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ORIGINAL ARTICLE

# A comparative evaluation of properties of zirconia reinforced high impact acrylic resin with that of high impact acrylic resin

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KEYWORDS

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**Abstract** *Statement of the problem:* Poly methyl methacrylate (PMMA) acrylic resin, although being the most popular denture base material, is associated with poor mechanical properties. It has been documented that acrylic resin can be strengthened with an addition of structural component (filler) added in the acrylic matrix, to form a composite structure.
*Objective:* To evaluate and compare the transverse strength, impact strength, surface hardness and water sorption of 10% and 20% zirconia ($ZrO_2$) reinforced high impact acrylic resin with that of high impact acrylic resin (Trevalon HI, Dentsply India).
*Materials and methods:* There were 30 specimens in each of the four tests, amounting to a total of 120 specimens. Each of the tests had 10 specimens fabricated from high impact acrylic resin (control); 10 specimens fabricated from 10% zirconia ($ZrO_2$) and 10 specimens fabricated from 20% zirconia ($ZrO_2$) reinforced high impact acrylic resin. Specimens were subjected to the test of transverse strength in Universal Testing Machine, impact strength in Izod pendulum impact testing machine and surface hardness by Vickers Microhardness tester according to ISO Specification No. 1567. Water sorption was assessed according to ADA Specification No. 12. Data were analyzed by means

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of a one-way ANOVA and Bonferroni multiple comparison test.
*Results:* A significant increase in the transverse strength was observed in the reinforced specimens when compared to the control group. Impact strength and surface hardness were found having lesser values compared to the control group. Water sorption was found to increase on the addition of 10% and 20% zirconia ($ZrO_2$) but the value lied below 0.8 mg/cm$^2$ i.e. within the limit of ADA Specification No. 12.
*Conclusion:* Reinforcement of acrylic resin with zirconia powder affects its physical and mechanical properties significantly.



## 1. Introduction

Poly methyl methacrylate (PMMA) acrylic resin is a preferred denture base material because of its low cost, ease of application, polishability along with its reliance on simple processing equipment.[1–3] But a major drawback of the use of PMMA as a denture base material is its low transverse and impact strength that leads to common occurrences of the fracture of prosthesis in-situ and ex-situ. Increase in impact strength is required to prevent the fracture of denture resulting from its accidental fall, while transverse strength helps to withstand higher flexural stresses developed during mastication.[4–7]

Fracture of denture base in the mouth occurs via fatigue mechanism in which, over a period of time, even the relatively small flexural stresses lead to the formation of microscopic cracks in areas of stress concentration. With continued load bearing, these cracks fuse to ever growing fissure that weakens the material. Catastrophic failure results from a final loading cycle that exceeds mechanical capacity of remaining sound portion of the material. Additionally, denture fracture is also frequently related to faulty design, fabrication and material choice.

Many attempts have been made in the past to improve mechanical properties of acrylic resins including its chemical modification by the addition of a rubber graft copolymer and also by the addition of various reinforcing materials like metals, metal fillers, carbon fibers, aramid fibers, glass fibers and ultra-high modulus polyethylene.[8,9]

The search of literature reveals that the addition of varying amount of metal fillers such as powdered silver, copper and aluminum into PMMA at various concentrations not only gives it an advantage of increased strength and improved thermal conductivity, but also reduces the polymerization shrinkage, decreases the warpage, makes the material radiopaque and inhibits the growth of bacteria over the denture surface. The major disadvantage of adding metal fillers however is its compromised esthetics.[10,11]

The recent approach to improve the properties of acrylic resins is the addition of zirconium oxide (zirconia, $ZrO_2$) as filler. Studies have shown that zirconia is biocompatible and additional advantage of zirconia as filler over other metal fillers is superior esthetics.[12–14] Ayad et al.[14] investigated the effect of reinforcing high impact resin with 5% and 15% $ZrO_2$ and concluded that reinforcement of high impact resin with zirconium powder increases its transverse strength significantly. Further studies were recommended with different concentrations of zirconia filler reinforcements to the different acrylic resin system to understand more on the effects of zirconia reinforcement on mechanical properties of acrylic resins.

