A COMPARATIVE EVALUATION OF THE IMPACT AND FLEXURAL STRENGTHS OF HIGH IMPACT ACRYLIC RESIN REINFORCED WITH SILANED ZIRCONIUM DIOXIDE AND TITANIUM DIOXIDE NANOPARTICLES AND ITS HYBRID COMBINATION - AN INVITRO STUDY

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Abstract
Heat cure acrylic resin is the most widely used material for the fabrication of removable dentures even today. But due to its low fatigue strength and fracture resistance, the dentures tend to flex and can also fracture. With the advancement in nanoscience, nano-sized fillers were found to exhibit improved fracture resistance. Hence in this study both ZrO₂(5%) & TiO₂(1%) nanoparticles and its hybrid combination (2%) were used to comparatively evaluate the impact and flexural strengths of high impact acrylic resin. A total of 45 specimens were subjected to impact strength testing in an impact tester and flexural strength testing using three point bending test in a Universal testing machine respectively. The present study shows that hybrid reinforcement can be considered a better reinforcement option for enhancing the impact and flexural strengths of acrylic resin.

Introduction:
Even though dental implants are a better treatment option for edentulous patients, conventional complete dentures are opted due to medical and financial reasons like ease of processing, affordability, lightness in weight, excellent aesthetic properties, reduced water sorption & solubility, good stability in oral environment and ease of repair. But the drawbacks include low fatigue strength, low fracture resistance, low surface hardness, high coefficient of thermal expansion and brittleness on impact. So when dentures are subjected to compressive, tensile and shearing loads in the oral environment, they tend to flex and fracture. In studies conducted earlier, fracture rate was reported to be 64% to 68% which can occur either due to expulsion of denture while coughing or by accidental dropping of dentures while cleaning. Other reasons could be excessive masticatory load, improper plane of occlusion, lack of stability, ill-fitting dentures and poor quality of the denture base material.

These limitations led to the enhancement of mechanical properties of denture base materials either by chemical modifications or by incorporation of metal oxides, fillers, fibres etc. or by curing the denture bases using different.
methods.\textsuperscript{3} Despite these efforts to improve the fracture resistance of PMMA, only few have obtained promising results.

With the development of nanotechnology and nano-phased materials, attention was directed towards the use of nano-sized fillers like Al\textsubscript{2}O\textsubscript{3}, ZrO\textsubscript{2}, AgO\textsubscript{2}, TiO\textsubscript{2}, nanogold, nanotubes etc. to reinforce denture base resins which was found to exhibit improved mechanical and physical properties like improved wear and tear resistance, anti-corrosion ability, fracture resistance and high polishability due to the alteration in the size of the nanoparticles and also because of surface treatment of nanofillers with silane coupling agent which was found to improve the bonding.\textsuperscript{6,8,9}

Zirconium dioxide being a biocompatible material is used in dentistry to fabricate endodontic posts, crown and bridge restorations and implant abutments.\textsuperscript{10} Few studies that incorporated ZrO\textsubscript{2} nanoparticles into acrylic resin has reported an increase in transverse strength and water sorption but a decrease in the values of impact strength and surface hardness.\textsuperscript{11,12}

Titanium dioxide nanoparticles are also widely used due to its nontoxicity, chemical inactivity, cost efficiency, antibacterial property, corrosion resistance and high microhardness.\textsuperscript{13} Reinforcements with TiO\textsubscript{2} nanoparticle has shown improvement in mechanical as well as physiochemical properties when added at very low concentrations.\textsuperscript{13}

The latest reinforcement technique is the hybrid combination of nanoparticles where the combination of different nanofillers improved the mechanical properties.\textsuperscript{6} Hence this study was done to evaluate and compare the impact and flexural strengths of high impact acrylic resin reinforced with silaned zirconium dioxide and titanium dioxide nanoparticles and its hybrid combination.

**Materials and Methods:-**

**Preparation of mould space:**

According to ISO specification no.1567\textsuperscript{11} metal troughs of dimensions 60mm × 7mm × 4mm for impact strength testing (Figure 1) and 65mm × 10mm × 2.5mm for flexural strength testing (Figure 2) were used respectively to prepare wax patterns (MODELLING WAX-No.2, The Hindustan Dental Product, Hyderabad) for sample fabrication. 45 wax patterns were prepared for impact strength testing and flexural strength testing respectively (Figures 3&4). All the wax patterns were invested in dental flask using dental plaster in two stage pour technique where 3 wax patterns were invested in each flask (Figure 5) following which dewaxing was done at 100°C for 10 minutes. A thin layer of separating medium was then carefully painted on both halves of the flask, left to dry and was then packed with nanoparticle (Sisco Research Laboratories Pvt. Ltd., Mumbai) reinforced high impact heat cured acrylic resin (TREVALON HI, Dentsply India Pvt. Ltd., Gurgaon).

