



RESEARCH ARTICLE

AN IN VITRO EVALUATION OF EFFECT OF DIFFERENT CONCENTRATIONS OF  
POLYHEDRAL OLIGOMERIC SILSESQUIOXANE NANOFILLER ON  
FLEXURAL STRENGTH OF DENTURE BASE RESIN

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ABSTRACT

**Purpose:** The purpose of this study was to evaluate the effect of three different concentrations (1wt%, 3wt % and 5wt %) of MA-POSS (methacryl polyhedral oligomeric silsesquioxane) on flexural strength of heat cured denture base resin (PMMA).

**Material and Methods:** Total 40 test specimens were made of heat cured denture base resin (DPI, Dental Products of India) with dimensions of 65 mm x 10 mm x 3 mm. Methacryl POSS (MA-POSS) was added in 1wt%, 3wt% and 5 wt% concentrations by physically blending in polymer matrix. All the specimens were stored in artificial saliva at 37<sup>0</sup> C for 10 days in a universal incubator and tested in universal testing machine at cross head speed of 0.5mm/min.

**Results:** The mean flexural strength of control group specimens was 72.25 Mpa which increased to 85.17 Mpa on reinforcement with 1 wt % POSS and further increased to 99.85 Mpa on reinforcement with 3 wt% POSS. However the flexural strength declined to 50.45 Mpa on reinforcement with 5wt% POSS

**Conclusion:** Concentration of POSS upto 3 wt % improved flexural strength of denture base resin however when concentration increased to 5 wt % the strength decreased drastically.

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INTRODUCTION

Polymethyl Methacrylate Resin since its discovery as a denture base material in 1937 by Dr Walter Wright, was found to fulfil the requirements of the ideal denture base material. Despite its favourable working characteristics, ease of fabrication, acceptable aesthetics, good colour stability, ease of processing, accuracy of fit, stability in oral environment and uncomplicated procedure of fabrication; fracture of dentures are often encountered clinically. In earlier studies, fracture rate was reported to be 64% and 68 % (Hargreaves, 1969). These fractures may occur inside due to excessive bite force, improper occlusal plane, highfrenal attachment, lack of balanced occlusion, poor fit or limitations in denture base material or outside the mouth due to expelling the denture from the mouth while coughing, or simply dropping it. When in function, midline fracture is the result of flexural fatigue failure caused by cyclic deformation of the base, and is more likely to occur because flexure of the denture base occurs along the midline (Darbar et al., 1994; Stafford and Smith, 1970). Various modifications have been suggested to overcome these shortcomings, which included plasticization

(Jagger et al., 1999), copolymerization with rubber (Stafford et al., 1980), use of crosslinkingagents such as polyethylene glycol dimethacrylate and reinforcement with metal strengthener's (Ruffino, 1985; Vallittu and Lassila, 1992). Last two decades has seen a dramatic increase in the use of fiber-reinforced composites with different fibers such as glass (Solnit, 1991; Vallittu, 1999; Uzun et al., 1999; John et al., 2001; Aydin et al., 2002), aramid (Uzun et al., 1999; John et al., 2001), carbon (Uzun et al., 1999; Schreiber, 1971; Manley et al., 1979; De Boer et al., 1984), nylon (John et al., 2001; Kelly, 1969), whiskers (Grant and Greener, 1967) and ultra-high modulus polyethylene fibers (Uzun et al., 1999; Dixon and Breeding, 1992; Ramos et al., 1996) being incorporated in the acrylic resin. More recently, nanomaterials are used as fillers to improve strength of the materials. Polyhedral Oligomeric Silsesquioxanes (POSS) are nanostructures consisting of silica cage core, as well as other organic functional groups attached to the corners of the cage (Sellinger and Laine, 1996; Ayandele et al., 2012). The reinforcement with specific amount of POSS can play a very important role in determining the mechanical properties of POSS-polymers. Studies have shown that POSS concentrations less than 10 wt % showed improved mechanical properties like fracture toughness, impact strength, polymerization shrinkage etc of Methyl methacrylate as well as Bis acryl composite resins.

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However the effect of POSS on flexural strength of PMMA denture base resin has not yet been documented. The purpose of this study was to evaluate the effect of three different concentrations (1wt%, 3wt % and 5wt %) of MA-POSS (methacryl polyhedral oligomeric silsesquioxane) on flexural strength of heat cured denture base resin (PMMA).

