

# A Comparative Evaluation of Physical Properties of Denture Base Resins with Addition of Fillers: An in Vitro Study

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## Abstract

The aim of the study was to evaluate and compare the physical properties of Heat & Chemically activated Denture Base Resin with addition of fillers. A total of 120 specimens were prepared. For evaluation of transverse strength and surface hardness of both Denture Base Resins, 80 specimens were prepared using standard mold measuring 65 mm x 10 mm x 2.5 mm, consisting of control plus experimental groups reinforced with three different fillers (5 wt% ZrO<sub>2</sub> nanoparticles, 5 wt% Al<sub>2</sub>O<sub>3</sub> nanoparticles, 2.5 wt% glass fibers). For evaluation of water sorption, 40 specimens were prepared using standard mold measuring 50 mm x 0.5 mm, consisting of control plus experimental groups reinforced with three different fillers.

**Results:** Both Denture Base Resins showed increase in transverse strength and surface hardness after addition of fillers. Highest transverse strength and surface hardness was found to be in Heat Cure and Cold Cure reinforced with ZrO<sub>2</sub> Nano Particles > Glass fiber > Al<sub>2</sub>O<sub>3</sub> Nano Particles whereas the results of water sorption were in accordance with ISO specification.

**Conclusion:** Reinforcement of both Denture Base Resins by addition of fillers can inhibit undesirable physical changes resulting from oral fluids and can prevent denture fractures by enhancing its properties.

**Keywords:** Denture Base Resin; Resin reinforcement; ZrO<sub>2</sub> Nano Particles; Al<sub>2</sub>O<sub>3</sub> nanoparticles; Glass fibers; Transverse strength; Surface hardness; Water sorption.

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## INTRODUCTION

**E**dentulism is defined as the loss of teeth which can be partial or complete and is the outcome of multi factorial process involving biological as well as non-biological factors related to dental procedures.<sup>1</sup> Conventional complete dentures are often chosen as a treatment modality because of multiple factors such as cost, treatment time and patient's unwillingness to undergo surgical



procedure and bone factors etc.<sup>2</sup>

The denture bases are subjected to compressive, tensile and shear stresses.<sup>3</sup> Some of the factors responsible for denture fracture include stress intensification, increased ridge resorption, deep incisal notching at the labial frenum and induced processing.<sup>4,5</sup> One of the factors for fracture of a denture is flexural fatigue. It occurs due to constant low and short repeated stress on a material which eventually fractures over a period of time.<sup>6</sup> This catastrophic failure results from a final loading cycle that will exceed the mechanical limits of the remaining sound portion of the material.<sup>7</sup>

To enhance the mechanical properties of DBR there is a need of structural modifications.<sup>8,9</sup> Thereby enduring structural modification, fillers like ZrO<sub>2</sub> nanoparticles, Al<sub>2</sub>O<sub>3</sub> nanoparticles and glass fibers can be incorporated into DBR. ZrO<sub>2</sub> particles have crystalline structure, transparent color and have been reported for having high mechanical properties.<sup>10</sup> The search of literature reveals that the addition of varying amount of metal fillers such as aluminum to PMMA not only gives it an advantage of increased strength but also reduces polymerization shrinkage and warpage.<sup>11,12</sup> Glass fibers are aesthetically stable and possess high degree of stiffness, toughness and strength.<sup>13</sup> It may improve the flexural strength of Denture Base Resins.

As, we know that DBR is material of choice for fabrication of denture but its longevity is hampered due to its insubstantial mechanical properties. Addition of fillers may improve the transverse strength and surface hardness of DBR. In oral conditions, it is likely that further polymerization reaction and water uptake mechanisms occur simultaneously.<sup>14,15</sup> Hence, this study was undertaken to evaluate the effect of addition of glass fibers, ZrO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles on the

transverse strength, surface hardness and water sorption of Cold Cure and Heat Cure Denture Base Resin.

## MATERIALS AND METHODOLOGY

This study was conducted in the Department of Prosthodontics and Crown & Bridge, Inderprastha Dental College and Hospital, Sahibabad, Uttar Pradesh.

The stainless steel molds of dimensions 65mm x 10 mm x 2.5 mm (Fig. 1) and dimensions 50 mm x 1.5 mm (Fig. 2) were prepared according to ADA Specification number 12 for evaluation of transverse strength, surface hardness and water sorption respectively.