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**Fig 1:** Metal trough of dimension 60mm × 7mm × 4mm.

**Fig 2:** Metal trough of dimension 65mm × 10mm × 2.5mm.
Surface treatment of nanoparticles:
To ensure even coating of the surface of the nanoparticles, silane coupling agent (MONOBOND-S, IvoclarVivadent) was first dissolved in acetone and then added to the jar containing the nanoparticles respectively following which the silanated nanoparticles were incorporated into the polymer.

Incorporation of nanoparticles into polymer powder:
The high impact acrylic resin specimens were prepared according to manufacturer’s instructions in the ratio of 3:1. The following amount of material was used to fabricate three samples at one time:

|            | 5% ZrO₂ | 1% TiO₂ | 2% hybrid (1% ZrO₂ +1% TiO₂) |
|-----------|----------|---------|-----------------------------|
| Polymer   | 0.45 grams of ZrO₂ + 8.55 grams of polymer | 0.09 grams of TiO₂ + 8.91 grams of polymer | 0.09 grams of ZrO₂ + 0.09 grams of TiO₂ + 8.82 grams of polymer |
| Monomer   | 3 ml     | 3 ml    | 3 ml                        |

Fabrication of test specimens:
The measured polymer and monomer were mixed in a clean and dry porcelain mixing jar for 30 seconds. When it reached dough stage, it was packed into the mould space and the flasks were closed and kept under pressure of 2000 psi for bench curing for 30 minutes. Once bench curing was done, the flasks were clamped and polymerized using short curing cycle of 74°C for 90 minutes and then boiled for 1 hour. Then the flasks were allowed to bench cool to room temperature and the specimens were retrieved. Excess material was trimmed and the specimens were polished using sand paper and pumice. All the specimens were stored in distilled water at room temperature for 2 weeks and then thermocycled for 5000 cycles between 5 and 55°C and subjected to their respective testing.
Sample Grouping:
The specimens were divided into three groups 15 samples each as follows:

| Group | IMPACT STRENGTH (Figure 6) | FLEXURAL STRENGTH (Figure 7) |
|-------|-----------------------------|------------------------------|
| Group 1 | 5% ZrO₂                     | 5% ZrO₂                      |
| Group 2 | 1% TiO₂                     | 1% TiO₂                      |
| Group 3 | 2% Hybrid                   | 2% Hybrid                    |

Mechanical Testing:
Impact strength testing:
A notch of 1.2 mm depth with a notch angle of 45° was made in the center on any one lateral surface of the specimen (Figure 8) using sand paper disk and tapered steel file and subjected to impact testing in impact tester (Krystal Equipments, Ichalkaranji, Maharashtra) using Izod method (Figure 9) in which the specimens were clamped at one end vertically where the notched surface was kept facing the pendulum (Figure 10) which was used to break the specimens. The energy absorbed until the specimen fractured was detected, and the values obtained were tabulated for statistical analysis. The impact strength was calculated using the equation:

\[ IS = \frac{EC}{h \times b \times A} \]

- **IS** - Impact strength (kJ/mm²)
- **EC** - Energy absorbed while breaking the test specimen (kJ)
- **bA** - Remaining thickness at the notch tip (7 - 1.2 = 5.8 mm)
- **h** - Specimen width (4 mm).

**Fig 6:** High impact PMMA specimens incorporated with ZrO₂, TiO₂ and ZrO₂-TiO₂ nanoparticles for impact strength testing.

**Fig 7:** High impact PMMA specimens incorporated with ZrO₂, TiO₂ and ZrO₂-TiO₂ nanoparticles for flexural strength testing.

**Fig 8:** ‘V’ shaped notch made at the center for impact strength test.
Flexural strength testing:
Flexural strength of the specimens were determined using three point bending test in a Universal testing machine (UTE-20, FIE, Fuel Instruments and Engineers Private Limited, Maharashtra) with a span length of 40 mm (Figure 11). A load was applied in the center of the specimen at a crosshead speed of 5 mm/min, until the specimen fractured (Figure 12). The flexural strength values of each specimen were calculated using the formula:

\[
S = \frac{3FL}{2bd^2}
\]

where

- S - Flexural strength (MPa)
- F - Fracture load (N)
- L - Span length (40 mm)
- b - Width of the specimen (10 mm)
- d - Thickness of the specimen (2.5 mm)

The results were statistically analysed using One way ANOVA followed by post hoc Tukey’s test for the comparison.

