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Effect of Incorporation of Silane-Treated Silver and Aluminum Microparticles on Strength and Thermal Conductivity of PMMA

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Abstract

Purpose: The purpose of this study was to study the effect of addition of metal filler particles on different strengths of polymethylmethacrylate (PMMA) and to evaluate the thermal perception in vivo.
Materials and Methods: The study was carried out in two parts. Part 1 of the study was an in vitro investigation regarding the effect of addition of metal fillers (aluminum and silver) in concentrations of 10%, 20%, and 30%, by volume on the tensile, compressive, and flexural strength of PMMA. Part 2 of the study comprised the clinical evaluation of the thermal perception by 10 edentulous patients provided with two sets of complete dentures, one fabricated with unfilled PMMA and another with 20% aluminum particle filled PMMA on the palatal portion of the maxillary denture. Recorded data were subjected to Student's *t*-test and ANOVA test.
Results: The mean tensile and flexural strength values among control and other groups were found to have statistically significant differences ($p < 0.05$) except for Al1 and Al2 groups. Mean compressive strength values among control and other groups were statistically significant ($p < 0.05$). In the clinical study, all 10 participants reported higher perception of hot and cold sensations in dentures with a metalized palatal portion.
Conclusions: Compressive strength increased progressively on increasing the filler concentration for both silver- and aluminum-filled PMMA. Silane-treated metalized PMMA showed reduction in tensile and flexural strength at 30% concentration. Metalized dentures led to an appreciable increase in thermal perception by the participants of this study.

Polymethylmethacrylate (PMMA) has been the most popular material for construction of dentures since the 1930s because of its many advantages including an improvement in esthetics, stability in the oral environment, easy laboratory manipulation, and inexpensive fabrication; however, the material does not fulfill all the desirable characteristics of an ideal denture base material. One of the major drawbacks is its low thermal conductivity, which compromises the patient's appreciation of taste and palatability.[1-4] Moreover, there is a lack of thermal stimulation to the underlying mucosa, resulting in reduction of its thickness, especially that of stratum corneum, predisposing the mucosa to injury due to functioning of the dentures.[5] It is therefore desirable to make the denture base thermally conductive, as it may serve to maintain the health of the underlying mucosa coupled with better appreciation of taste by conducting hot and cold sensations.[1,6-8]

Metal denture bases, despite having good thermal conductivity, still are not used as a routine material due to a complicated and tedious fabrication process, difficulty in relining, lack of metal and acrylic union, and seepage at interface. This is why although cast metals have better thermal conductivity, 95% of dentures are fabricated in PMMA.[9]

An ideal denture base material should have the advantages of both metal and acrylic resin, but at the same time eliminate or minimize their individual disadvantages. Unfortunately such a material has not yet been developed. Efforts to develop a new denture base material or modify the present ones so as to improve the mechanical properties and thermal conductivity are ongoing. Such modifications have been introduced in the structure of the polymer to improve its mechanical properties, such as flexural, tensile, and impact strengths, and fatigue resistance by the inclusion of fibers and metal inserts. These fibers include glass, carbon, aramid fibers, nylon, and ultra-high modulus polyethylene polymers. Metal inserts can be in the form of wires, plates, and fillers.

This study was undertaken to study the effect of addition of silver and aluminum filler particles on tensile, flexural, and compressive strength of PMMA. These fillers were pretreated with a silane coupling agent (Silano, Angelus, Brazil) and were added in different percentages. A clinical study was also done to evaluate the difference in thermal perception, if any, by the participants provided with conventional and metal-reinforced dentures.

Materials and methods

The study was carried out in two parts. Part 1 of the study was an in vitro investigation regarding the effect of addition of metal fillers (aluminum and silver) in concentrations of 10%, 20%, and 30% by volume on the tensile, compressive, and flexural strength of PMMA (Travelon, Dentsply, York, PA). Part 2 of the study comprised the clinical evaluation and comparison of thermal perception by 10 edentulous patients provided with two sets of complete dentures, one fabricated with unfilled PMMA and another with 20% aluminum particle filled PMMA on the palatal portion of the maxillary denture.

