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EVALUATION OF FLEXURAL STRENGTH OF HEAT CURE DENTURE BASE MATERIAL REINFORCED WITH 1WT% OF DIFFERENT NANOPARTICLES AND SIZES-AN INVITRO STUDY

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Abstract

Purpose: *To evaluate the effect of adding 1wt% of Al_2O_3, SiO_2, and ZrO_2 nanoparticles to heat cure acrylic resins on their flexural strength.*

Materials and Methods: *PMMA is the widely accepted denture base material but has got the disadvantage of low mechanical strength. So the study evaluates whether reinforcement with nanoparticles improves its flexural strength. A total of 88 specimens are prepared in 4 groups (Control, 1wt% Al_2O_3, 1wt% SiO_2, 1wt% ZrO_2) and 3 subgroups (5 nm & 15 nm of all Nano groups) (n =22/group). The PMMA powder and nanoparticles are mixed with each other by means of ball milling and is invested in type III dental stone for processing. Flexural strength is measured via three-point bending tests. Subsequently, SEM analysis is performed for specimens from each group to ensure homogenous distribution.*

Results: *The flexural strength of polymethylmethacrylate (PMMA) after adding 1wt% of aluminium trioxide 15nm significantly increased ($p < 0.05$) followed by 1wt% of silicon dioxide 15nm. The scanning electron microscopy analysis revealed that the particles were homogeneously dispersed in PMMA matrix.*

Conclusion: *The mechanical properties of heat cure PMMA can be increased by addition of nanoparticles to PMMA powder. 1wt% of aluminum trioxide-15nm diameter has significantly improved the flexural strength of PMMA.*

Keywords: **nanoparticles, Flexural strength, Aluminum trioxide, Zirconium dioxide, Silicon dioxide.**

Introduction

Denture base is defined as the part of a denture that rests on foundation tissue to which the teeth are attached. The denture base material is defined as any substance of which the denture can be made. -GPT9

Poly (methyl methacrylate), is widely used in rehabilitation of edentulous patients because of its satisfactory esthetics, ease of use, low cost, light weight and stability in the mouth[5,8]. Many different materials such as bone, wood, ivory, vulcanized rubber, polystyrene, light activated UDMA are used but PMMA remains preferred. But the material has the disadvantage of low mechanical properties such as low flexural strength and impact strength resulting in fracture of the denture when subjected to occlusal flexural strength of standard denture base polymers prescribed is 65 MPa by the ISO 1567.

Nanoparticles are effective reinforcing materials and used as fillers for polymeric materials to improve their mechanical properties. They are characterized by their smaller size, larger specific surface area, and also strong interfacial interaction with organic polymers. The dimension of the material is in nanometer, which leads to unique properties. In the current study 1wt% of Al_2O_3, SiO_2, & ZrO_2 of diameter 5nm and 15nm are added to improve the mechanical properties.

The purpose of the study is to evaluate the flexural strength of heat cure PMMA on adding 1wt% of Al_2O_3, SiO_2, & ZrO_2 of diameter 5nm and 15nm.

Materials and Methods

Polymer is a macromolecule composed with repeated unit. They are formed by condensation polymerization or addition polymerization. Acrylic resins are polymer esters of methacrylic acids. PMMA is the combination of methyl methacrylate with chemical formula $(C_5H_8O_2)$ n. PMMA is a linear thermoplastic polymer which lack of methyl group on its backbone carbon chain.

PMMA is commonly used denture base material. The PMMA employed in the current study is IVOCLAR Vivadent SR triplex hot pink heat cure veined denture base material. The powder liquid ratio is followed according to the manufacturer's instruction as 20 ml for 43.8 gm. of powder by means of measuring jar and graduated cylinder. The specimens of the dimension 65mm in length, 10mm wide and 3 mm thickness are fabricated as per the ISO 1567 standard, by using templates. In the present study, three different nanoparticles were added in ratio of weight 1 wt% nanoparticles (Al_2O_3, ZrO_2, SiO_2) to the heat cure acrylic resin. 1wt% nanoparticles (Al_2O_3, ZrO_2, SiO_2) has been used so as to prevent agglomeration and to ensure homogenous distribution without adversely affecting the mechanical properties.