Results:
The results of impact strength of high impact acrylic resin reinforced with 5% ZrO₂ nanoparticle, 1% TiO₂ nanoparticle and 2% hybrid (ZrO₂-TiO₂) nanoparticle showed a significant difference between the groups and the
same has been graphically represented in Graph 1. One-way ANOVA analysis also showed significant differences in the impact strength between Group 1, 2 and 3 (P<0.05) (Table 1). Table 2 depicts Post hoc Tukey’s test where there was a significant difference in the impact strength between the high impact acrylic resin specimens reinforced with 5% ZrO₂ and 1% TiO₂ nanoparticles & 5% ZrO₂ and 2% hybrid nanoparticles (P<0.05). But a non-significant difference was observed between 1% TiO₂ and 2% hybrid nanoparticles (P>0.05).

The flexural strengths of high impact acrylic resin reinforced with silaned ZrO₂ nanoparticle, TiO₂ nanoparticle and its hybrid (ZrO₂-TiO₂) combination is graphically represented (Graph 2). One-way ANOVA analysis and Post hoc Tukey’s test showed a significant difference in the flexural strengths between the three groups (P<0.05) (Tables 3&4).

Table 1: Comparison of impact strength between different groups (One way ANOVA).

| Impact strength | Mean (kJ/mm²) | Standard deviation | F value | Significance |
|-----------------|---------------|--------------------|---------|--------------|
| ZrO₂            | 1.78067       | .361494            | 21.185  | 0.000 (H.S)  |
| TiO₂            | 2.41800       | .233520            |         |              |
| ZrO₂-TiO₂       | 2.22267       | .203135            |         |              |

P < 0.05, HS – Highly Significant

Table 2: Multiple comparison of mean impact strength between the different groups (Post hoc Tukey’s test).

| Impact strength | Mean difference (kJ/mm²) | Standard error | Significance | 95% confidence interval |
|-----------------|--------------------------|----------------|--------------|-----------------------|
| ZrO₂            | -63733                   | .100327        | .000 (H.S)  | -.88108 -.39359       |
| ZrO₂-TiO₂       | -442000                  | .100327        | .000 (H.S)  | -.68574 -.19826       |
| TiO₂            | 195333                   | .100327        | .138 (N.S)  | -.04841 .43908        |

P < 0.05, HS – Highly Significant
P > 0.05, NS – Not Significant

Table 3: Comparison of flexural strength between different groups (One way ANOVA).

| Flexural strength | Mean (MPa) | Standard deviation | F value | Significance |
|-------------------|------------|--------------------|---------|--------------|
| ZrO₂              | 222.66133  | 9.257730           | 73.530  | 0.000 (H.S)  |
| TiO₂              | 247.15200  | 11.584178          |         |              |
| ZrO₂-TiO₂         | 274.49800  | 13.843090          |         |              |

P < 0.05, HS – Highly Significant

Table 4: Multiple comparison of mean flexural strength between the different groups (Post hoc Tukey’s test).

| Flexural strength | Mean difference (MPa) | Standard error | Significance | 95% confidence interval |
|-------------------|-----------------------|----------------|--------------|-----------------------|
| ZrO₂              | TiO₂                  | -24.49066      | 4.276706     | .000 (H.S)         | -.34.8809 -14.1004  |
| ZrO₂-TiO₂         | 51.83666              | 4.276706       | .000 (H.S)  | -62.2269 -41.4464  |
| TiO₂              | ZrO₂-TiO₂             | -27.34600      | 4.276706     | .000 (H.S)         | -.37.7362 -16.9557  |

P < 0.05, HS – Highly Significant
Discussion:
Polymethyl methacrylate (PMMA) heat cured acrylic resins are most commonly used for the fabrication of complete and partial dentures due to their low cost, simple processing procedure, ability to be repaired, biocompatibility and stability in the oral environment because of which PMMA has replaced previous denture base materials such as vulcanite, nitrocellulose, phenol formaldehyde, vinyl plastics, porcelain etc.³

