Effect of Silica Nanoparticles On Flexural Strength And Surface Hardness Of Heat Polymerized Acrylic Resin.

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ABSTRACT

Statement of problem
Various studies are present to increase the strength and surface hardness of heat polymerized acrylic resin by addition of material or surface treatments. The present study to evaluate an effect of silica nanoparticles on flexural strength and surface hardness of heat polymerized acrylic resin.

Purpose
The purpose of this study was effect of silica nanoparticles, incorporated into Polymethyl Methacrylate, on flexural strength and surface hardness. The present study compared the effect of silanated and non silanated silica nanoparticles on the flexural strength and surface hardness of heat polymerized acrylic resin.

Material and Methods
A total of 270 acrylic bars were fabricated, in two batches of 135 each, for testing flexural strength by universal testing machine and surface hardness determined using a digital micro Vickers hardness tester. The control group and subgroups had a sample size of 15 each with varied concentrations of nanoparticles by weight. The fabricated samples were tested for flexural strength and surface hardness.

Results
Flexural strength was highest for PMMA (Polymethyl methacrylate) with 0.5% silanated silica nano particles as fillers. In both the groups, the flexural strength decreased with increase in filler concentration. Surface hardness was highest in the PMMA group with non-silanated nano particles as fillers at 5% concentration. In both groups the surface hardness improved with an increase in filler concentration. ANOVA and TUKEY's HSD test were used. $P<0.05$ was considered statistically significant.

Conclusion
Lower concentrations of surface-treated silica nanoparticles should be used as fillers to enhance the flexural strength of commercially available heat polymerized acrylic resin.

Keywords
polymethyl methacrylate, silanated silica nano particles, nanofillers.

INTRODUCTION

Complete denture prosthesis made from polymethyl methacrylate (PMMA) has seen more chances of fracture clinically, either due to flexural fatigue or impact forces of mastication. Repetitive masticatory load results in distribution of cracks, weakening of the base of the denture and its eventual fracture.[1,2] Various attempts have been made to improve the impact strength and flexural strength of denture base acrylic resin. Properties of polymer were modified by adding specific fillers distributed at a nano-metric level inside the polymer matrix. These nanostructured materials possess novel properties, such as stiffness and high thermal stability. Nanoparticles have commonly known

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intrinsic characteristics related to their composition, shape, and size, as well as their ability to enhance the existing properties of polymers.[3]

Another method to change mechanical properties by adding small particle size of silica filler treated with hydrophobic surface to gain a large surface area. Addition of accurate concentration of filler to obtain clinically acceptable mechanical properties. Very low volumes of nano-sized filler (1 to 5 volumes %) considerably improves mechanical properties. Silica glass fibres have been shown to enhance the mechanical properties of acrylic denture base resins. Silica should be silanized by a coupling agent to assure the interfacial adhesion between the filler and matrix. There is no consensus regarding the accumulation of silica to the acrylic resin for effective use in denture bases.[4]

Hardness is another important parameter that necessities to be evaluated when testing the mechanical properties. Acrylic resins become more resistant to scratching and abrasion with increasing hardness. The purpose of this study was to evaluate the effect of surface-treated silica nanoparticle fillers on the flexural strength and surface hardness of conventional heat polymerized acrylic resin.

MATERIALS AND METHODS

Ethical clearance was obtained from institutional Ethical committee (Reference No: ABSM/EC-34/2017.)

Fabrication of metal dies and specimen:

Rectangular dies of dimension 60mm×10mm×3.3mm for testing flexural strength (Fig. 1) and 30mm×10mm×3mm for testing surface hardness (Fig. 1) were fabricated in metal.

The metal bars were invested in standard dental flasks to obtain the molds for testing flexural strength and surface hardness. Acrylic testing bars were made from commercially available heat polymerized acrylic resin (Trevalon, Dentsply, India). The polymer and monomer were mixed in a standardized ratio according to manufacturer's instructions. The silica nanoparticles, both silanated and non silanated (Fig. 2.) were incorporated according to percentage by weight.

