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Original Research Article

Comparative evaluation of flexural strength of conventional heat polymerized denture base resin reinforced with non-silanized and silanized aluminum oxide powder- An in vitro study

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ABSTRACT

Background: Although denture base acrylic resins have been used since many decades, the problem of fracture of denture base still remains. Many researchers have tried to reinforce the denture base by addition of metallic oxide nanoparticles. Silanization of such nanoparticles is recommended as it improves the bond between fillers and added nanoparticles.

Aims & Objective: To evaluate and compare the flexural strength (FS) of heat polymerized acrylic resin denture base material reinforced with 2.5 wt % of non-silanized and silanized aluminum oxide powder (Al_2O_3).

Material and Methods: Seventy-five specimens of heat polymerized acrylic resin were fabricated. The specimens were divided into three groups ($n = 75$) coded as Group N (non-silanized Al_2O_3), Group S (silanized Al_2O_3) and Group C (Control). The specimens of the remaining two groups were reinforced with 2.5 wt % of non-silanized and silanized Al_2O_3 . The flexural strength of the specimens was measured using 3-point bending test in a Universal Testing Machine.

Results: Data analyses using analysis of variance and Tukey's post-hoc test showed that adding 2.5 wt% of silanized Al_2O_3 ; Group S significantly increased the flexural strength compared to Group C and Group N ($p < 0.0001$).

Conclusion: Within the limitation of this study, Al_2O_3 fillers are potential reinforcers in denture bases which increased flexural strength.

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1. Introduction

In current century, no single grade of substance has influenced modern living than the synthetic plastics. Polymethyl methacrylate (PMMA) introduced by Dr. Walter Wright in 1937, proves to be the material of choice because of its favourable working characteristics, ease of processing, accurate fit, biocompatibility, stability in the oral environment, superior esthetics, and use of low cost

equipment.^{1,2} Despite of these advantages; poor mechanical strength, low fatigue strength, brittleness and poor thermal conductivity makes it less ideal in fulfilling the mechanical requirements of the denture base material.^{2,3}

In removable prosthodontics, fracture of acrylic resin dentures is an unsolved problem. Research has shown that 68 % of the complete dentures fabricated fractured within the first three years of service.⁴

There have been constant efforts to improve PMMA by increasing strength, dimensional stability, abrasion

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resistance and to attain radiopacity.³ Inclusion of metalwires, cast metal plates, fibres, metal oxides, and ceramic powders etc. have been carried out.³ The principle difficulty with the use of any strengthening filler is poor adhesion between the filler and resin, which leads to insignificant enhancement of mechanical properties.² To overcome this, surface modification of an inorganic particle with a silane coupling agent is a beneficial way to decrease its surface energy, increase its compatibility with polymer matrix by homogenous dispersion and thus improving the properties of the polymer/ inorganic particles thereby resulting in significant increase in flexural strength.⁵

Currently incorporation and processing of metal-composite systems in various dentures has been studied and found to be biocompatible thus improving the mechanical properties. Ceramic being white in colour also does not compromise the aesthetics.¹

It has been reported that untreated Al_2O_3 powder enhance physical and mechanical properties of acrylic resin although there have been no investigations regarding the effect of silanized Al_2O_3 powder on the mechanical properties of a conventional heat-cured acrylic resin. Thus this study was an effort to find and compare the flexural strength of heat polymerized denture base resin reinforced with non-silanized and silanized Al_2O_3 .

2. Materials and Methods

All the materials used in the current study are depicted in Table 1

2.1. Silanization of Al_2O_3 fillers

Reactive groups were introduced onto filler surface and this was achieved by reaction of the 3-methoxy propyl trimethoxy silane (MPS) with Al_2O_3 fillers.⁵

5 gm of aluminum oxide powder was added to 100ml of pure toluene solvent in a glass beaker; sonicated at room temperature for 20 mins. A magnetic stirrer was placed in the beaker which was kept on a stirring machine at room temperature. 0.12 ml silane coupling agent i.e. 3-methacryloxy propyl trimethoxy silane (2.5% wt to Al_2O_3) was added drop wise to it.^{5,6} The beaker was covered by a parafilm and was left standing for 2 days. The toluene solvent was removed by a rotary evaporator under vacuum at 60°C and rotation of 150 rpm for 30 mins.⁶