Part 1: Lab investigation

Preparation of dies

Two types of stainless steel dies were prepared. Both dies were in two parts: upper and lower member. In the first die, four molds were prepared—two dumbbell-shaped and two cylindrical. Each dumbbell-shaped mold had a total length of 100 mm. The test area was $24 \times 6 \times 3$ mm^3. Each cylindrical mold had a 9-mm length with a diameter of 6 mm as per IS:1816-1979 standardization. The second die had a $65 \times 10 \times 2.5$ mm^3 rectangular mold as per American Dental Association specification No. 12 for denture base resins.

Preparation of specimens

Silver and aluminum powder of 99.99% purity with approximately 1 μm particle size were selected as fillers. The metal fillers were preweighed to ensure filler concentration of 10%, 20%, and 30% by volume for both silver and aluminum and were treated with 1, 2, and 3 ml silane coupling agent, respectively. These silane-treated metal fillers were then mixed with the required amount of PMMA. Metalized PMMA and unfilled PMMA (control) in their correct volumes were then mixed with monomer separately in a porcelain mixing jar and packed in the stainless steel die molds. Polymerization was carried out at 74°C for 8 hours followed by boiling for 2 hours. Three sets each for eight groups of heat-polymerized PMMA specimens ($n = 15$/group) were prepared for testing tensile, compressive, and flexural strength.

The specimens were designated into groups as follows:

1. C (control group)
2. Ag1 (PMMA reinforced with 10% by volume silver particles pretreated with silane coupling agent)
3. Ag2 (PMMA reinforced with 20% by volume silver particles pretreated with silane coupling agent)
4. Ag3 (PMMA reinforced with 30% by volume silver particles pretreated with silane coupling agent)
5. Al1 (PMMA reinforced with 10% by volume aluminum particles pretreated with silane coupling agent)
6. Al2 (PMMA reinforced with 20% by volume aluminum particles pretreated with silane coupling agent)
7. Al3 (PMMA reinforced with 30% by volume aluminum particles pretreated with silane coupling agent)
8. Al3*(PMMA reinforced with 30% by volume aluminum particles without silane treatment)

This led to preparation of 120 specimens. For identification, all specimens were marked with color-coded dots on both ends. Control group specimens were left unmarked.

Figure 1 Metalized PMMA denture (intraoral view).

Testing of specimens

All specimens were tested for tensile, compressive, and flexural strength on a Universal testing machine (Lloyd LRX, Lloyd Instruments Ltd., Fareham, UK) at a 5 mm/min crosshead speed. Data were collected by a PC using the Nexygen system (Nexygen, Lloyd Instruments Ltd.).

Part 2: Clinical study

Ten participants were chosen from the outpatient Department of Prosthodontics, Subharti Dental College, Meerut, India. Permission to conduct the study was granted by the Ethical Committee of the Subharti Institute of Medical Sciences. Inclusion criteria for subjects were as follows:

1. Completely edentulous and in apparent good health.
2. Age range from 45 to 55 years.
3. First 10 people who reported in the outpatient department fulfilling the above two criteria.

Based on the abovementioned inclusion criteria, 10 edentulous male participants were chosen for the study. For each participant, two identical sets of dentures were made using duplicated casts. One set of dentures was made using unfilled PMMA, and in the second set, the palatal portion of the maxillary denture was made using 20% aluminum-filled PMMA containing fillers pretreated with silane coupling agent (Fig 1).

During finishing, it was ensured that the thickness of dentures in the mid-palatal region opposing the first premolars was 2 mm for both types of dentures for all the patients. This was measured using a Boley's gauge, and necessary corrections, if any, were made.