Modelling wax was melted in the wax water bath. It was then poured into stainless steel mold of dimension 65 mm x 10 mm x 2.5 mm and of dimension 50 mm x 1.5 mm as per ADA Specification number 12 and was allowed to cool. After the wax solidified, the specimens were retrieved from stainless steel mold (Fig. 3 and Fig. 4) and were invested in dental flask (Fig. 5 and Fig. 6). Upon completion of the setting process, after 30 minutes, dewaxing was done.

Separating media was applied on the mould space and PMMA Cold Cure Denture Base Resin was mixed in the polymer to monomer ratio of 2.5:1 by weight in a porcelain mixing jar and fillers (5 wt% ZrO<sub>2</sub> nanoparticles, 5 wt% Al<sub>2</sub>O<sub>3</sub> nano particles, 2.5 wt% glass fibers) were added separately for each sub group and was allowed to reach the dough like consistency. The resin was placed into the mold cavity. The flask assembly was then placed into hydraulic press and was kept on bench press unit for bench curing for 30 min and then cured. Curing of specimens was done for 2 hours at 74°C followed by 1 hour at 100°C.



Fig. 1: Stainless steel mold measuring 65mm x 10mm x 2.5mm



Fig. 2: Stainless steel mold measuring 1.5 mm x 50 mm

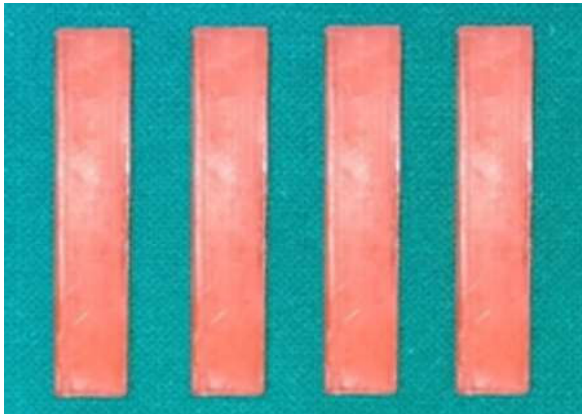


Fig. 3: Wax Mold (65mm x 10mm x 2.5mm)

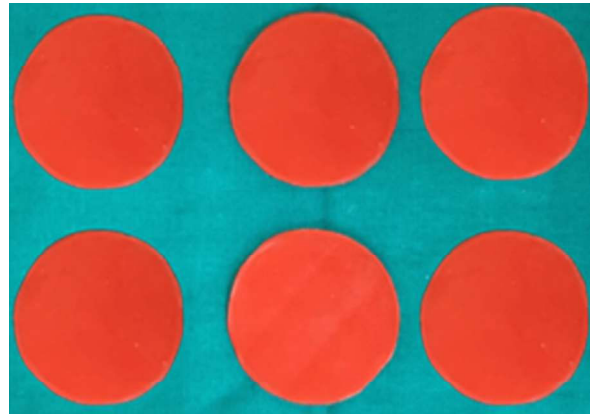


Fig. 4: Wax Mold (50mm x 1.5mm)



Fig. 5: Investment of wax pattern



Fig. 6: Investment of wax pattern

## TESTING OF SPECIMENS

### *Evaluation of surface hardness*

Hardness measurement was obtained with a Vickers hardness tester (Fig. 8). It was measured under a 20 gram load and 30 seconds penetration period.

Vickers Hardness Number=Test force/surface area of indentation.

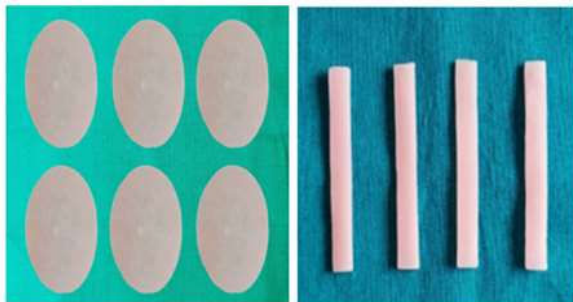


Fig. 7: Polymerized specimen



Fig. 8: Digital Vickers microhardness tester



Fig. 9: Universal testing machine



Fig. 10: Specimen stored at 37°C in incubator



Fig. 11: Electronic balance

## STATISTICAL ANALYSIS

Inferential statistical analysis has been carried out in the present study. Results on continuous measurements are presented on Mean  $\pm$  SD (Min

- Max). Significance is assessed at 5% level of significance. ANOVA with post hoc bonferroni for multiple comparisons tests has been used to find the significance of study parameters on ordinal scale between more than two groups.

