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# Evaluation of the Effect of Silanized Zirconia Nanofillers on the Flexural Strength and Surface Hardness of Heat Cure Poly-Methyl Methacrylate Resin: An In Vitro Study

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## ABSTRACT

**Aim**: This study aimed to evaluate the effect of silanized zirconia nanofillers on the flexural strength and surface hardness of heat cure poly-methyl methacrylate resin.

**Methods and Material:** A total of 60 specimens of heat cure acrylic resin were fabricated and divided into two groups (n=30), Group A and Group B for testing flexural strength and surface hardness, respectively. Again, Group A and Group B are divided into two subgroups, Subgroup 1 (n=15): Control group with unreinforced heat cure acrylic resin and Subgroup 2 (n=15): Heat cure acrylic resin reinforced with 0.25% Silanized zirconia ($ZrO_2$) nanofillers. Group A1 and A2 were subjected to flexural strength by using a universal testing machine, and Group B1 and Group B2 were subjected to shore D surface hardness test.

**Results:** Mean flexural strength for Group A1 and A2 in (M Pa) are 89.753, 88.304, respectively. Mean surface hardness values for Group B1, and B2 are 90.04 and 91.04, respectively. The Student t-test analysis showed a statistically insignificant difference for both flexural strength ($P= 0.825$) and surface hardness ($P=0.802$).

**Conclusions:** The specimens of heat-cure acrylic resin reinforced with 0.25% silanized $ZrO_2$ nanofiller provided no superior flexural strength and surface hardness than those with conventional heat cure acrylic resin.

**Keywords:** Heat cure acrylic resin, Silanized Zirconia Nanofiller, Flexural strength, Shore-D surface hardness.

## INTRODUCTION

Heat cure Polymethyl methacrylate (PMMA) resin is the most commonly used denture base material for the fabrication of complete dentures because of various advantages such as good esthetics, ease of manipulation etc. However, heat cure acrylic resin has poor mechanical properties. [1] Several studies conducted to enhance the properties of denture base resin by incorporating fillers, fibers, nanofillers. [2 -5] Recently, zirconia nanofillers are commonly used because of their biocompatibility and white color, which makes it less likely to alter the esthetic properties. [6] To enhance the adhesion between the resin matrix, silanized $ZrO_2$ nanofillers are used in this study. This study aimed to evaluate the effect of silanized $ZrO_2$ nanofillers on the flexural strength and surface hardness of heat cure acrylic resin.

## MATERIALS AND METHODS

Materials: Heat cure acrylic resin (Lucitone 199, Dentsply, Inc.USA).

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Silanized $ZrO_2$ nano powder (Size 30-50nm, purity 99.9%, Nano labs Pvt ltd).

Preparation of stainless-steel dies:

For this study, stainless steel dies measuring 65mm x 10mm x 3mm for flexural strength, and 30mm x 10mm x 3mm for surface hardness were fabricated according to American dental association specification no.12.

Grouping of specimens:

Total of 60 specimens was fabricated and divided into two groups.

Group A (n=30) specimens for testing flexural strength
Subgroup A1 (n=15): Unreinforced heat cure acrylic resin.
Subgroup A2 (n=15): Heat cure acrylic resin reinforced with 0.25% silanized $ZrO_2$ nanofillers.
Group B (n=30) specimens for testing surface hardness.
Subgroup B1 (n=15): Unreinforced heat cure acrylic resin.
Subgroup B2 (n=15): Heat cure acrylic resin reinforced with 0.25% silanized $ZrO_2$ nanofillers.

A total of sixty samples (n=60) were prepared by using these stainless-steel dies. The metal dies were coated with cold mold seal (Dental products of India, Mumbai) and allowed to dry. Type III dental plaster (Neelkanth stone plaster, Rajkot) was mixed according to the manufacture instructions and added to the lower member of the flask and vibrated to prevent air bubble formation. The metal dies were inserted about half of their depth, placed parallel to each other on the flat surface, and the dental plaster was allowed to dry. [Figure1] Later, the lower half was coated with cold mold seal, allowed to dry and then the upper half of the flask was placed and filled with type III dental plaster, vibrated, and the lid of the flask was placed. It was allowed to sit for one hour, and the flask was separated. The stainless-steel dies were retrieved from the stone to create the mold space for the packing of acrylic resin.