These were categorized into the following 4 groups pure PMMA – Group I, PMMA with 1wt% Al_2O_3-Group II, PMMA with 1wt% SiO_2 – Group III, PMMA with 1wt% ZrO_2 – Group IV and 3 subgroups PMMA with 5nm 1wt% Al_2O_3 (Sub Group II a) & 15 nm 1wt% Al_2O_3 (Sub Group II b), PMMA with 5nm 1wt% SiO2 (Sub Group III a) & 15nm 1wt% SiO_2 (Sub Group III b), PMMA with 5nm 1wt% ZrO_2 (Sub Group IV a) & 15 nm 1wt% ZrO_2 (Sub Group IV b).

The investment is made using type III gypsum product. The PMMA powder and Nano particles are mixed by means of ball milling machine (RETSCH PM 100). The speed is set at 350 rpm for one hour with four steel balls of diameter 10mm with an interval at 30 minutes.

The PMMA powder (IVOCLAR Vivadent SR Triplex Hot) along with the nanoparticles (Matrix Nano) is mixed with the liquid (monomer) and left for 10 minutes and then packed into the flask. The flask is approximated and kept for curing in the curing unit (DELTA – Tempsen Polybath) for two hours and then boiled for 1 hour. The flasks were allowed to cool for 30 minutes and placed under running water for 20 minutes to ensure complete cooling. Then the specimens were deflasked, trimmed and polished using 200, 400 and 600 grit sand paper and the specimens are immersed in distilled water. The samples are then subjected to three-point bending test using universal testing machine (INSTRON 3345 – Bluehill 3). After being subjected to three-point bending tests the specimens are analyzed under scanning electron microscope (CAREL ZEISS

EVO18) to ensure homogenous distribution of nanoparticles.

Statistical Analysis

The data was expressed in mean and standard deviation. Statistical Package for Social Sciences (SPSS 20.0) version used for analysis. One-way ANOVA (Post hoc) followed by sh used for analysis. p value less than ($p<0.05$) considered statistically significant at 95% confidence interval.