Dentures fabricated with PMMA can fracture due to flexural fatigue or impact forces. Flexural fatigue of dentures as evidenced by midline fracture is due to the stress concentration around the microcracks formed in the material due to continuous application of small forces. Hence, repetitive nature of masticatory load results in propagation of cracks which weaken the denture base and finally results in fracture. Fracture of dentures by impact forces, on the other
hand, results from the sudden application of force to the dentures more likely due to the accidental dropping of dentures on surfaces.\textsuperscript{15}

Flexural strength is a material property, which is defined as the stress in a material just before it yields in a flexure test.\textsuperscript{16} Impact strength is the capability of the material to withstand a suddenly applied load and is often measured with Izod impact strength test or Charpy impact test, both of which measure the impact energy required to fracture a sample. Therefore, a denture base material must be able to withstand crack propagation and impact forces which is an important factor that can greatly affect its performance.\textsuperscript{4,17}

Attempts to improve the mechanical properties of PMMA have been made in the past by modifying the composition with copolymers, by using different curing methods, by reinforcing with fibres like aramid, nylon, carbon/graphite, polyethylene, polypropylene and particularly glass fibres due to their biocompatibility, superior aesthetics and mechanical properties or by adding fillers like Al\textsubscript{2}O\textsubscript{3}, ZrO\textsubscript{2}, TiO\textsubscript{2}, SiO\textsubscript{2} etc.\textsuperscript{6,18} Acrylic resin also has been chemically modified through the incorporation of rubber in the form of butadiene styrene which has proved to be successful. High impact acrylic resin is a dough-molded, micro-dispersed rubber phase polymer. It is methyl methacrylate and butadiene-styrene copolymerized in an emulsion with a second coating of methyl methacrylate added to cover the bead.\textsuperscript{27}

Nanofillers like nanoclays, nanotubes, nanofibres and nanoparticles have been suggested to improve PMMA properties due to their high surface area, fine size and homogenous distribution and is dependent on the size, shape, surface area, type, concentration and their dispersion into the resin matrix. Nanoparticles such as Alumina (Al\textsubscript{2}O\textsubscript{3}), Zirconia (ZrO\textsubscript{2}), Titanium (TiO\textsubscript{2}), Silver, Gold, Platinum, Hydroxyapatite and Silica based (SiO\textsubscript{2}) are among the nanofillers that has been used as reinforcement options.\textsuperscript{6}

Zirconium oxide (ZrO\textsubscript{2}) is a metal oxide that possesses several advantages such as good mechanical strength, fracture toughness, hardness, wear and chemical resistance and good thermal stability, thus making it beneficial for use in dental materials for reinforcement of denture base resins.\textsuperscript{20} Several studies that incorporated zirconium dioxide (ZrO\textsubscript{2}) nanoparticles showed improvement in the mechanical properties of PMMA.\textsuperscript{3,4,11,12,20,22}

Titanium (Ti) and Ti-based alloys are the materials preferred in the production of implants for medical and dental applications due to their low toxicity, ease of availability, low cost, chemical stability, resistance to corrosion, good strength, high refractive index and biocompatibility. Studies have shown that the addition of TiO\textsubscript{2} nanoparticles into acrylic resin also affected the physical and mechanical properties of the resulting hybrid material.\textsuperscript{18,24,25} Reinforcement of PMMA by more than one type of fibre was first suggested by Vallittu in 1997. It was concluded that hybrid reinforcement significantly increased the flexural strength, impact strength, surface roughness, tensile strength, flexural modulus, hardness, thermal conductivity as well as compressive and fatigue strengths.\textsuperscript{6} Hybrid reinforcement reported to be better than single additive mainly due to the size difference between titanium and zirconium.\textsuperscript{8}

Surface modification of nanoparticles with silane coupling agent is necessary to reduce the surface energy and increase its compatibility with polymer matrix as this could eliminate aggregation of nanoparticles and aid in homogenous dispersion.\textsuperscript{4} In the oral cavity, the denture prostheses are usually under conditions of thermal variations due to the ingestion of hot and cold liquids.\textsuperscript{28} In order to simulate the temperature variations and the aging process in the oral cavity, all the samples were thermocycled for 5000 cycles between 5 and 55°C.\textsuperscript{14}

In the previous studies, impact and flexural strength tests were carried out between unreinforced acrylic resin and samples reinforced with ZrO\textsubscript{2}, TiO\textsubscript{2} and hybrid (ZrO\textsubscript{2}-TiO\textsubscript{2}) nanoparticles individually. Since there was no comparison among these three reinforcements, the present study aims to evaluate & compare the impact and flexural strengths of 5% silaned ZrO\textsubscript{2} nanoparticle, 1% silaned TiO\textsubscript{2} nanoparticle and 2% silaned hybrid (ZrO\textsubscript{2}-TiO\textsubscript{2}) nanoparticle incorporated to high impact acrylic resin.