This study was planned to evaluate the effect of 10% and 20% zirconium oxide reinforcement on the properties of high

**Table 1** Showing mean values and standard deviation for the control group, Group A (10% $ZrO_2$ reinforcement) and Group B (20% $ZrO_2$ reinforcement) specimens for transverse strength, impact strength, surface hardness and water sorption respectively.

| | | No. of samples | Mean X | Std. deviation SD |
|---|---|---|---|---|
| Flexural transverse strength (Mpa) | Control | 10 | 76.2490 | 6.21694 |
| | Group A | 10 | 101.18 | 9.30953 |
| | Group B | 10 | 94.1200 | 7.92012 |
| | Total | 30 | 90.5173 | 13.12489 |
| Izod impact strength (J) | Control | 10 | 1.8690 | 0.05183 |
| | Group A | 10 | 1.6767 | 0.09084 |
| | Group B | 10 | 1.7499 | 0.08001 |
| | Total | 30 | 1.7652 | 0.10899 |
| Surface hardness (VHN) | Control | 10 | 17.264 | 0.3590 |
| | Group A | 10 | 15.778 | 0.7110 |
| | Group B | 10 | 16.687 | 0.2765 |
| | Total | 30 | 16.576 | 0.7795 |
| Water sorption (mg/cm$^2$) | Control | 10 | 0.49430 | 0.009286 |
| | Group A | 10 | 0.58500 | 0.020950 |
| | Group B | 10 | 0.79670 | 0.008274 |
| | Total | 30 | 0.62533 | 0.129585 |

impact acrylic resin. The properties evaluated were transverse strength, impact strength, surface hardness and water sorption.

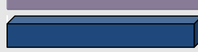
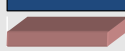
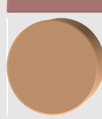
## 2. Materials and methods

This *in-vitro* study was conducted at Darshan Dental College and Hospital, Udaipur, India and testing was done at Praj Laboratory, Pune, India. High impact acrylic resin (Trevalon HI, Dentsply India) was used as a control element in this study. $ZrO_2$ (99.56% pure, Star Earth Minerals Private Limited India) was added to the high impact acrylic resin in a concentration of 10% and 20% by weight.[13–15] Rectangular metal dies of dimensions 65 mm length × 10 mm width × 2.5 mm thickness were used for measuring transverse strength, 60 mm length × 7 mm width × 4 mm thickness for measuring impact strength, and 30 mm length × 10 mm width × 2.5 mm thickness for measuring surface hardness respectively were prepared according to International Standards Organization (ISO) Specification No. 1567. Cylindrical disk shaped die with 50 mm diameter × 0.5 mm thickness was prepared in addition for testing water sorption according to ADA Specification No. 12.[14]

Furthermore, ten specimens were fabricated from each test material for testing transverse strength, impact strength, surface hardness and water sorption respectively, to account for a total of 120 specimens. Specimens retrieved were inspected for any irregularity. Faulty specimens were discarded and final specimens were selected for each group. Resin specimens were then stored in water for 4 weeks before testing. Specimens thus fabricated were subjected to test the transverse strength, impact strength, surface hardness and water sorption.

The transverse strength test was conducted according to ISO Specification No. 1567 for the denture base polymers. Specimens were tested for transverse strength by three point bending test in Universal Testing Machine (software based, Star Testing Systems, India. Machine No: STS 248, Traceability: National Physical Laboratory, New Delhi). A three point testing design was used whereby the specimen beam was centrally loaded at a cross head speed of 5 mm/min over a two-point support span set at a distance of 50 mm. Specimens were deflected until rupture occurred. The maximum load required for rupture was recorded as its transverse strength.