2.2. Specimen preparation

Brass metal dies measuring $65 \times 10 \times 3$ mm (ISO 1567 Standard) (Figure 1) were used for fabrication of stone molds, used as matrices for the fabrication of heat-polymerized acrylic resin specimens.²

A conventional heat-cured PMMA (DPI Heat CureTM, (Dental products of India Ltd, Batch no: 3148) was used as a matrix component and 2.5 wt % Al_2O_3 powder with a

grain size of 3 microns was used as a reinforcing agent.⁷ For the test, 75 specimens were prepared; specimens were divided into 3 groups ($n = 75$).

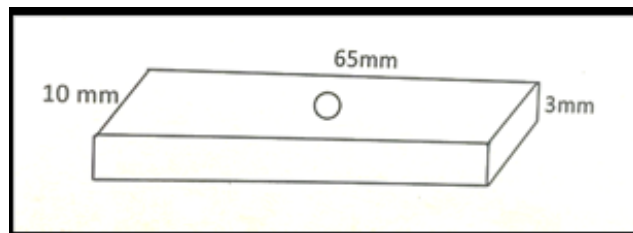


Figure 1: Metal die configuration for preparation of test specimens

2.3. Proportioning and mixing of the acrylic

Addition of silanized and non silanized Al_2O_3 fillers:

The incorporation of silanized and non-silanized filler powder to monomer was done at 2.5 wt %.⁵ The fillers were well dispersed in the monomer by ultra-sonication, using a probe sonication apparatus at 20W, 60 KHz for 3 minutes to break them into individual nano-crystals.⁵ The monomer-filler suspension was immediately mixed with acrylic powder which reduced the possibility of particle aggregation and phase separation. The proportion for mixing of acrylic resin was 2.5:1 powder/liquid ratio by weight.⁶ All the materials were mixed and manipulated as per manufacturer's instructions.

Specimens of Group C were fabricated in a conventional manner using manufacturer's recommendation.

The polymerized specimens were carefully removed and were finished using sandpaper. Polishing was done with felt buff cone and pumice. The finished and polished samples were then stored in distilled water for 1 week at 37°C .⁷

2.4. Flexural strength testing

The flexural strength of the specimens were determined using a 3-point bending test in a universal testing machine Star Testing System, India. Model No. STS 248, at a crosshead speed of 5 mm/min. The device consisted of a loading wedge and a pair of adjustable supporting wedges placed 50 mm apart. Specimens were loaded until fracture occurred.¹

Flexural strength was calculated using the following equation:⁷

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

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).

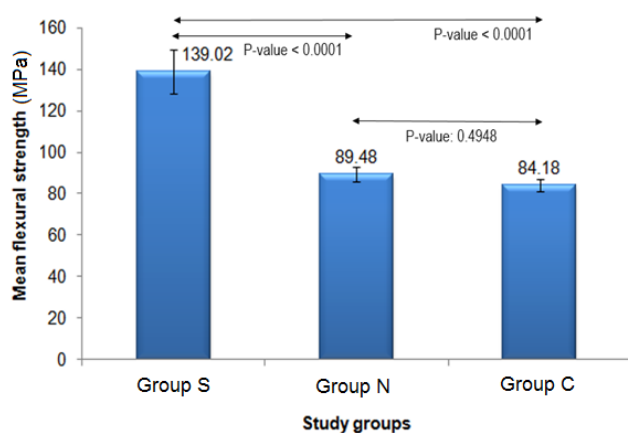
Table 1: Materials used in the current study

S.No.	Materials	Manufacturer	Batch no.
1	Heat polymerised acrylic resin	DPI Heat Cure™, (Dental products of India Ltd)	3148
2	Die stone	Ultrarock; Kalabhai Karson Pvt Ltd, India	140205
3	Aluminum oxide powder (Al ₂ O ₃)	SDFCL (s d fine-chem limited), India	25140K05
4	Silane coupling agent i.e. 3-methacryloxy propyl trimethoxy silane.	Alpha Aesar (A Johnson Matthey Company)	10181188
5	Toluene (solvent)	Emplura, India	K3F630294

3. Results

The data was analyzed using SPSS INC 18.0 Chicago. The p value was taken as significant when less than $p < 0.05$. The statistical tests used for the analysis of the result were ANOVA (one way analysis of variance) which was used to calculate the mean, median and standard deviation (Table 2) while pair wise comparison of means was carried out and tested for statistical significance using Tukey's post-hoc test.