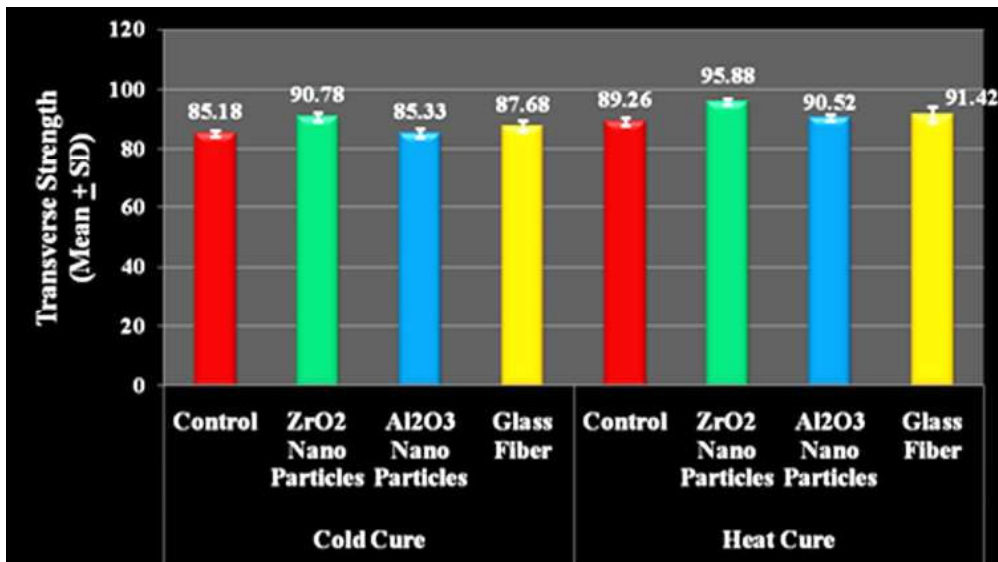
**RESULTS**

Transverse Strength, surface hardness and water

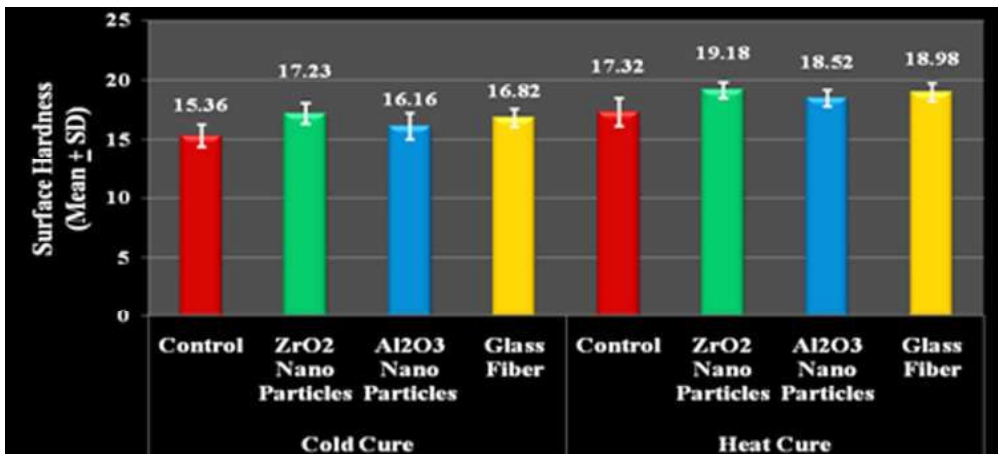
sorption of heat and cold cure resin reinforced with ZrO<sub>2</sub> nanoparticles, Al<sub>2</sub>O<sub>3</sub> nanoparticles and glass fibers.