A total of 270 acrylic resin bars were prepared in two batches of 135 each, one with the standardization for flexural strength (Fig. 3) and another for surface hardness (Fig. 4). The control group of each batch had 15 samples, which were fabricated using heat polymerized acrylic resin without any nano particles. Groups A and B constituted 60 samples each of acrylic resin with silanated and non-silanated silica respectively. Both groups had four sub-groups a, b, c, d of 15 samples each with 0.5%, 1%, 2.5% and 5% weight of silica nanoparticles respectively. The samples were flasked by compression molding technique using conventional method of acrylisation.

Testing of samples:

A) Flexural strength: The specimens were placed on universal testing machine (UTM) for a three-point bending test at a cross head speed of 5mm/min (Fig. 5). Load was applied using a centrally located rod until fracture occurred. The load applied at the time of fracture was noted.

The flexural strength of the specimen was calculated by standard equation,

$S= 3FL/2bd^2$

Where,

F= Exerting force at the middle of the specimen

L= Distance between two supports, set at 50mm.

b= Width of the specimen

d= Thickness of the specimen

B) Surface hardness:

The specimens were polished with the help of silicon carbide paper. The surface hardness was determined using a digital Micro Vickers Hardness tester (Fig. 6). 50-gram force was applied for 10 seconds. Indentation time on three distinct points was noted. The Micro Vickers Hardness was calculated digitally and the average value at the three points was considered for the specimen.

RESULTS

Descriptive statistics of quantitative variables were presented as mean, standard deviation and confidence intervals. ANOVA was employed for comparison of flexural strength and surface hardness between different treatment groups. TUKEY's HSD test was used for comparison of mean flexural strength and

surface hardness between different concentrations in each treatment group. $p<0.05$ was considered to be statistically significant. The data were surveyed using IBM SPSS Statistics, Version 24 (Armonk, NY: IBM Corp)

Table 1. compares the flexural strength between different groups (Silanated, Non- Silanated and Control) in each subgroup (0.5%, 1%, 2.5% and 5%) shows that the mean difference between control and non silanated group were statistically significant ($p<0.001$). Acrylic resins with lower concentrations of silica nanoparticle fillers exhibited greater flexural strength.

The mean difference between the acrylic resins with various silica fillers (i.e. silanated, non silanated and control) at different concentrations of filler particles (0.5%, 1%, 2.5%, 5%) are given in Table 2. There was no statistically significant difference ($p>0.05$) in the flexural strength in between the PMMA reinforced with silanate, non-silanated and those without silica nanofillers 0.5% and 1% filler concentration. However, significance was noted at 2.5% and 5% of filler concentration between control and silanated group ($p<0.001$). PMMA without silica nanofillers showed lower flexural strength as compared to PMMA with non- silanated silica nanofillers at 2.5% concentration ($p<0.001$).

Table 3. shows comparison of surface hardness of acrylic resin with silanated and non-silanated silica nano fillers at various percentage concentrations (0.5, 1, 2.5, 5). Flexural strength of acrylic resin with silanated fillers decreased with increase in the filler concentration ($p<0.001$), Surface hardness was seen to decrease with an increase in the filler concentration in PMMA with non silanated filler particles ($p<0.001$).

With increase in filler concentration, the surface hardness of the reinforced material increased.

Table 4. Compares mean surface hardness between PMMA reinforced with Silanated and non-silanated silica nanofillers at different concentrations (0.5%,1%,2.5%,5%). At 0.5% filler concentration, mean surface hardness for silanated, non silanated and control group were 18.08kg/mm^2, 19.98 kg/mm^2 and 19.30 kg/mm^2 respectively. PMMA with silanated silica nanofiller showed decreased surface hardness compared to control at 0.5% filler concentration. PMMA with non silanated silica nano filler showed surface hardness close to control group and was found to be statistically highly significant ($p<0.001$). For 1% filler concentration the mean surface hardness for silanated and non silanated filler reinforced PMMA was 17.78 kg/mm^2 and 21.22 kg/mm^2 respectively. At 2.5% filler concentration the mean surface hardness for silanated and non silanated filler reinforced PMMA was 18.61 kg/mm^2 and 21.48 kg/mm^2 respectively. Similarly, for 5% filler concentration the mean surface hardness for silanated and non silanated filler reinforced PMMA was 20.64 kg/mm^2 and 21.89 kg/mm^2 respectively. In filler concentrations of 1% and 2.5% the surface hardness of PMMA decreased for the silanated filler reinforced PMMA and increased for the non silanated filler reinforced PMMA when compared to the control group (PMMA not reinforced with nanofillers (19.30)). All of the above showed great statistical significance. ($p<0.001$)