Table 3 shows highly significant difference in the means of Group S and Group N ($p < 0.0001$). Also, the difference between Group S and Group C were statistically significant ($p < 0.0001$). However, the difference between Group N and Group C were statistically insignificant as revealed by p-value of 0.4948 ($p > 0.05$). This has been graphically shown in Graph 1.



Graph 1:

4. Discussion

The ultimate flexural strength of a material reflects its potential to resist catastrophic failure under a flexural load.¹ With respect to the fracture of dentures, Smith analyzed the practical situation and showed two types of failures, i) outside the mouth, caused by impact forces, i.e. a high stress rate and ii) inside the mouth usually in function, which is probably a fatigue phenomenon, i.e. a low and repetitive stress rate.⁸

Various attempts to improve the strength of PMMA include addition of metal wire to resin and fabrication of cast metal. The primary problem with using metal wire is poor adhesion between the wire and resin thus resulting in failure due to stress concentration around the embedded inserts. Although metal plates increase strength but they are expensive and also prone to corrosion.⁸

Incorporation of fibres enhanced the mechanical properties; this method has various problems which includes irritation to the tissues, increased fabrication time, difficulties in handling, the need for precise orientation and placement or bonding of the fibres within the denture base resin.⁷

One of the most phenomenal events in the field of dental material sciences is the introduction of composite materials being of increased strength and low mass. These materials are a combination of very high modulus fibres and a resin matrix which binds them, so that the reinforced material work together to resist the load.¹

The fundamental cause for the use of ceramic filler in opposition to metal filler is its low filler density.² The density of aluminum (3.99g/cm³) is least, thus light weight of acrylic resin denture bases is retained. Furthermore, these composite powders have the advantage of being white, and therefore are less likely to alter the finished appearance of the denture base material than the metal powders.^{1,2}

A recent systematic review have been studied on Methyl methacrylate monomer of denture base resins which was modified with several monomers to achieve better physico-mechanical properties without compromising the biocompatibility. There have been very less research on effect of incorporation of aluminum oxide in denture base resin.⁹

Aluminum oxide (Alumina-Al₂O₃), possess strong ionic inter atomic bonding, thereby enhancing the material characteristics. Amongst the oxide ceramics alpha phase alumina is the stiffest and strongest. It has increased hardness, excellent dielectric properties, refractoriness, better thermal properties, decreases polymerization shrinkage, reduces warpage, imparts radio-opacity to the material and inhibits the growth of bacteria over the denture surface.^{7,10}

Table 2: The mean, standard deviation, and minimum and maximum values for flexural strength

Groups	Mean (MPa)	SD	Median (MPa)	Min (MPa)	Max (MPa)
Group S	139.02	26.26	136.13	96.28	194.44
Group N	89.48	8.29	87.30	76.52	111.63
Group C	84.18	7.51	82.72	67.86	101.43

Group S: Silanized Al₂O₃, Group N: Non- Silanized Al₂O₃, Group C: Control group

Table 3: Comparative evaluation between control and experimental groups

Groups	P-value	Significance
Group S and Group N	< 0.0001	Highly significant
Group S and Group C	< 0.0001	Highly significant
Group N and Group C	0.4948	Not significant

Group S: Silanized Al₂O₃, Group N: Non- Silanized Al₂O₃, Group C: Control group

Another important factor to be considered is the particle size, as larger particle size tends to settle down when mixed with monomer and thus decrease the tensile strength.¹¹ The average particle size of resin powder is 121.2 microns.¹² Marie et al (1994),¹¹ Saritha Mk et al (2012)¹ and Asar et al (2013)¹² used metal oxides of particle size 10mm, 5-22mm and 7-13mm respectively. Mahroo Vojdani et al (2012)⁷ used aluminum oxide of 3 mm particle size as a reinforcing agent, in accordance with which, the present study was performed.