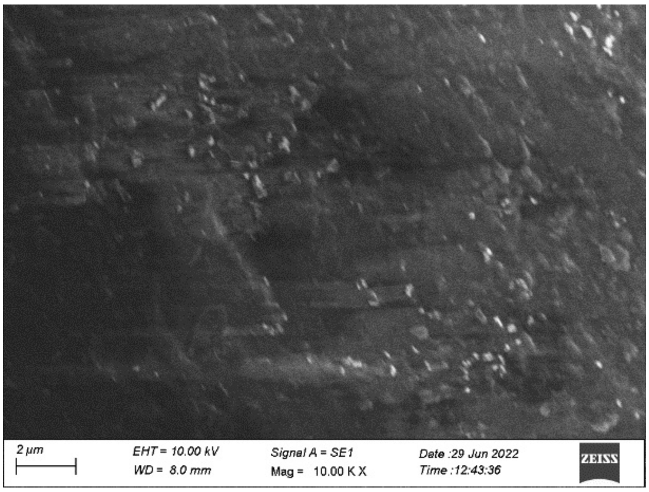

Fig 1: Aluminum trioxide 5nm – Group II a

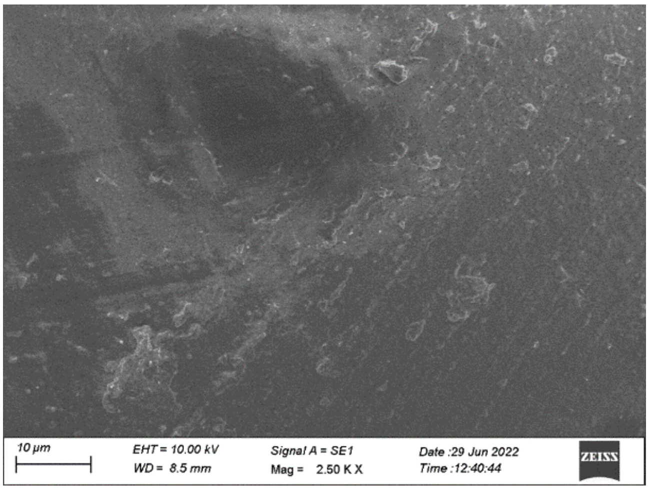

Fig 2: Aluminum trioxide 15nm – Group II b

Table I –Group II a - aluminum trioxide 5nm samples

	Flexural Strength [MPa]	Flexural Strain [%]	Load at Maximum Flexure load [N]	Flexural Modulus [MPa]
1	70.2	2.8	84.26710	2980
2	80.1	3.1	96.11877	3240
3	67.6	3.1	81.09328	2840
4	72.0	2.8	86.41303	2960
5	78.9	3.1	94.65240	3270
6	42.3	1.6	50.80054	2770
7	74.0	3.3	88.77422	2860
8	77.0	3.3	92.39271	2960
9	75.2	3.0	90.26265	3060
10	59.4	2.3	71.24817	2990
11	76.0	3.1	92.65420	3260
Maximum	80.1	3.3	96.11877	3270
Minimum	42.3	1.6	50.80054	2770

Table II –Group II b - aluminum trioxide 15nm samples

	Flexural Strength [MPa]	Flexural Strain [%]	Load at Maximum Flexure load [N]	Flexural Modulus [MPa]
1	77.9	3.5	93.50188	2750
2	75.1	3.1	90.10218	2940
3	93.6	4.4	112.28961	3000
4	73.3	3.6	88.00625	2690
5	82.2	3.3	98.60590	3230
6	82.4	3.5	98.83340	2890
7	88.3	4.3	105.91770	2800
8	72.4	3.2	86.82915	2900
9	78.9	3.7	94.67442	2870
10	87.2	3.8	104.69430	3100
11	81.2	3.3	90.00590	3210
Maximum	93.6	4.4	112.28961	3230
Minimum	72.4	3.1	86.82915	2690

Results:

The values of control group exhibit a maximum flexural strength value of 94.3 MPa and a minimum of 55.8 MPa with an average mean of 71.96 ± 11.26 MPa.

The flexural strength of heat cure PMMA reinforced with 1wt% of 15 nm aluminum trioxide (81.13 ± 6.60 MPa) (table III) exceeded those of other groups followed by 1wt% of 15nm Silicon dioxide (73.84 ± 6.46 MPa) (table III) and the group reinforced with 1wt% of 5 nm Zirconium dioxide (63.40 ± 19.56 MPa) (table III) had the least strength. The values of 15nm Al_2O_3 and 15nm SiO_2 were found to be statistically significantly higher than those of the control group ($p < 0.05$).

Table-III: Mean flexural strength (MPa) of different groups

Groups	Material	Flexural strength (MPa) (MEAN±SD)
Group-I	Heat cure PMMA	71.96±11.26
Group-IIa	Al2 O3 – 5nm	70.24±10.94
Group- IIb	Al2 O3 – 15nm	81.13±6.60
Group- IIIa	SiO2 – 5nm	71.51±8.04
Group- IIIb	SiO2 – 15nm	73.84±6.46
Group- IVa	ZrO2 – 5nm	63.40±19.56
Group- IVb	ZrO2 – 15nm	68.74±7.98

Table IV- Multiple comparison of mean flexural strength (MPa) between the groups

Groups	Flexural strength (MPa) (MEAN±SD)
Group-I	71.96±11.26
Group-IIa	70.24±10.94
Group-IIb	81.13±6.60
Group-IIIa	71.51±8.04
Group-IIIb	73.84±6.46
Group-IVa	63.40±19.56*
Group-IVb	68.74±7.98

Discussion

PMMA is commonly used material for denture base because of its ease of use, polishability and light weight. But it has got the disadvantage of low flexural strength and impact strength resulting in the fracture of denture. Reinforcement with nanoparticles is expected to improve the mechanical properties.