Addition of Silanized $ZrO_2$ nanofillers to monomer:

25mg of silanized $ZrO_2$ nanofillers were weighed using an electronic balance and added to 100ml of monomer to make 0.25%. The nanoparticles were dispersed in the monomer by using a probe sonication apparatus for 3 minutes to break them into individual nanoparticles. The monomer with nanofillers was immediately mixed with acrylic powder to reduce the possibility of particle aggregation.

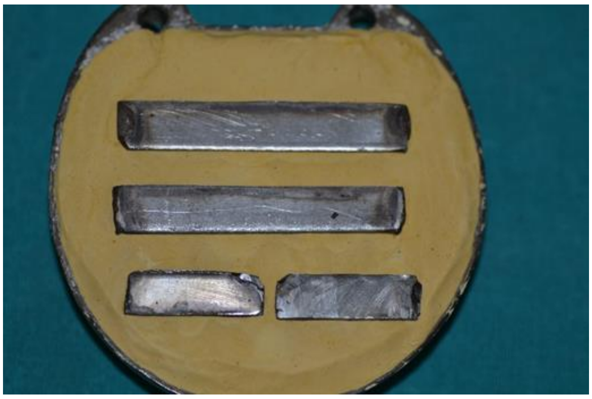

Fig 1: Stainless steel dies invested in dental stone.

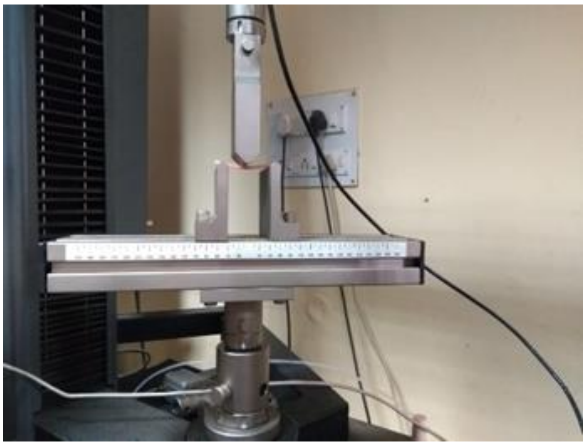

Fig 2: Testing of specimen for flexural strength.

Fig 3: Testing of specimen for surface hardness.

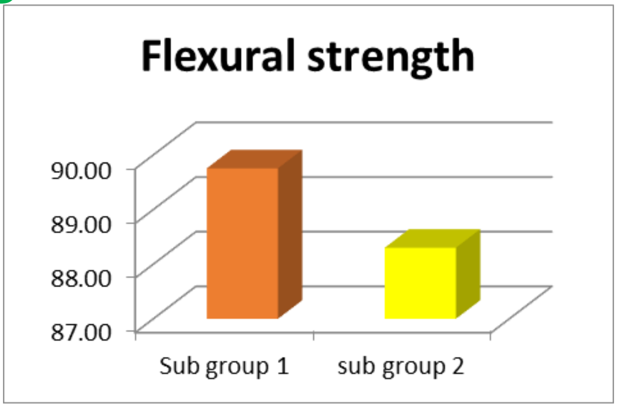


Fig 4: Mean and Standard deviation values of flexural strength.