Showing dimensions and shape of master dies for different tests.

| Test | Master die | Dimensions (ISO specifications 1567) |
|---|---|---|
| Transverse strength |  | 65 mm length × 10 mm width × 2.5 mm thickness |
| Impact strength |  | 60 mm length × 7 mm width × 4 mm thickness |
| Surface hardness |  | 30 mm length × 10 mm width × 2.5 mm thickness |
| Water sorption |  | Disk shaped specimen with 50 mm diameter × 0.5 mm thickness |

These metal dies were invested in a flask using type III dental stone. Once the dental stone was set, the two halves of the flask were separated and the dies were retrieved from molds without distorting the mold space. Afterwards, sodium alginate separating media was applied to the mold and left to dry.

For fabricating specimens of the control group in respective tests, resin powder of high impact denture base resin was mixed with monomer in a ratio of 2.7:1 and packed into mold space in the dough stage. Trial closure was done and flasks were clamped. Following this, bench curing was done for 20 min and resin specimens were acrylized according to the manufacturer's instructions. After curing, the flasks were bench cooled to room temperature and control group specimens were retrieved.

For fabricating zirconia reinforced test specimens, zirconia powder of 99.56% purity with an average particle size of 5–10 μm was selected as filler. The zirconium oxide filler and PMMA were pre-weighed in order to ensure a filler concentration of 10% and 20% by weight. Filler particles were treated with 1 wt% of silane coupling agent (Dentsply, India) before the mix. Mixing and blending were done to obtain a consistent and uniform mix. This powder was then mixed with monomer in a ratio of 2.7:1 and specimens were prepared like the control group. Specimens with 10% and 20% $ZrO_2$ reinforcements were denoted as the group A and group B respectively.

Izod pendulum impact testing machine (Zwick, Germany) was used for testing the impact strength of the specimens. The machine is a pendulum-type with a disk-shaped hammer carrying a knife edge. The un-notched specimens were mounted vertically, clamped at one end with 2.5 cm length of the specimens inside the clamp and 3.5 cm above the level of holder, so that pendulum strikes the specimens at a velocity of 5.5 m/s, at same site to all specimens. The impact strength of a specimen was recorded as number of joules of energy absorbed in breaking the specimen. The machine gives a digital reading with least count of 0.01 J.

Digital Vickers Microhardness tester (Reichert, Austria) was used for testing surface hardness. The specimens were polished (SiC #120 and pumice) from one surface and a 50 gf load was applied for 5 s indentation time and VHN was obtained digitally.

Water sorption was tested according to ADA Specification No. 12. Specimens were placed in a desiccator (Mahavir, India) at 37 ± 10 °C for 23 h and then allowed to stand at an ambient temperature for 1 h. Each specimen was weighed by an electronic balance (LWL, Germany), and the previously described cycle was repeated till the specimens reached a constant mass (w1). These specimens were placed in distilled water at 37 ± 10 °C for 7 days and were then removed and weighed again (w2). Water sorption was calculated in mg/$cm^2$ using the following formula[14–17]

**Table 2** Showing cumulative results of Mean, Standard deviation, one-way ANOVA and Bonferroni multiple comparison tests among different groups for properties tested.

| | Control | Group A | Group B | ANOVA | | Bonferroni multiple comparison test | | |
|---|---|---|---|---|---|---|---|---|
| | X ± SD | X ± SD | X ± SD | $F$ | $P$ | $P_1$ | $P_2$ | $P_3$ |
| Transverse strength (MPa) | 76.25 ± 6.2 | 101.20 ± 9.3 | 94.12 ± 7.9 | 26.35 | 0.001* | 0.001* | 0.001* | 0.169 |
| Impact strength (J) | 1.87 ± 0.05 | 1.67 ± 0.09 | 1.74 ± 0.08 | 16.23 | 0.001* | 0.001* | 0.005* | 0.121 |
| Surface hardness (VHN) | 17.26 ± 0.36 | 15.87 ± 0.71 | 16.68 ± 0.27 | 23.68 | 0.001* | 0.001* | 0.040* | 0.001* |
| Water solubility (mg/cm$^2$) | 0.49 ± 0.01 | 0.58 ± 0.02 | 0.79 ± 0.01 | 1.21 | 0.001* | 0.001* | 0.001* | 0.001* |

$P_1$ – Between the group A and control group.
$P_2$ – Between the group B and control group.
$P_3$ – Between the group A and group B.
* $P < 0.05$ – significant value.