The present study evaluated the flexural strength of heat cure PMMA reinforced with different nano particles and sizes. Different nanoparticles, such as aluminum trioxide, silicon dioxide, zirconium dioxide of 5nm and 15nm diameter were used. According to the study conducted by Karci et al[34] particles of size as low as 15 nm produced better results so in the current study comparison between 5nm and 15 nm were done to test the efficacy. The mean flexural strength of the control group obtained is -GROUP I- 71.96 ± 11.26 MPa. The mean flexural strength of other groups when compared to the control group clearly states that group IIb (81.13 ± 6.60 MPa) (table III) has the highest values followed by Group IIIb (73.84 ± 6.46 MPa) (table III). Group IVa has got the low-

Table V Comparison of mean flexural strength (MPa) of group-Iva with other groups

Groups	Flexural strength (MPa) (MeanSD)	P value
Group-I	71.96±11.26	0.621
Group-IIa	70.24±10.94	0.906
Group-IIb	81.13±6.60*	0.037
Group IIIa	71.51±8.04	0.809
Group IIIb	73.84±6.46	0.556
Group-IVa	63.40±19.56	
Group-IVb	68.74±7.98	0.971

(*p<0.05 significant differene compared group-Iva with other groups) est strength (63.40 ± 19.56 MPa) (Table III)

The minimum value of flexural strength of standard denture base polymers prescribed is 65 MPa by the ISO 1567. The values (table III) of Group IIa (70.24 ± 10.94 MPa), IIb (81.13 ± 6.60 MPa), IIIa (71.51 ± 8.04 MPa), IIIb (73.84 ± 6.46 MPa), IVb (68.74 ±7.98 MPa) and control - group I (71.96 ± 11.26 MPa) satisfies this requirement. The values of group IVa - zirconium dioxide 5nm (63.40 ± 19.56 MPa) is less than that of the ISO 1567 requirement. On comparing mean flexural strength of aluminum trioxide 5nm (70.24 ± 10.94 MPa), silicon dioxide 5nm (71.51 ±8.04 MPa) and Zirconium Dioxide 5nm (63.40 ±19.56 MPa) the silicon dioxide 5nm had the highest mean flexural strength. On comparing mean flexural strength of aluminum trioxide 15nm (81.13 ± 6.60 MPa), with silicon dioxide 15nm (73.84 ± 6.46 MPa) and Zirconium Dioxide 15nm (68.74 ± 7.98 MPa) the aluminum trioxide 15nm (81.13 ± 6.60 MPa) had the highest mean flexural strength (Table IV).

On comparing mean flexural strength of aluminum trioxide 5nm (70.24 ± 10.94 MPa) and 15nm (81.13 ± 6.60 MPa) no significant difference was observed. The p value is 0.502 (P>0.05).On comparing mean flexural strength of Silicon Dioxide 5nm (71.51 ± 8.04 MPa) and 15nm (73.84 ± 6.46 MPa) no significant difference was observed. The p value is 1.00 (P>0.05). On comparing mean flexural strength of Zirconium Dioxide 5nm (63.40 ± 19.56 MPa) and 15nm (68.74 ± 7.98 MPa) no significant difference were observed. The p value is 0.971 (P>0.05). On comparing the mean flexural strength of Group IIa with other groups no significant difference were observed (p>0.05). On comparing the mean flexural strength of Group IIb and Group IVb with other groups significant difference were observed (p<0.05). On comparing the mean flexural strength of Group IIIa and Group IIIb with other groups no significant difference were observed (p>0.05). On comparing the mean flexural strength of Group IVa with other groups significant difference were observed (p<0.05) (Table V).

Multiple comparison of mean flexural strength (MPa) between the groups states there is significant difference on comparing Group III a with Group IVa. (p<0.05). In accordance with the study conducted by Unal et al[39] the shape and size of the filler particles, distribution in the polymer matrix, and connection to the matrix play a very important role, and also the size of metal oxides should be sufficiently low for homogenous mixtures. This prevents a heterogeneous mixture, and nanoparticles will fill in the cracks between polymer matrix, thereby preventing the movement of the chain. In addition to that, the percentage of the nanoparticles should be kept low so that the particles will be embedded in the resin without agglomeration. Balos et al[23] also concluded that low concentration provides better properties.

According to the study conducted by Mahroo et al[49] 2.5 wt% of aluminum trioxide with grain size of 3 micrometer significantly increased the flexural strength. The addition of 5 wt% Al_2O_3 powder caused a 5.82% reduction in flexural strength. Possible explanations for this decrease in strength could be a decrease in cross-section of the load-bearing matrix of polymer; stress concentration due to too many filler particles; mode of crack propagation through the specimen because of increased amount of fillers; void formation from the entrapped air and moisture; incomplete wetting of fillers by resin; and acts as an interfering factor in the integrity of the polymer matrix. In this study, 1wt% has been used so as to prevent agglomeration and to ensure homogenous distribution without adversely affecting the mechanical properties.