DISCUSSION:

Fatigue or impact forces are the main causes for clinical failure of PMMA dentures. Flexural fatigue usually manifests as midline fracture due to intensity of stress around micro cracks. Research has highlighted on modifying the composition or reinforcing the PMMA with stronger materials.[1,2,5]

Flexural strength of silanated group with 0.5% nanofillers showed the highest mean of 99.58MPa, and control group showed a mean of 91.17MPa. As the filler concentration increased, the strength of the reinforced material decreased. A study conducted by Da Silva et al,[4] on microwave heat-cured resin showed similar results where the flexural strength decreased with increase in filler concentration. The mean flexural strength of non silanated group was similar to that of control group (91.16MPa) (Table 1). These findings were in line with that of Cevik et al,[3] who found that silica reduced the strength of the acrylic resin at higher concentrations. Salman et al,[6] however reported contradictory results where the strength of the reinforced material was found to increase with higher concentration of silica nanofillers. Studies[3,4,6] conducted on the effect of surface treated silica nanoparticles, found that silica fillers in lower concentration proved to be better reinforcing.

Various factors such as sample preparation, shape, dimensions, testing techniques, homogeneity of the mixture, and method of incorporation of nanoparticles may affect the flexural strength. The dispersion of

particles in the mixture is critical because it could aggregate and agglomerate in the matrix. These agglomerates could act as stress concentrating centres and have detrimental effect on the reinforced material.[8]

Dental prostheses fabricated, from acrylic resins, have low surface hardness can be damaged by mechanical roughening the surface, brushing, favouring plaque retention, thereby compromising the aesthetics appearance and longevity of dental prosthesis.[10] Unlike flexural strength, the surface hardness increased with increase in filler concentration. This wasn't in agreement with the study conducted by Da Silva et al,[4] where the surface hardness decreased with increase in filler concentration. A study by Fatihallah[7] showed a higher value for surface roughness for PMMA incorporated with 5% silanated silica nanofillers. This was concurrent with the findings in the present study. The difference in surface hardness in the present study from other studies in the literature may be because the particles of silica varies in roughness than that of acrylic denture base resin. The size of the nano particles as well as the silane coupling agent used should be taken into consideration. The distribution of hard silicon dioxide particle randomly in acrylic denture base particles within the matrix of the specimen can also be an adjunct to variation in surface hardness values.

TABLES

Table 1- Comparison of flexural strength between acrylic resin without nanofillers and those with silanated and non- silanated silica nanofillers at percentage concentrations of 0.5%,1%, 2.5% and 5%.

Subgroup	Group	N	Mean of Flexural Strength (kg/mm²)	Std. Deviation	ANOVA	
					F	p-value
0.5%	Silanated	15	99.580	11.484		
	Non- silanated	15	91.167	13.872		
	Control	15	91.173	12.768	2.177	0.126**
1%	Silanated	15	88.193	9.719		
	Non- silanated	15	88.467	9.207		
	Control	15	91.173	12.768	0.357	0.702**
2.5%	Silanated	15	58.653	9.292		
	Non-silanated	15	61.200	12.590		
	Control	15	91.173	12.768	36.086	<0.001*
5%	Silanated	15	47.373	9.235		
	Non- silanated	15	52.540	10.656		
	Control	15	91.173	12.768	71.251	<0.001*

*p< 0.001- Significant; **p>0.05 -Non-Significant

Table 2- Mean flexural strength within acrylic resin with silanated and non-silanated silica nano fillers at various concentrations.(0.5%,1%,2.5% and 5%.)

Group	N	Mean flexural strength(kg/mm²)	Std. Deviation	F	P
Silanated 0.5%	15	99.580	11.484		
1%	15	88.193	9.719		
2.5%	15	58.653	9.292		
5%	15	47.373	9.235	90.412	<0.001*
Non silanated 0.5%	15	91.167	13.872		
1%	15	88.467	9.207		
2.5%	15	61.200	12.590		
5%	15	52.540	10.656	41.024	<0.001*

*p< 0.001-Very Highly Significant; **p>0.05 -Non-Significant

Table 3 -Comparison of surface hardness between acrylic resin with silanated and non-silanated silica nano fillers at various percentage concentrations .(0.5%,1%,2.5% and 5%.)