Ws = w2 − w1/A (area of specimen)

The mean transverse strength, impact strength, surface hardness and water sorption properties for each group were calculated and data were tabulated and analyzed by means of a one way ANOVA test, followed by Bonferroni multiple comparison test. Statistical significance level was set at $P < 0.05$.

## 3. Results

Table 1 shows the mean values (X) and standard deviation (SD) for transverse strength, impact strength, surface hardness and water sorption for the test groups A and B (10% and 20% $ZrO_2$) and the control group. Table 2 shows results of ANOVA and Bonferroni comparison test among different categories and properties evaluated.

The results revealed an increase in values of transverse strength and water sorption and a decrease in values of impact strength and surface hardness in both group A (10% $ZrO_2$) and group B (20% $ZrO_2$) as compared to the control group. The one-way ANOVA test indicated that there were significant differences among the groups for all the four properties evaluated.

The Bonferroni multiple comparisons show a statistically significant difference between the two test groups (A and B) and the control group for all properties evaluated ($P < 0.05$). Among the test groups (A and B) there was a statistically significant difference in surface hardness and water sorption ($P < 0.05$). For transverse and impact strengths, the difference among the test groups (A and B) however, was statistically insignificant ($p > 0.05$).

## 4. Discussion

The fractures of the denture can be reduced by increasing the strength of PMMA which is commonly used as a denture base material. Although the incorporation of fibers increases the impact strength, a large amount of fibers tend to decrease the surface hardness with no significant increase in strength.[9,18,19] The addition of metal fillers increases thermal conductivity and compressive strength but is also associated with compromised esthetics and a decrease in tensile strength.[10]

The incorporation of zirconia in various dental materials has been studied and it was found to be biocompatible and had significant beneficial effects on the mechanical properties.[12–15] Also, the white color of zirconia powder does not compromise the esthetic appearance unlike its metal filler counterparts like aluminum, copper or silver.

Zuccari et al.[12] in their study concluded that (1) addition of particles generally increases the water sorbed by the resins systems; (2) however, two volume % admixtures in a PMMA resin matrix show significant improvements in the mechanical properties; (3) among the oxide particles, zirconia exhibited the greatest improvement in modulus of elasticity, transverse strength, toughness and hardness; and (4) mechanical properties (transverse strength, 0.2% offset yield strength and modulus of elasticity) were linearly correlated to hardness numbers.

Apart from its filler type, size, distribution and composition, the mechanical properties of the resin are also affected by adhesion at polymer-filler interface.[5,11,14,15] In this study zirconia powder was added in concentrations of 10% and 20% after treating with silane coupling agent to obtain the necessary chemical bond between zirconium oxide and acrylic resin. The use of zirconia as filler particles of size 7–18 μm when added to PMMA helps in filling the matrix interstitially. Amount of filler used to reinforce acrylic resin is another important factor affecting mechanical properties. Percentage of filler used for reinforcement should be such that the filler particles should disperse evenly into the resin matrix without interrupting the continuity of the resin matrix. The increase in the filler fraction does not necessarily lead to an increase in the strength however, because excessive filler fractions create more defects the material weakens.[5,10]

The present study reveals that reinforcement of high impact denture base resin with zirconium powder results in an increase in its transverse strength. On the addition of 10% $ZrO_2$ (group A) filler particles, there was 32% increase in transverse strength as compared to the control group specimens whereas with the addition of 20% $ZrO_2$ (group B) filler, this increase was only 23%. This increase in transverse strength with the reinforcement of zirconia filler particles is statistically significant. The results of this study are consistent with the findings reported by other authors who concluded that reinforcements of ceramics, dental restorative resins, provisional restorative resins and acrylic resins with zirconia could exhibit improvement in their mechanical properties.[12–15] This increase in transverse strength can be explained on the basis of interstitial filling of acrylic resin matrix with $ZrO_2$ which interrupted with the crack propogation.[12,13] The addition of 20% $ZrO_2$ resulted in a decrease in the transverse strength as compared to 10% $ZrO_2$. The reasons for this decrease can be attributed to higher filler content. The effect of higher filler content on reducing strength can be explained on the basis that after reaching a saturation point the resin cannot incorporate further more filler particles. Any attempt to add filler particles after reaching saturation of matrix leads to interruption in the resin matrix continuity and thus causing a decrease in the strength of reinforced specimens.[20,21]