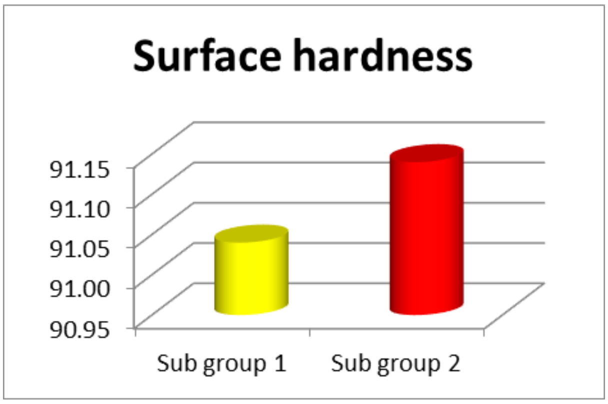


Fig 5: Mean and Standard deviation values of surface hardness.

Table 1: Mean and standard deviation values for flexural strength.

| Flexural strength | Mean | SD |
|---|---|---|
| Sub group A1 | 89.75 | 18.69 |
| Sub group A2 | 88.30 | 17.12 |
| t value | 0.22 | |
| P value | 0.82586773 NS | |

Table 2. Mean and standard deviation values for surface hardness.

| Surface hardness | Mean | SD |
|---|---|---|
| Sub group B1 | 91.04 | 0.84 |
| sub group B2 | 91.14 | 1.28 |
| t value | 0.25 | |
| P value | 0.80283268 NS | |

Specimen preparation:

The cold mold seal was applied to the mold space on all the surfaces and allowed to air dry. The mold space was packed with unreinforced heat cure acrylic resin in the ratio of 3:1 polymer and monomer for preparing Group A1 and Group B1 specimens. For preparing Group A2 and Group B2, the mold space was packed with heat cure acrylic resin reinforced with 0.25% salinized $ZrO_2$ nanofillers.

A clean, dry, airtight porcelain mixing jar was taken, and it is applied with cold mold seal on all the sides of porcelain jar. A pipette was used to transfer the required volume of monomer to porcelain jar. Both the monomer and polymer were mixed with a clean dry wax spatula, and packing was done in the dough stage.

The flasks were bench cured for 30 minutes and polymerized by using a long curing cycle with 74°c for 8 hours. After completion of curing, the specimens were bench cooled, and then de-flasking was done. Each subgroup of samples was finished using various grits of sandpapers 180, 220, 400, followed by polishing with pumice.

Testing of samples for flexural strength:

A three-point bending test using a Universal Testing Machine was done, whereby the specimen was centrally loaded at a crosshead speed of 5mm/min over a two-point support span set at a distance of 50mm. Specimens will deflect until rupture occurs[Figure 2]. The maximum load required for rupture were recorded as its flexural strength.

The flexural strength was calculated by using the following formula

$FS=3PL/2bd^2$

FS=flexural strength in MPa

P=Peak load applied in N

L=span (distance between support) in mm

b=specimen width in mm

d=specimen thickness in mm

Testing of samples for surface hardness:

According to the ADA specification, Shore D hardness tester was used to test the surface hardness of specimens. The instrument consists of a bluntly pointed tip with a diameter of 0.8mm; the indenter was attached to the digital scale the readings range from 0-100 units. The indenter was pressed firmly and quickly on the specimen, and the maximum reading is taken as shore D hardness, measurements were directly taken from the digital scale readings. Three measurements were recorded from different areas of each specimen, and an average of these readings was taken.

After examining the samples and following data collection, statistical analysis was done.

## RESULTS

The results were analyzed using Student t-test for both flexural strength and surface hardness. Mean and standard deviation values of Group A and Group B are presented in Table 1 & Table 2, respectively. The comparative mean values of the flexural strength and surface hardness of Group A and Group B are represented in fig.4 & 5, respectively.

Student t-test showed that t value is 0.22 and 0.25 for flexural strength and surface hardness respectively. Since $P > 0.05$, there is insignificant difference in the flexural strength and surface hardness of the control group and test group.

## DISCUSSION

The acrylic resins were used in the dental profession since 1946, 98% of all the denture bases were constructed from methyl methacrylate polymers or copolymers. Over a period of time, fracture of the denture in the mouth occur due to low flexural strength.