Subgroup	N	Mean of surface hardness (kg/mm²)	Std. Deviation	F	p
0.5% Silanated	15	18.080	1.116		
Non silanated	15	19.980	0.478		
Control	15	19.307	1.257	13.680	<0.001*
1% Silanated	15	17.780	1.201		
Non silanated	15	21.227	1.335		
Control	15	19.307	1.257	27.937	<0.001*
2.5% Silanated	15	18.613	1.216		
Non silanated	15	21.480	1.578		
Control	15	19.307	1.257	18.149	<0.001*
5% Silanated	15	20.640	0.827		
Non silanated	15	21.893	1.907		
Control	15	19.307	1.257	12.763	<0.001*

*p< 0.001- Significant; **p>0.05 -Non-Significant

Table 4- Mean surface hardness of acrylic resin at various concentrations of silanated and non silanated nano fillers. (0.5%,1%,2.5% and 5%.)

Group		N	Mean surface hardness(kg/mm²)	Std. Deviation	F	P
Silanated	0.5%	15	18.080	1.116		
	1%	15	17.780	1.201		
	2.5%	15	18.613	1.216		
	5%	15	20.640	0.827	20.525	<0.001**
Non- silanated	0.5%	15	19.980	478		
	1%	15	21.227	1.335		
	2.5%	15	21.480	1.578		
	5%	15	21.893	1.907	5.005	0.004

*p< 0.001-Highly Significant; **p>0.05 -Non-Significant

Conclusion

PMMA with 0.5% silanated nanofiller improved the flexural strength marginally. There was no significant difference in strength of non-silanated filler group. Rise in percentage of filler particle contributed to reduction in flexural strength of the acrylic resin. There was significant improvement in the surface hardness of acrylic reinforced with silanated and non-silanated fillers as compared to the control. Thus, silica nanoparticles in lower concentrations can be used as a reinforcing agent. More studies need to be conducted to evaluate any difference in surface treated and untreated particles.

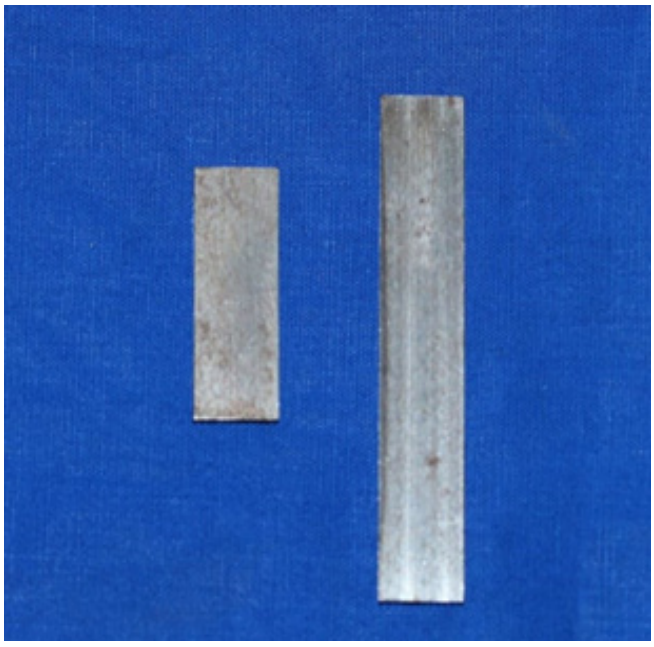

Figure 1. Metal die

Figure 2. Silica nanoparticles

Figure 3. Acrylic bars for testing flexural strength (60mm×10mm×3.3mm)