With the addition of 10% $ZrO_2$ (group A) filler particle, there was a 10% decrease in impact strength as compared to the control group specimens; whereas with the addition of 20% $ZrO_2$ (group B) filler there was only a 6% decrease in impact strength values. The reinforcement of high impact acrylic resin with 10% and 20% $ZrO_2$ showed a decrease in impact strength values compared to the control group. This result was similar to the previous studies.[14] Although impact test is popular because of its ease and speed of testing, it is not completely indicative of the intrinsic strength of the material. The test methodology depends on specimen dimensions, notch depth, radius, impact velocity, etc. Furthermore the total measured impact energy contains kinetic, frictional and vibrational energies that are not directly correlated with fracture resistance property of the denture base material.

With the addition of 10% $ZrO_2$ (group A) filler particle, there was an 8% decrease in surface hardness as compared to the control group specimens whereas with the addition of 20% $ZrO_2$ (group B) filler there was a 3% decrease in surface hardness values as compared to the control group specimens. The results obtained are consistent with previous studies.[14]

With the addition of 10% $ZrO_2$ (group A) filler particle, there was an 18% increase in water sorption as compared to the control group specimens whereas with the addition of 20% $ZrO_2$ (group B) filler there was a 61% increase in water sorption values as compared to the control group specimens, both showing statistically significant results. Water sorption occurs because of weak secondary bonds in polymer materials; water molecules are able to penetrate between polymer chains in the process. The interface between the particle and polymer is water sensitive because of high surface energy of the particle related to the polymer and the permeability of the polymer allows water to reach the interface. Increase in filler concentration contributes to more particle–polymer interface which in turn results in more water sorption phenomenon.[13] According to ADA Specification No. 12, water sorption should not exceed 0.8 $mg/cm^2$. The results of this study lie within this specification and are in agreement with those of the other authors who found that the addition of reinforcing particles generally increases the water sorption by acrylic resin.[13,17,22,23]

The present study shows that incorporation of zirconium oxide fillers into high impact denture base resin, improves the transverse strength of the material and thus increases the fracture resistance of the material. This increase in transverse strength is accompanied with an adverse effect on the impact strength. Impact strength is required in the cases like accidental dropping/fall of denture, etc. while flexural stresses are a constant phenomenon during mastication which is counteracted by transverse strength of the material, thus making transverse strength a much more significant feature. This justifies the incorporation of zirconia as filler to high impact acrylic resins. Furthermore zirconia is biocompatible and being white in color, it does not adversely affect the esthetic appearance of the denture base.

Clinically, zirconia reinforced PMMA may be useful in situations where masticatory forces are relatively more, like distal extension bases opposing natural teeth, single complete denture, overdentures, long term provisional restorations and implant supported complete arch prosthesis.

This study is limited with the use of zirconia only, rather other forms like YSZ and nano-composites of titanium, aluminum with zirconium oxide can also be used for reinforcement. Further studies are required to check the effect of zirconium reinforcement on the denture base resins when specimens are stored in water and artificial saliva for a longer duration. In-situ studies are suggested to investigate the clinical performance of this material in oral cavity. These studies would help in the development of PMMA/Zr composite reinforcements.

## 5. Conclusion

Within the limitation of this study it can be concluded that the addition of zirconium oxide as a filler in the high impact acrylic resin increases their transverse strength as compared to the control group (Trevalon HI). Impact strength and surface hardness of the zirconia reinforced specimens were found to have relatively lesser values compared to the control specimens. Water sorption of the zirconia reinforced specimens was found to increase but was within the limit of ADA Specifications No. 12.

## Conflict of interest

None declared.

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