Testing thermal conductivity

Thermal perception was tested on each participant at the time of denture delivery. As the patients were blindfolded before starting the procedure, they were not aware of the type of denture to be inserted at the time of testing. First a maxillary denture made of unfilled PMMA was inserted in the mouth. A gutta-percha stick with its end dipped in boiling water was applied at the chosen point in the center of the palatal portion for 30 seconds, or less if the patient raised a finger as instructed, on perception of sensation of heat. The procedure was repeated five times with a 1-minute gap between heat applications. The same procedure was repeated for a second maxillary denture having 20% aluminum filler in the palatal portion. In case of perception of heat, if any, time was noted for each application.

For the perception of cold sensation Johnson earbuds (Johnson & Johnson Limited, Thane, India). were used to maintain uniformity. One end was saturated with ethyl chloride spray and applied on the central part of the palatal portion of the denture. The procedure was repeated five times as done for heat application. Appreciation of cold, if any, was noted along with the time. Then both sets of dentures were delivered to the participants, who were instructed to use them alternately for 1 week. All the participants were recalled after 4 weeks to ascertain their perception of hot and cold sensations for food and beverages. Findings were recorded for all 10 participants.

Data were recorded, interpreted, and subjected to statistical analysis. Mean, standard deviation, and standard error of mean were calculated, and the data were subjected to Student's *t*- test and ANOVA-F test.

Results

All values of tensile, compressive, and flexural strength for control and other groups are expressed in terms of mean and standard deviation in Table 1. An unpaired *t*-test was used to test the significant difference in pairs between the control and other groups for different strengths. A significant difference was observed between control and Ag1, Ag2, Ag3, Al3, and Al3* for tensile and flexural strengths at 5% level of significance, whereas all the groups were statistically significantly different from the control group for compressive strength (Table 2). The mean tensile strength value of group C was found to be 67.16 MPa, which was less than the values for groups Ag1, Ag2, Al1, and Al2. Ag1 showed the highest mean tensile strength value of 72.46 MPa with the lowest standard deviation and standard error of mean (i.e., 0.45 and 0.17, respectively). The tensile strengths of Al3 and Ag3 were less than group C. The lowest tensile strength obtained was for group Al3* (34.48 MPa).

The mean flexural strength value was the highest for group Ag1 (98.04 ± 7.22 MPa). The flexural strength of control group C was 80.64 MPa, which was less than that of groups Ag1, Ag2, and Al1. Little difference in the strength values for groups Al2 and C was observed. Flexural strength values for Al3 and Ag3 were significantly less than group C. Group Al3* again had the lowest value (45.1 ± 1.48 MPa).

The mean compressive strength value for each group was greater than for group C, and the difference was statistically significant. Group Al3 achieved the highest mean strength (136.4 MPa).

ANOVA-F test was applied to test the significance between control and other groups simultaneously for all the three types of strengths. A significant difference was observed at $\alpha = 0.05$ level of significance (Table 3).

Results of the clinical study, carried out to evaluate the thermal perception of 10 edentulous participants are presented in Table 4. Perception of hot and cold sensation was almost lacking in case of unfilled PMMA dentures, as there were only seven positive responses for hot and three for cold sensation tests for 50 applications each when compared to metalized dentures, which showed 100% responses.

After using both sets of dentures for a period of 4 weeks, the patients were asked about their preferences for PMMA dentures and dentures with a metalized palatal portion of the maxillary dentures, regarding their perception of hot and cold sensations. All 10 patients reported higher perception of hot and cold sensation in dentures with a metalized palatal portion and preferred using these dentures.

Discussion

Since its introduction in 1937,[10] PMMA has been the most widely used denture base material; however, one of the major drawbacks of using acrylic as a denture base material is its low thermal conductivity. Another shortcoming is its lack of strength, which at times leads to the breakage of the denture by accidental dropping or while in use. Attempts have been made to increase its strength by reinforcing it with organic fillers and also by metallic wires and meshes.[11-16]

A review of the available literature reveals that only a few studies have been carried out on the effects of adding metal fillers to PMMA.[17-22] Moreover, there is no clinical study regarding the efficacy of adding metal fillers on the thermal conductivity of PMMA-based denture. Therefore, this study was undertaken to check the effects of metalizing PMMA on its mechanical and thermal properties.