Group	Sub Group	Transverse Strength		Surface Hardness		Water Sorption	
		Mean	SD	Mean	SD	Mean	SD
Cold Cure	Control	85.18	1.01	15.36	0.93	13.75	1.02
	ZrO2 Nano Particles	90.78	1.51	17.23	0.86	12.22	1.07
	Al2O3 Nano Particles	85.33	1.41	16.16	1.14	12.57	1.34
	Glass Fiber	87.68	1.85	16.82	0.76	13.32	0.97
Heat Cure	Control	89.26	1.30	17.32	1.19	20.58	1.56
	ZrO2 Nano Particles	95.88	1.08	19.18	0.66	17.52	0.69
	Al2O3 Nano Particles	90.52	0.99	18.52	0.67	18.22	0.70
	Glass Fiber	91.42	2.59	18.98	0.75	19.72	0.89
ANOVA (F)		25.727		11.717		50.063	
p - Value		< 0.001 (VHS)		< 0.001 (VHS)		< 0.001 (VHS)	

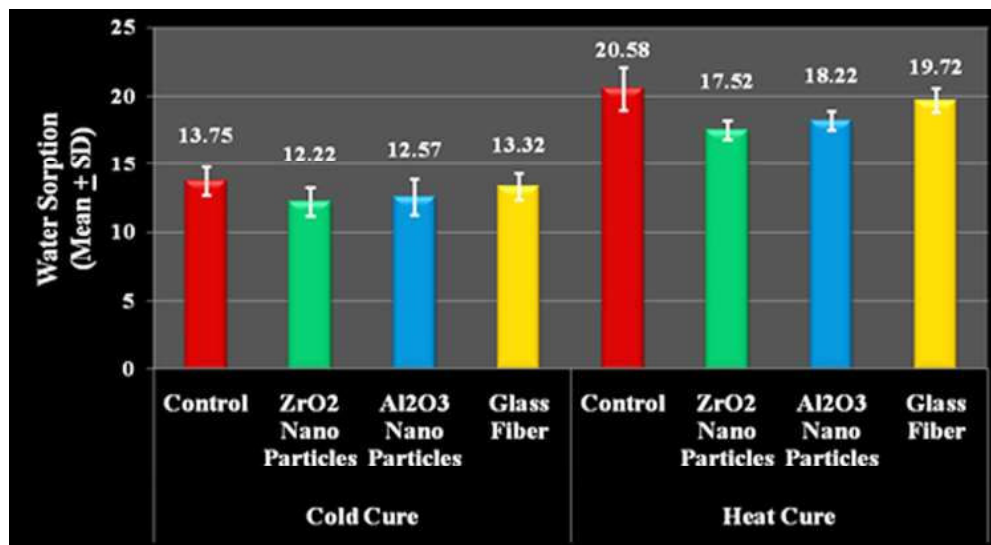
SD - Standard Deviation, VHS - Very Highly Significant



Graph 1: Transverse strength of Heat Cure resin and cold cure resin



Graph 2: Surface hardness of Heat Cure resin and cold cure resin



Graph 3: Water sorption in Heat Cure resin and cold cure resin

## DISCUSSION

PMMA Denture Base Resin currently is the material of choice for denture base fabrication<sup>16</sup> because of its favourable working characteristics, processing ease, accurate fit, low solubility, stability in the oral environment and cost effectiveness.<sup>17</sup>

In spite of the material's popularity, assessments of the clinical longevity of dentures fabricated with poly-methyl-methacrylate concluded that nearly two third (64-68%) of PMMA dentures fracture after a few years of service in the patient's mouth.<sup>18</sup>

Fractures occur primarily due to two reasons: impact failure or due to fatigue failure, where it is repeatedly undergoing deformation under functional forces i.e., low and repetitive stress which commonly occurs over a period of time.

Cold cure material was included in the study because, denture repair is often required urgently when it is fractured. Material of choice for immediate denture repair is Cold CureDBR<sup>19</sup> because it has almost all properties comparable to Heat Cure denture base material.

The main objective of this study was to bring about an improvement in clinically relevant parameters such as flexural strength, surface hardness and water sorption of Heat Cure and Cold Cure Denture Base Resins, as it generally affects the durability, mechanical performance and the longevity within the functional range of forces of the dentures.