According to the study conducted by Neveen et al[11] High Impact acrylic resins reinforced with 5% and 15% ZrO_2 showed that it increased flexural strength and the flexural strength was proportional to the concentrations. In contrast to this, Ihab and Moudhaffar[28] compared the flexural strength after adding ZrO_2 nanoparticles and stated a statistically significant reduction of more than 5%. So addition of nanoparticles can influence the flexural strength both in a positive as well as negative aspect.

According to the study conducted by Reem Abualsaud, the Nano SiO_2 particles increased the flexural strength. Additionally silane treatment of the nanoparticles enabled stronger bonds with the matrix.

In the current study highest values are obtained by using 15nm Aluminum trioxide (81.13±6.60 MPa) followed by 15nm silicon dioxide (73.84±6.46MPa) (table III). The p value is significant for the 15nm 1wt% Al_2O_3 ($p<0.05$). The zirconium dioxide 5 nm had the lowest value (63.40±19.56 MPa) with a significant p value ($p<0.05$).

On comparing the diameter among the same groups no statistically significant results were obtained ($p>0.05$) but still 15nm sub-groups of all the three major groups had better results when compared to the 5nm sub-groups (Table I and Table II). In the study conducted by Karci et al[34] the specimens after testing for flexural strength are then subjected to SEM analysis to ensure homogenous distribution. Similarly in the present study, specimens after three point bending test were analysed under SEM for ensuring homogenous distribution. (Fig. 1 and Fig. 2). This homogenous distribution as stated by Karci et al[34] has promoted the mechanical properties of the material. A few studies recommend using coupling agents to prevent the agglomeration of particles. They have also stated that the well-distributed particles are only capable of better stress transfer and reinforcement. The null hypothesis was rejected, as the flexural strength has changed after the addition of nanoparticles. The flexural strength of heat cure PMMA reinforced with 1wt% of 15 nm aluminum trioxide exceeded those of other groups followed by 1wt% of 15nm Silicon dioxide and the group reinforced with 1wt% of 5 nm Zirconium dioxide had the least strength.

Variations in the particle size were found to be effective in improving the strength. According to the study conducted by Karci et al[34] the nano particles of diameter as low as 15 nm were yielding a good improvement. In the current study comparison between 5nm and 15nm was done to evaluate whether 5nm diameter particles provided better results. The results do not show statistically significant results but 15nm groups had better results compared to the 5nm sub groups.

In the present study, the saliva was not considered since this study was an in-vitro study, whether it might affect the results clinically is not evident. Furthermore, the leaching of particles might vary the biological properties of heat activated denture base material. This has proven to be a major limitation of the study and thence an in-vivo study can be considered in the near future. The optical properties of aluminum trioxide were not that satisfactory as it caused greyish discoloration. Henceforth, efforts have to be undertaken for improving the optical properties of aluminum trioxide nanoparticles.

Summary and Conclusion

Reinforcing PMMA with the nanoparticles such as aluminum trioxide, silicon dioxide and zirconium dioxide has proved to be beneficial in improving the mechanical properties. The flexural strength has been increased on addition of these particles. Adding nanoparticles can adversely affect the mechanical properties also. It is observed that Lower concentrations of nanoparticles prevents agglomeration and have better results. Concentration as low as 1wt% has yielded positive results. The Particle size also plays a vital role in ensuring reinforcement. Particle size of about 15nm has yielded better results.

The following conclusions can be drawn from this study

i. Aluminum trioxide Nanoparticles of diameter 15nm increase flexural strength better than silicon dioxide and zirconium dioxide.

ii. The groups with 15nm dimension have superior property than the 5nm diameter.

iii. In vivo studies are recommended to understand clinical results.

iv. Optical properties of aluminum trioxide have to be improved for better esthetics.

v. SEM images shows that the nanoparticles were distributed homogeneously in the groups. The homogenous distribution in turn increases the flexural strength by uniform stress distribution.

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