In this study, silver and aluminum were chosen as fillers because they not only have higher thermal conductivity, but also have been used in the oral cavity in one form or the other without any harmful effects.[6,23,24] Metal particles with an average size of 1 μm were selected so the resultant composite materials could be finished easily by conventional means. Also, the larger the filler particles as compared to the matrix particles, the more the powder settles when mixed with monomer, making the mechanical properties of the composite inferior.[19,25] Groups Ag1, Ag2, and Al1 showed improvement in tensile and flexural strength compared to group C. The mean tensile strength and flexural strength values for group C were found to be 67.16 and 80.64 MPa, respectively. Ag1 showed the highest mean tensile strength and flexural strength with values of 72.46 and 98.04 MPa, respectively.

Table 1 Mean and standard deviation (SD) values of tensile, compressive, and flexural strength for control and test groups

		Mean ± SD							
S. No.	Strength	Control	Ag1	Ag2	Ag3	Al1	Al2	Al3	Al3*
1	Tensile	67.16 ± 3.26	72.46 ± 0.45	71.34 ± 0.92	57.8 ± 1.74	68.94 ± 1.31	68.1 ± 1.79	53.66 ± 1.24	34.48 ± 2.71
2	Compressive	118.3 ± 0.44	126.4 ± 1.42	128.6 ± 1.25	132.4 ± 0.69	125.1 ± 0.65	128.4 ± 0.65	136.4 ± 0.86	124.1 ± 0.82
3	Flexural	80.64 ± 1.97	98.04 ± 7.22	87.84 ± 2.53	70.72 ± 2.40	81.60 ± 1.40	80.34 ± 2.91	60.08 ± 1.29	45.1 ± 1.48

Table 2 Results of unpaired *t*-test for tensile, compressive, and flexural strength

		Probability of t (tensile strength)		Probability of t (compressive strength)		Probability of t (flexural strength)	
S. No.	Group pairs	*p*-Value		*p*-Value		*p*-Value	
1	Control and Ag1	0.0213	$p < 0.05$	0.0000	$p < 0.05$	0.0044	$p < 0.05$
2	Control and Ag2	0.0434	$p < 0.05$	0.0000	$p < 0.05$	0.0012	$p < 0.05$
3	Control and Ag3	0.001	$p < 0.05$	0.0000	$p < 0.05$	0.0001	$p < 0.05$
4	Control & Al1	0.3067	$p > 0.05$	0.0000	$p < 0.05$	0.4040	$p > 0.05$
5	Control and Al2	0.5921	$p > 0.05$	0.0000	$p < 0.05$	0.8546	$p > 0.05$
6	Control and Al3	0.0003	$p < 0.05$	0.0000	$p < 0.05$	0.0000	$p < 0.05$
7	Control and Al3*	0.0000	$p < 0.05$	0.0000	$p < 0.05$	0.0000	$p < 0.05$

The *p* values <0.05 shows a significant difference between control and other groups at 5% level of significance.

Table 3 One-way ANOVA for tensile, compressive, and flexural strength

	Source of variation	Sum of squares	Degree of freedom	Mean sum of squares	Fischer's ratio (F ratio)	F critical
Tensile strength	B/W group	5,763.31	7	823.33	230.57	2.31
	Within group	114.27	32	3.57		
	Total	5,877.58	39			
Compressive strength	B/W group	1,040.18	7	148.59	182.55	2.31
	Within group	26.05	32	0.81		
	Total	1,066.23	39			
Flexural strength	B/W group	9,660.75	7	1380.11	133.75	2.31
	Within group	330.20	32	10.32		
	Total	9990.96	39			

In an earlier study, a reduction in PMMA tensile strength with metal fillers was observed. It was attributed to the decrease in cross-section of load-bearing polymer matrix, stress concentration set up by the presence of filler particles, a change in modulus of elasticity of metal-filled PMMA due to fillers, and the mode of crack propagation through metal-filled PMMA.[20] Voids, due to entrapped air and moisture, also reduce tensile strength because of incomplete wetting of fillers by resins.[26] However, in this study increased tensile strength at 10% and 20% filler concentration of silver and aluminum could be attributed to the use of coupling agent and selection of smaller particle size metal fillers.