The transverse strength of a material reflects its potential to resist catastrophic failure under flexural loads. Relatively small stresses caused by mastication over a period of time can contribute to

formation of a small crack, which propagate through the denture thereby resulting in a fracture.<sup>20</sup>

Second parameter included in the study was hardness. Hardness is the resistance of a material to a permanent surface penetration by a hard indenter. Scratching or abrasion resistance is a function of material hardness.<sup>21</sup>

Last parameter added for evaluation was water sorption. Absorption has a negative effect since it interferes with the entanglement of polymer chains acting as plasticizers<sup>22</sup> and it also causes hydrolytic degradation.<sup>22</sup> Therefore, water sorption of these materials should be as low as possible.

Reinforcement of PMMA with numerous types of fibers<sup>23</sup> and nanoparticles like glass fibers, ZrO<sub>2</sub>, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> nanoparticles,<sup>24</sup> metal inserts have been quite successful previously in order to enhance its mechanical properties.

In the present study Heat Cure and Cold Cure Denture Base Resins were reinforced with ZrO<sub>2</sub> nanoparticles, Al<sub>2</sub>O<sub>3</sub> nanoparticles and Glass fibers. Their use can be justified by following reasons.

Nanomaterials have captured attention because of their unique structures and properties. They have small size, large surface area, high surface energy, a large proportion of surface atoms and unique effects like, small size effect, quantum size effect and quantum tunnelling effect.<sup>25</sup>

ZrO<sub>2</sub> nanoparticles were taken as fillers in the study because nano composite denture base has higher interfacial shear bond strength between the resin matrix and nano materials,<sup>26</sup> compared to the conventional resin matrix because super molecular bonding creates thick interface, which enhances

the bond between the resin molecules and creates higher molecular weight polymers.<sup>26</sup>

Second filler which was taken in the study was Aluminium oxide ( $\text{Al}_2\text{O}_3$ ), has strong ionic interatomic bonds.<sup>27</sup> Aluminium ions of alpha phase have small ionic radii which fills two third of the octahedral voids forming a close hexagonal packed structure thus improving physical and mechanical properties of PMMA resin.

The particle sizes of  $\text{Al}_2\text{O}_3$  nanoparticles and  $\text{ZrO}_2$  nanoparticles were selected between the range of (20-50nm). Small particle size of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  nanoparticles filled the interstitial of polymer particles to give a heterogenous mixture and did not force the displacement of the segments of polymer chain.<sup>27</sup>

To achieve reinforcing effect with nanoparticles much lower volume fraction of reinforcing media is needed because nanoparticles with aspect ratio larger than 30 could nearly make the reinforcing effect reach saturation.<sup>28</sup> Hence, lower percentage of  $\text{Al}_2\text{O}_3$  nanoparticles (5 wt %) and  $\text{ZrO}_2$  nanoparticles (5 wt %) were used in this study.

Third filler which was taken in the study was glass fibers. Glass fibers are known for their acceptable appearance and excellent mechanical properties.<sup>29</sup> Modulus of elasticity of glass fibers is very high, most of the stresses are received by them without deformation.

The results of the present study showed that, there was significant increase in transverse strength and surface hardness with addition of fillers (Glass fibers,  $\text{Al}_2\text{O}_3$  nanoparticles and  $\text{ZrO}_2$  nanoparticles) as compared to specimens in the control group.

Three point bending test was used to record transverse strength. Its principle is similar to the load applied on the maxillary denture in situ.<sup>21</sup>

The results showed that addition of 5 wt%  $\text{ZrO}_2$  nanoparticles to both Denture Base Resins delivered the greatest improvement in transverse strength followed by glass fibers and then  $\text{Al}_2\text{O}_3$  nanoparticles. The maximum increase in transverse strength at 5 wt% of  $\text{ZrO}_2$  nanoparticles can be attributed to the high interfacial shear strength between nano filler and matrix due to formation of cross links or supra molecular bonding which covers or shields the nano fillers. Similar study revealed increase in transverse strength at 5 wt% of  $\text{ZrO}_2$  nanoparticles.<sup>30</sup>

Increase in transverse strength of Denture Base Resins were also observed with addition of glass

fibers. The high modulus of elasticity of glass fibers as well as the strong bond between the matrix and fibers leads to hindrance of crack initiation and propagation under the failure load.<sup>31</sup> The results of the current study agree with those of a previous study by Yu et al.<sup>31</sup>