*“The null hypothesis was that flexural strength of denture base resin would not be affected by adding POSS.”*

## MATERIALS AND METHODS

Total 40 test specimens were made of heat cured denture base resin (DPI, Dental Products of India) with dimensions of 65 mm x 10 mm x 3 mm complying with the ADA specification no. 12 for denture base polymers.

### Fabrication of standard test specimen

Three brass dies (65 mm x 10 mm x 3 mm) coated with petroleum jelly were used to prepare dental stone molds using the conventional flasking technique. Polymer: monomer ratio for each specimen was 3:1 by volume in all groups. MA-POSS (Hybrid plastics, USA) was added in 1wt%, 3wt% and 5 wt% concentrations by physically blending in polymer matrix. Samples were packed and bench cured for 30 minutes and curing was done in an acryliser with step-up curing cycle at 73±1°C for one and half hour followed by 100°C for 30 minutes in a curing unit. After bench cooling for 30 minutes, samples were removed and finished with abrasive paper.

### Grouping of specimens

All 40 specimens were divided in four groups of 10 specimens each. Group without POSS was named as Group A. Specimens with 1 wt%, 3 wt% and 5 wt % POSS were grouped as Group B, Group C and Group D respectively.

### Storage of specimens

All the specimens were stored in artificial saliva at 37°C for 10 days in a universal incubator.

### Testing of prepared specimens

Width and thickness of each specimen was measured with a digital vernier caliper. The specimens were tested with 3- point bending test on Instron universal testing machine (Star Testing System, India. Model No. STS 248, Accuracy of the machine: ±1%). Cross head speed : 3mm /minutes. The specimens were placed on jigs that were 50 mm apart (L) and then loaded at the center until fracture occurred. Maximum load at fracture was recorded and converted into flexural strength using the formula:

$$\text{Flexural strength } \sigma = 3x F x L/2 x b x d^2$$

where F is the maximum load applied, L is span between two supports, b is width of the specimen and d is the thickness of the specimen. After that the values were compared.

## RESULTS

The mean flexural strength of control group specimens was 72.25 Mpa which increased to 85.17 Mpa on reinforcement

with 1 wt % POSS and further increased to 99.85Mpa on reinforcement with 3 wt% POSS. However the flexural strength declined to 50.45 Mpa on reinforcement with 5wt% POSS. (Table 1) An analysis of the difference in flexural strengths was performed with one way Analysis of Variance (ANOVA) for all the specimens in all four groups. The results were found to be highly significant with the P value of 0.0003. (Table 2)

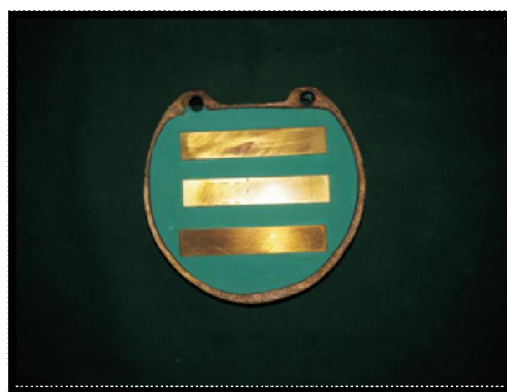
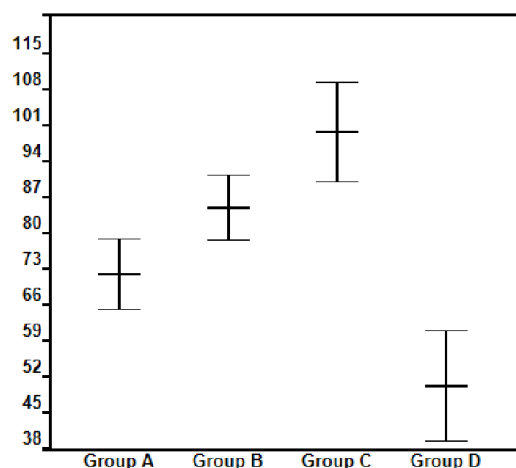
Table 1.

comparison of mean flexural strength				
	Group A	Group B	Group C	Group D
1	65.46	72.33	99.10	57.23
2	66.73	87.91	98.34	44.00
3	69.38	79.89	89.44	57.91
4	80.26	88.12	102.15	41.94
5	76.93	86.88	88.40	39.88
6	83.67	91.22	90.11	64.68
7	79.38	78.50	112.34	30.28
8	66.45	89.18	100.66	58.21
9	71.24	93.48	119.48	50.86
10	63.01	84.20	99.50	59.58
n	10	10	10	10
$\bar{X}$	72.251	85.171	99.952	50.457
s	7.243	6.490	9.913	10.957
$\bar{X}_{ave}$	76.958			