The addition of Fillers, Fibers, Nanofillers increase the properties of heat cure acrylic resin but compromise esthetics. The incorporation of zirconia had significant beneficial effects on the mechanical properties and didn't compromise the esthetic appearance, unlike its metal fillers like aluminium, copper and silver. [7]

In this study, Silanized $ZrO_2$ nanofillers were added in the concentration of 0.25% to heat cure acrylic resin. The percentage of nanofiller used to reinforce the acrylic resin is another important factor affecting mechanical properties. The filler particles should disperse homogeneously into the resin matrix without interrupting its continuity to reinforce the mechanical properties. Schulz et al. reported that an increase in filler concentration doesn't necessarily lead to an increase in strength. Because higher filler fractions create more defects that weaken the material. [8]

The nanofillers are usually hydrophilic in nature and possess high surface energy. However, the hydrophobic polymer does not bond with the filler due to the difference in surface energies. In order to improve the adhesion between the resin polymer and filler, surface modification was done using a silane coupling agent. Silanization is simple and effective; the concentration of the silane and reaction time must be carefully controlled to prevent forming thick polymerized silane network on the filler surface.[9] Additionally, silanization requires special equipment and routine use in dental laboratories is not possible due to difficulty in handling. So as to avoid these problems commercially available silanized $ZrO_2$ nanofillers were selected in this study.

The null hypothesis of the study accepted as the results of the survey reported that the addition of 0.25% salinized $ZrO_2$ nanofillers doesn't improve the flexural strength and surface hardness of heat cure acrylic resin. The present study revealed that reinforcement of heat cure acrylic resin with 0.25% silanized $ZrO_2$ decreased the flexural strength. This decrease in flexural strength with reinforcement of silanized $ZrO_2$ was statistically insignificant. The results are similar to previous studies, it may be due to clustering of particles within the resin, which weakened the material or it may be due to low nanofiller content. [10] The results of this study are in contrast with, Asopa et al. showed that reinforcement of high impact acrylic resin with 10% $ZrO_2$ results in increases its flexural strength. [11]

When the addition of 0.25% silanized $ZrO_2$, there was a slight increase in the surface hardness, but this increase in surface hardness was statistically insignificant. The results are consistent with Ihab study, showed that hardness value increases by adding various concentrations of nano $ZrO_2$ to heat cure acrylic resin, but statistically not significant. [12]

Our results differ from most of the previous studies, and these difference may be caused by the difference in the percentage of silanized $ZrO_2$, filler size, type of acrylic resin used and non-homogenous distribution of nano$ZrO_2$ within the resin matrix. The scanning electron microscopic studies are required to see the homogenous distribution of silanized nano$ZrO_2$ within the resin matrix.

The limitations of this study included the fact that only two mechanical properties were investigated (flexural strength and surface hardness). For future research, other properties can be investigated, and other nanofiller materials can also be used for reinforcement of the heat cure acrylic resin. Besides, the mechanical tests were done in the laboratories, not similar to the conditions inside the oral cavity. In the oral cavity, denture bases are exposed to varying forces acting in different directions. The same situation could not be simulated in this in vitro Study. In clinical conditions, a uniform thickness of denture base resin may not be 3mm, as used in this study. Further studies are required to evaluate the effects of silanized nano-ZrO2 reinforcement on the properties of heat cure acrylic resins when specimens stored in water or artificial saliva for a longer duration.

## CONCLUSION

Within the limitations of this study, it can be concluded that the specimens of heat-cures acrylic resin reinforced with 0.25% silanized $ZrO_2$ nanofiller provided no superior flexural strength and surface hardness than those with conventional heat cure acrylic resin. Further studies are needed to investigate its effect on other mechanical and physical properties with a different concentration on different materials.

### Clinical significance

Heat cure acrylic resin is commonly used material for fabrication of complete denture and partial denture in the elderly population. Fracture of the denture in the oral cavity is a common problem due to low flexural strength. So the addition of various nanofillers may enhance the properties of heat cure acrylic resin.

## CONFLICTS OF INTEREST

The authors declare they have no potential conflict of interests regarding this article.

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