Increase in transverse strength of DBRs were also observed with addition of  $\text{Al}_2\text{O}_3$  nano particles as compare to controlled group because interstitial spaces between polymer particles were filled heterogeneously without causing any displacement of the polymer chains.<sup>24</sup> The results of the current study agree with those of a previous study by Ayman.<sup>27</sup>

The Vickers micro hardness values indicated that the Denture Base Resins reinforced with fillers had higher surface hardness than the specimens in the control group. In the present study, change in surface hardness were significantly greater in both Denture Base Resins reinforced with  $\text{ZrO}_2$  nanoparticles > glass fibers >  $\text{Al}_2\text{O}_3$  nanoparticles.

The maximum increase in surface hardness at 5 wt% of  $\text{ZrO}_2$  nanoparticles can be attributed to super molecular bonding, it creates thick interface, which enhances the bond between the resin molecules and creates higher molecular weight polymers. The results of the current study agree with those of a previous study by Alhotan et al.<sup>32</sup>

Addition of glass fibers also increased surface hardness. This increase in hardness was due to a strong bond between the matrix and fibers, which was in agreement with the findings in another study.<sup>28</sup>

Addition of 5 wt%  $\text{Al}_2\text{O}_3$  nanoparticles showed higher surface hardness than the specimens in the control group.  $\text{Al}_2\text{O}_3$  possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics. Similar study stated increase in surface hardness by addition of  $\text{Al}_2\text{O}_3$  fillers.<sup>33</sup>

While improving the mechanical properties of a Denture Base Resins, it is also of great importance not to compromise its physical properties such as water sorption and solubility. Denture Base Resins are notable for their tendency to absorb water, which causes corresponding dimensional change and the hydrolytic degradation of polymers.<sup>34</sup>

The rate at which the materials absorbed water varied considerably with the type of material, the amount of plasticizer or filler content, the solution in which they were immersed and resin polarity,

dictated by the concentration of polar sites available to form hydrogen bonds with water and network topology.<sup>35</sup>

Polymers can be degraded by the physico-chemical process after absorbing a high amount of water, starting with swelling which causes dimensional distortion.<sup>34</sup> It also increases its flexibility and potential to fracture, reduces hardness.

The results of water sorption of both Denture Base Resins reinforced with fillers after immersion in water for 7 days were in accordance with ISO specification 1567. According to this specification, water sorption should not exceed 32 µg/mm<sup>3</sup> for Heat Cured or Cold Cured materials. Among all fillers used in the current study, ZrO<sub>2</sub> nanoparticles showed least water sorption followed by Al<sub>2</sub>O<sub>3</sub> nanoparticles and then glass fibers. In present study, Cold Cured resin showed lower water sorption than heat cured resin, which was in agreement with the findings in another study.<sup>36</sup>

To summarize, the present study concluded that reinforcement of both Denture Base Resins with 5 wt% ZrO<sub>2</sub> nanoparticles delivered greatest improvement in transverse strength and surface hardness followed by 2.5 wt% glass fibers and then 5 wt % Al<sub>2</sub>O<sub>3</sub> nanoparticles. On comparing both Denture Base Resins considered in the study. Heat Cure Denture Base Resin showed higher transverse strength and surface hardness than Cold Cure resin.

The results of water sorption of both Denture Base Resins reinforced with fillers were in accordance with ISO specification. Among all fillers used in the current study, ZrO<sub>2</sub> nanoparticles showed least water sorption followed by Al<sub>2</sub>O<sub>3</sub> nanoparticles and then glass fibers. Cold Cure Denture Base Resin showed less water sorption in comparison to Heat Cure Denture Base Resin.

## CONCLUSION

From the results of this study, it could be concluded that incorporating 5 wt% ZrO<sub>2</sub> nanoparticles, 5 wt% Al<sub>2</sub>O<sub>3</sub> nanoparticles, 2.5 wt% glass fibers in Cold cure and Heat Cure Denture Base Resin results in increase in both transverse strength and surface hardness, while the increase was maximum in DBR reinforced with ZrO<sub>2</sub> nanoparticles followed by glass fibers and then Al<sub>2</sub>O<sub>3</sub> nanoparticles. Increase in the transverse strength and surface hardness of the acrylic resin base material could lead to more patient satisfaction.

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