Yadav (2011)¹³ found decrease in flexural strength with 5% Al₂O₃. This decrease in flexural strength was due to irregular distribution of untreated Al₂O₃ filler particles and decrease bonding between filler particles and acrylic resin. Thus, he suggested Al₂O₃ should be silane treated. In the present study, the same consistent result was obtained for Group N (89.48 Mpa) as that of Mahroo Vojdani (2012)⁷, who got highest flexural strength (90.51 Mpa) at 2.5 wt % concentration of aluminum oxide without discoloration and hence, this was taken as optimum concentration for evaluation of flexural strength.

Matinlinna JP et al (2004) reviewed silane coupling agent for surface modification and concluded that 3-methacryloxy propyl trimethoxy silane enhances the surface bonding with resin matrix, reduces surface energy, increases compatibility with polymer matrix, provides dispersion homogeneity and improves the properties of the polymer particles.¹⁴

The result obtained in this study are; the mean flexural strength of Group S (139.02 MPa) is 65.5% and Group N (89.48 Mpa) 4.76% greater than Group C (84.18 Mpa). This study shows that flexural strength can be increased by reinforcement with 2.5% by wt silanized aluminum oxide powder.

In the literature, various investigators incorporated untreated Al₂O₃ in different percentages and found increase in flexural strength. Sehjpal and Sood(1989)¹⁵ found an increase in flexural strength at 25% concentration by volume, Marie et al (1994)¹¹ found the same at 5% by volume, Ayman E. Ellakwa (2008)² found the same at 15 % by wt, Yadav P et al (2012)¹³ found the same at 30% by

volume and Saritha M.K et al (2012)¹ found the same at 15 % by wt . Mahroo Vojdani et al (2012)⁸ found similar results at 2.5% by wt.(mean flexural strength 90.51 Mpa) In the present study, a highly significant increase in flexural strength was found by reinforcement with 2.5 % by wt of silanized Al₂O₃, which was a much lower concentration than the ones mentioned. The mean flexural strength, at this concentration of silanized Al₂O₃ powder, (139.02 MPa) was higher than that obtained using non silanized Al₂O₃ powder in previous studies.

5. Clinical Implications of the Study

When the entire spectrum of this study is analyzed, it becomes evident that the heat polymerized acrylic dentures reinforced with silanized Al₂O₃ powder increases the flexural strength of the denture base material and thus, reduces the probability of occurrence of fracture.

Other clinical implication with the use of this material is that it increases the thermal diffusivity of the denture base material, which enhances the patient's perception to hot and cold, hence improving the adaptability of the patient to the denture. This in turn, aids in better comfort and satisfaction with the prosthesis in place.^{15–17}]In addition to this, it imparts radio-opacity to the material so that any fractured remnants can be detected radiographically.^{18,19}

6. Limitations of the Study

In the oral cavity, reinforced denture base is exposed to forces of varying magnitudes acting in different directions. The same situation could not be simulated in this in vitro study. SEM examination of the samples to evaluate the adhesion of aluminum oxide fillers on the surface of PMMA was not performed.

7. Scope for Further Studies

1. Fatigue testing of these materials under dynamic loading using the denture base configurations in simulated oral conditions, using saliva or its substitutes

is an area for further research. Further research is required to evaluate the effect of aging on the new reinforced denture base material.

2. Other physical and mechanical properties like thermal diffusivity, hardness, abrasion resistance, color stability and disinfectant property can be studied.
3. Further research is needed to quantify the filler distribution within the polymer matrix.

8. Conclusion

Within the limitations of the study following conclusions can be drawn:

1. Specimens with reinforcement showed increase the flexural strength.
2. Reinforcement with 2.5 % by wt silanized Al₂O₃ (Group S) showed highly significant increase in flexural strength.
3. There was no significant increase in the flexural strength of heat polymerized acrylic resin denture base specimens reinforced with 2.5 wt% non-silanized aluminum oxide powder (Group N).

9. Source of Funding

None.

10. Conflict of Interest

We clarify that there is no conflict of interest with any financial organization regarding the material discussed in the manuscript.

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