There was a significant decrease in tensile and flexural strength as the volume fraction of filler was increased from 20% to 30% both for silver and aluminum. This is contrary to the belief of Sahu and Broutman, as quoted by Landen et al,[20] that composite strength should decrease rapidly with the first addition of filler, and remain essentially at that level with further addition. It is felt that the decrease in tensile strength of PMMA with 30% filler content could be improved by increasing the amount of coupling agent to ensure the better wettability of the filler.

Both types of metallic fillers at 10%, 20%, and 30% concentration contributed to a progressive increase in the compressive strength of PMMA with the increase in filler percentage. Compressive strength can further be increased by facilitating bonding between polymer and filler particles. This can be done by etching of filler particles and use of coupling agent. Metals have been treated with phosphoric acid or nitric acid for an etching-like effect[25] or with a 1% solution of stearic acid in benzene to form a submicron layer on the fillers.[19] Use of silane coupling agents has also been advocated.[5] At present, gamma methacryloxy propyl silane is the most common coupling agent used. The role of these additives is not altogether clear.[18,26,27] The coupling agents may change the chemical structure of the matrix or may act by preventing ingress of moisture or make up for what is otherwise not a perfectly clean surface. These may provide an intermediate layer at the filler matrix interface.

Table 4 Perception of hot and cold sensations by the participants provided with unfilled PMMA denture and metalized PMMA denture

	S1		S2		S3		S4		S5		S6		S7		S8		S9		S10	
	MP	UP	MP	UP	MP	UP	MP	UP	MP	UP	MP	UP	MP	UP	MP	UP	MP	UP	MP	UP
Hot perception (seconds)																				
	4.48	–	1.47	9.33	1.69	–	2.12	–	5.83	–	2.08	–	17.34	–	3.84	–	2.82	–	4.16	–
	5.23	–	2.04	–	1.72	–	2.04	–	6.76	16.12	3.16	–	18.16	–	3.69	–	2.13	–	4.35	–
	5.62	–	2.08	–	2.01	–	1.86	–	6.34	–	2.84	–	17.31	–	3.45	–	2.35	–	4.54	–
	5.93	20.92	1.73	11.42	1.88	9.46	2.34	–	5.81	–	3.14	–	17.84	–	4.04	21.06	2.86	–	3.86	–
	7.78	–	1.42	–	1.80	–	2.23	–	6.73	27.4	3.83	–	18.25	–	4.36	–	2.12	–	4.02	–
Cold perception (seconds)																				
	4.17	–	2.93	–	4.56	–	3.26	–	13.16	–	6.53	–	21.06	–	5.34	–	2.82	–	4.16	–
	4.32	19.08	1.69	–	3.12	–	3.08	–	12.21	–	6.10	–	21.54	–	5.06	–	2.13	–	4.35	–
	4.69	–	2.49	–	4.04	21.56	3.24	–	13.46	–	6.26	–	22.01	–	5.13	–	2.35	–	4.54	–
	4.56	–	3.19	–	3.86	–	3.62	–	13.08	–	5.93	–	21.35	–	6.08	21.06	2.86	–	3.86	–
	3.84	–	3.84	–	3.64	–	3.13	–	13.13	–	6.13	–	21.30	–	5.89	–	2.12	–	4.02	–

MP = metalized PMMA; UP = unfilled PMMA; – = no perception.