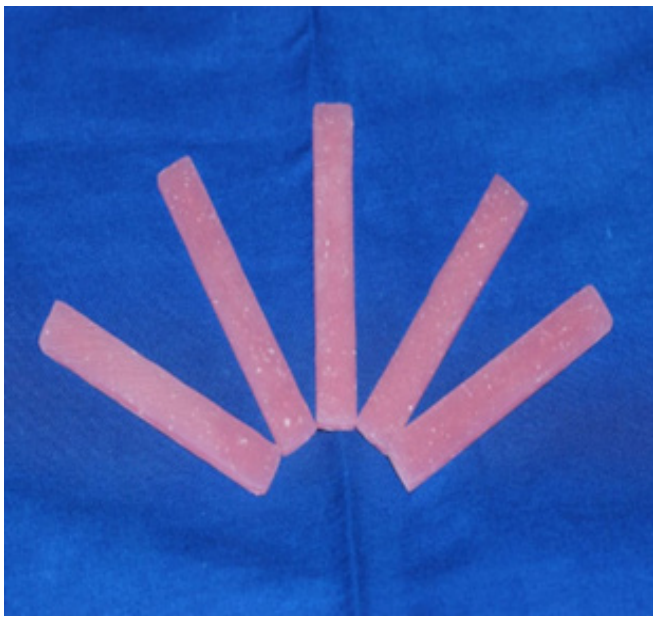

Figure 4. Acrylic bars for testing surface hardness (30mm×10mm×3.3mm)

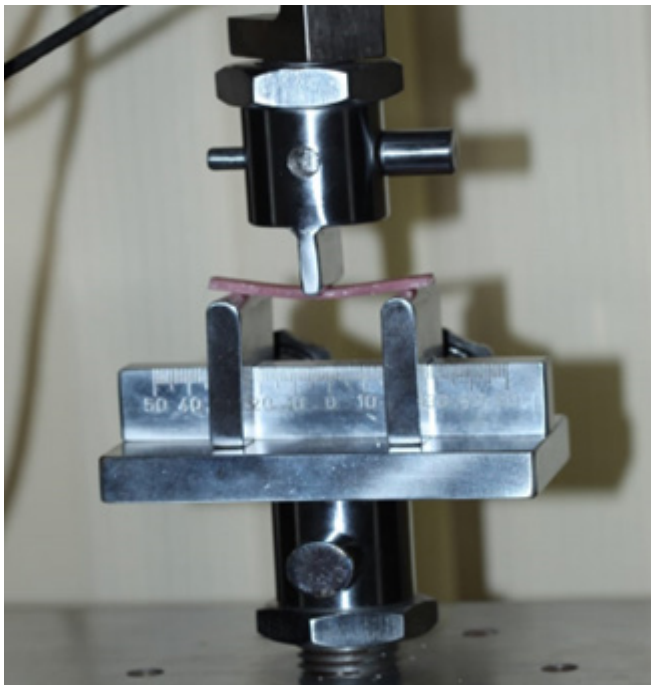

Figure 5. Load application to check flexural strength on universal testing machine

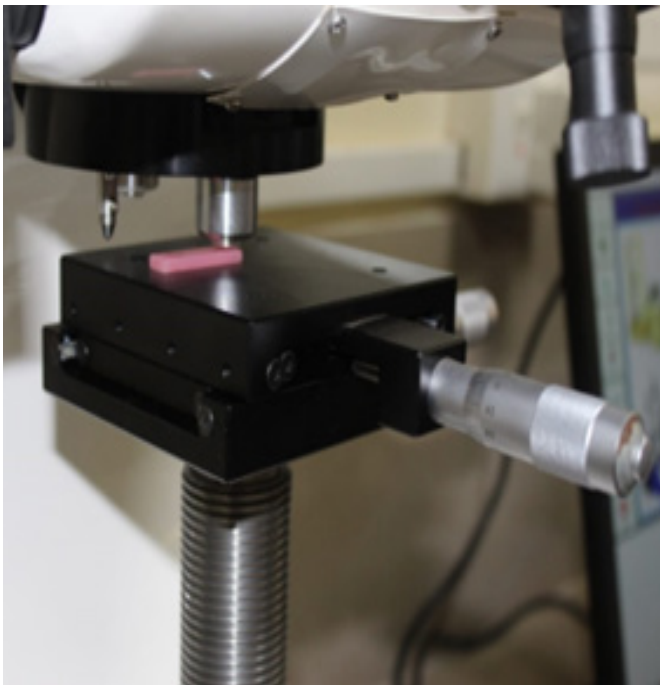

Figure 6. Surface Hardeness

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