Table 2.

source	df	SS	MS	F	P-value
treatments	3	13206.363	4402.121	56.2746	0.0003
error	36	2816.125	78.226		
total	39	16022.488			

Graph 1



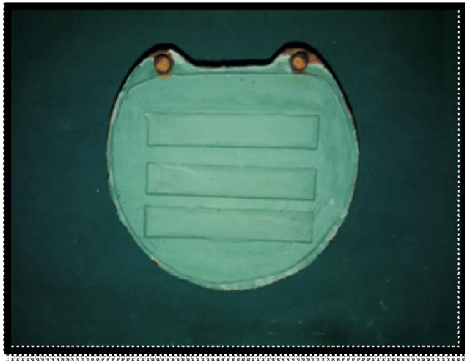


Fig. 1. Brass dies and mould fabrication in brass flask



Fig. 2. Specimen fabrication



Fig. 3. Verification of dimensions



Fig. 4. Specimens

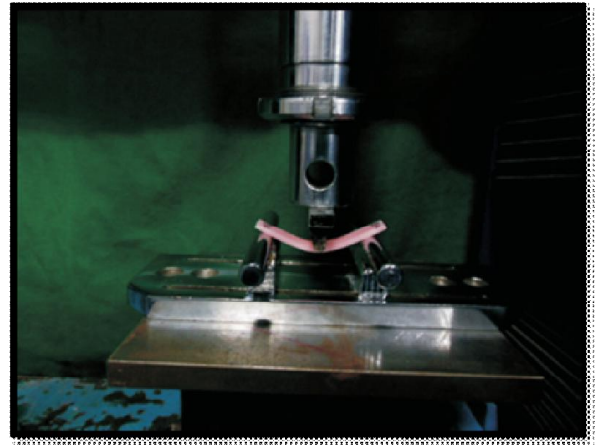


Fig. 5. Testing of specimens

## DISCUSSION

The present study shows that flexural strength improved with the addition of MA-POSS at 1wt % and 3 wt % concentrations. However there was a drastic decline in flexural strength values at 5 wt % concentration. These results were found to be in agreement with the study conducted by Xiaorong Wu *et al.* (2010) where they used 2wt %-5wt % concentrations of POSS as reinforcement for composite resins, in that with 2wt% POSS there was 15% increase in flexural strength and also increase in other properties. Whereas in case of 5wt% POSS Flexural strength declined from 87Mpa to 75 Mpa. This alter in the mechanical properties might be due to the fact that, POSS interacts covalently via free radical polymerization (FRP) or via atom transfer radical polymerization (ATRP) with the polymer matrix. Kuo *et al.* (2011) showed that more soluble the POSS better is the reinforcement due to which multifunctional POSS monomer (MA-POSS) is homogeneously dispersed in the methacrylate network as a polyhedral junction at low POSS concentrations i.e 1-3wt % resulting in better hybrid materials with improved mechanical properties (Kuo and Chang, 2011). As the amount of POSS concentration increased, there was a decrease in the flexural strength values of the PMMA this is because POSS concentration above 5% tends to aggregate and crystallize in a polymer matrix, and the more aggregation, the worse the mechanical properties. Another reason is that, probably increased POSS concentration leads to phase separation between the POSS-MA and the methacrylate resin, this act as a structural deficiency in the formulated resin and thus decreasing mechanical properties (Ayandele *et al.*, 2012; Kuo and Chang, 2011). This in vitro study may not duplicate the stress environment and failure mode seen clinically. Clinical studies have shown that the fracture of denture base resins occurs with accumulative effect, so cyclic loading may provide additional valuable data. Further research is needed to evaluate the effect of aging on these reinforced denture base materials as well as effect of POSS-MA on other mechanical properties of interim materials before clinical application.

## Conclusion

Within the limitations of this study, the results suggest that addition of upto 3 wt % POSS improved the flexural strength of the denture base resin but as the concentration was increased to 5wt % the strength deteriorated rapidly. Further research is

suggested to evaluate effect of POSS on other mechanical properties of denture base resins.

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