This layer may change the pattern and reduce magnification of stress.[26-29]

For the clinical study, two sets of complete dentures for each of the 10 patients were made. For one set of complete dentures, aluminum fillers were added in the palatal portion, as aluminum is considered more biocompatible than silver. The biocompatibility of aluminum has been shown by Kawahara et al.[30] In a tissue culture study they found copper to be cytotoxic whereas aluminum had little effect. Copper could give rise to local pigmentation whereas silver could cause generalized pigmentation of skin and mucous membrane. Aluminum appears to be one of the most suitable metals for making denture bases. Aluminum was used in a 20% concentration because this percentage does not affect the strength adversely, and it was shown from a previous study that 20% aluminum has a relative thermal conductivity 3.49 times greater than that of the control group.[17] Also, use of aluminum as a denture base material has been reported by many researchers and clinicians.[30-33]

In this study, metalized PMMA was used only in the palatal region due to esthetic reasons. Conduction time for perception of hot sensation was somewhat faster than the time for perception of cold sensation. Participants responded to cold sensation in an average of 7 seconds, whereas perception of hot sensation took an average of 4 seconds. Perception of hot and cold sensation was almost lacking in the case of unfilled PMMA dentures, as there were only 3 responses for cold sensation and 7 for hot sensation as compared to 50 (100%) responses to cold sensation and 50 (100%) responses to hot sensation in metalized dentures, in the 10 patients studied (Table 4). Metalized dentures thus contributed to a considerable extent in conduction of heat, which in turn leads to better appreciation of hot and cold sensations and better palatability of food.

All participants were instructed to wear conventional and metalized dentures alternately for 4 weeks. They were recalled after 4 weeks and asked for their preferences. All reported a higher perception for hot and cold sensation in metalized dentures and preferred using these dentures over the conventional PMMA denture. This is also supported by previous studies.[34,35]

Regarding the conduction of hot and cold, better results can be achieved with the use of metal denture bases, but they have inherent disadvantages. To overcome these problems, an alternate option is to metalize the conventional heat-cured PMMA, which could have advantages of both metal and resin. In addition, use of silane coupling agent is advocated so that strength of the metalized PMMA would not be compromised.

From the results of the study it can be interpreted that addition of metal fillers to PMMA can result in a composite material for denture bases that would improve thermal conductivity and mechanical properties—even more so by the use of a coupling agent to improve the bond between the metal and PMMA matrix. This will result in a material having advantages of both acrylic resin and metal while at the same time eliminating or minimizing their individual disadvantages.

In the light of this study it is suggested that complete dentures with a metalized palatal portion with 20% filler content are a preferred option, as they can contribute to better appreciation of taste by improving conduction of heat. They may also contribute to maintenance of the health of underlying mucosa.

Conclusions

Within the limitations of this study, the following conclusions could be drawn:

1. Addition of silane-treated silver and aluminum particles to PMMA in the concentration of 10%, 20%, and 30% by volume leads to a progressive increase in compressive strength of PMMA specimens.
2. Improved tensile and flexural strengths were observed in PMMA with only 10% and 20% silver fillers and 10% aluminum fillers. Only the results observed with 10% and 20% silver fillers were statistically significant.
3. Reduction in flexural strength of 20% aluminum-filled PMMA when compared to the control group was largely overcome by the use of coupling agent, as it was a mere reduction of 0.37% of control group.
4. Reduction in tensile and flexural strength was seen in specimens with 30% filler concentration for both silver- and aluminum-filled specimens of PMMA.
5. Nonuse of silane coupling agent as in 30% aluminum-filled PMMA, resulted in a marked reduction in tensile, compressive, and flexural strength when compared to silane-treated specimens.
6. Metalized PMMA dentures resulted in better appreciation of hot and cold sensations. Moreover, improvement in the palatability of food was recorded in all patients provided with a metalized palatal portion of maxillary dentures. It is advisable to provide such dentures to patients to improve the taste of food and maintenance of the health of the oral mucosa.

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