reduction in strength is due to less number of silver fillers per unit area of the PMMA matrix as compared to sapphire because of larger silver filler particle size. This may also enhance the chances of void formation from entrapped air and moisture and incomplete wetting of the fillers by resin. Therefore, the net effect of embedding metal fillers was to weaken the polymer. Similar reasons were cited by Sehjpal and Sood [24] who reported that addition of silver, aluminium, or copper powder to PMMA in a concentration of 25% by volume significantly decreased (by as much as 35%) the tensile strength of the acrylic resin polymer.

Evaluation of Thermal Diffusivity

Thermal diffusivity is a material property related to thermal conductivity (k) which measures the rate at which a body with nonuniform temperature reaches equilibrium. The two parameters are related by the equation:

$D = k/ C_p r$, where C_p is the specific heat of the material and r is the density. 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.[10,11] In edentulous patients, the ability of the denture base to transmit thermal changes from the oral cavity to the underlying tissues of the palate may affect patients' general satisfaction with dentures and is related to thermal diffusivity. [12]

This study investigated the use of thermally conducting sapphire or silver fillers as additives to improve the thermal diffusivity of denture base polymers. An overall improvement in thermal diffusivity of the PMMA resin upon the addition of sapphire and silver fillers can be attributed to the formation of thermally conducting pathways within the polymer material. Ellakwa et al[10] reported that incorporating Al_2O_3 powder (alumina) from 5 to 20% by weight into conventional heat-polymerized denture base resin resulted in an increase in the thermal diffusivity in proportion to the percentages of the fillers. Messersmith et al[11] reported thermal diffusivity of denture base acrylic resin was increased by the addition of thermally conducting sapphire whiskers at concentrations of 9.35 and 15%.

The reinforcement with sapphire fillers, however, improved the thermal diffusivity more effectively than silver fillers. It is believed to be due to the proper distribution of sapphire fillers than the silver fillers, and therefore the ability of alumina particles to form continuous pathways for the conduction of heat through the insulating matrix [10, 11] Spherical particles of silver also significantly improved thermal diffusivity but the improvement was less as compared to sapphire fillers. Sehjpal and Sood[24] reported that the addition of silver, aluminium or copper powder to PMMA in a concentration of 25% by volume significantly increased the thermal conductivity by as much as 4.5 times that of unmodified acrylic resin.

Evaluation of Water Sorption

While fabricating the denture base from the acrylic resin, it comes in contact with water during polishing as well as cleaning, consequently during the use of denture it is constantly wetted by oral fluids. It has been shown that water molecules act according to the laws of diffusion. The diffusion presumably occurs between the macromolecules of the polymer which are forced slightly apart. This separation renders the molecules mobile and the inherent stresses created during heat-curing of the acrylic resin can be relieved with consequent intermolecular relaxation and possible changes in the shape of the denture.[30]

Polymethylmethacrylate (PMMA) absorbs water slowly over a period of time when placed in an aqueous environment. Though relatively moderate, this water exerts significant effects on the mechanical and dimensional properties of the polymer. It causes plasticization and lowers mechanical properties. According to Anusavice, it has been estimated that for each 1% increase in weight produced by water absorption, acrylic resin expands 0.23% linearly.[31]

In the present study, a decrease in water sorption of the samples upon addition of fillers was noted. This was because by the addition of filler particles, the actual number of PMMA molecules available on the surface of the disk for water sorption to occur decreases as compared to the control samples. Among the two types of fillers, as the sapphire fillers were smaller than the silver fillers and we had incorporated similar percentages of the two fillers individually by volume, the numbers of sapphire fillers were more in any given sample. The fewer and larger silver filler particles decreased the potential sites of water exchange to occur. On the other hand, because of the larger number of sapphire fillers, more sites were present on the surface of cured polymer (PMMA with sapphire fillers) for water molecule diffusion to occur by capillary action. Takaichi, Hiroyuki and Midori, Journal of the Japanese Society for Dental Materials and Devices (Journal Detail)[32] have also reported that the water absorption and solubility of PMMA resin decreased with the increase in the percentage of silver powder (5-20%) .

In view of the above discussion, it can be inferred that as compared to silver fillers, sapphire fillers are better for the reinforcement of polymethylmethacrylate resin. This is because

they are highly esthetic (so do not cause discoloration), have low density (so low weight of prosthesis) and bring about an improvement in the mechanical properties (flexural strength and fatigue strength) and thermal properties (thermal diffusivity) of polymethylmethacrylate (PMMA) resin.

Further research is needed to quantify the filler distribution within the polymer matrix. It may be suggested that filler particles should be distributed evenly within the acrylic resin matrix. Also, it must be noted that surface treatment and use of silane coupling agents could be done to enhance the bonding between the filler particles with the resin matrix. Further research is also needed to evaluate the effect of aging on these new reinforced denture base materials before clinical application.

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