The results of the impact strength testing showed that reinforcement with 5% silaned ZrO\textsubscript{2} nanoparticle decreased the impact strength value which could be because of the effect of tetragonal-to-monoclinic phase transformation of ZrO\textsubscript{2} that might cause microcracks on the surface of ZrO\textsubscript{2} when the crack propagation is not arrested. Impact strength also depends on specimen dimensions, notch depth and impact velocity. 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.\textsuperscript{11} Similar results were seen in a study done by Asopa V et al.\textsuperscript{11} But impact strength increased when reinforced with 1\% silaned TiO$_2$ nanoparticle. Same conclusion was drawn by Ghahremani L et al in 2017 where colour modified acrylic resin reinforced with 1 wt\% TiO$_2$ showed significantly higher tensile and impact strengths compared to the conventional acrylic resin as the applied load is mainly tolerated by the nanoparticles where the polymer matrix provides structural integrity and load distribution which inhibits the crack propagation.\textsuperscript{29} Due to the presence of ZrO$_2$ nanoparticle, the hybrid combination (ZrO$_2$-TiO$_2$) showed a slight decrease in impact strength when compared to specimens reinforced with TiO$_2$ nanoparticles but this difference is non-significant as the formation of cross-links or supramolecular bonding between the nanoparticles and the matrix prevents the propagation of crack by transferring the stress from the matrix to fillers. Though the hybrid reinforcement showed a decrease in the impact strength yet it is believed that the impact strength values still would be higher than unreinforced acrylic resin as observed in a study by Salman TA and Khalaf HA in 2015.\textsuperscript{8}

The results of the flexural strength testing showed that reinforcement with 5\% silaned ZrO$_2$ nanoparticle caused an increase in the flexural strength which is similar to the study conducted by Asopa V et al in 2015 and the reason was attributed to the interstitial filling of acrylic resin matrix with ZrO$_2$ which interrupted the crack propagation.\textsuperscript{11}

Reinforcement of acrylic resin with TiO$_2$ nanoparticles also showed an increase in the flexural strength values due to the reduction in size of the nanoparticle that helps in filling the interstitial matrix space. The results are in agreement with that obtained by Nazirkar G et al in 2014\textsuperscript{2} and Harini P et al in 2014.\textsuperscript{9} Since reinforcement with ZrO$_2$ and TiO$_2$ nanoparticles both caused an increase in the flexural strength values, the hybrid (ZrO$_2$-TiO$_2$) reinforcement also caused a significant increase in flexural strength. This is in agreement with the study conducted by Salman TA and Khalaf HA in 2015.\textsuperscript{8} This could be attributed to the better dispersion of nanoparticles in the resin matrix due to the difference in the sizes of ZrO$_2$ and TiO$_2$ nanoparticles and also due to transformation toughening.\textsuperscript{8}

The present study showed that incorporation of ZrO$_2$ nanoparticles into high impact denture base resin improved the flexural strength thus increasing the fracture resistance of the material. But this increase in flexural strength is accompanied with an opposite effect on the impact strength. Impact strength is required in cases like accidental dropping or fall of denture 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 ZrO$_2$ nanoparticle as filler to high impact acrylic resins. When comparing the impact and flexural strengths of high impact acrylic resin reinforced with 5\% silaned ZrO$_2$ nanoparticle, 1\% silaned TiO$_2$ nanoparticle and 2\% silaned hybrid (ZrO$_2$-TiO$_2$) nanoparticle, since hybrid reinforcement showed a non-difference in impact strength when compared to TiO$_2$ nanoparticle (which showed the highest impact strength among the three) and the highest flexural strength when compared to ZrO$_2$ and TiO$_2$ nanoparticles, within the limitations of this study it can be concluded that hybrid reinforcement with ZrO$_2$ and TiO$_2$ nanoparticles can be considered a better reinforcement option compared to single reinforcement with ZrO$_2$ or TiO$_2$ nanoparticles if future studies on their other mechanical properties, physical and antimicrobial properties also prove the same.

**Limitations:-**
1. Hence further studies comparing the effects of these three nanoparticles namely ZrO$_2$, TiO$_2$ and its hybrid (ZrO$_2$-TiO$_2$) combination are required for a more definite conclusion.
2. Since the present study is an invi tro study, they are limited in their ability to predict the success of a material or technique in a clinical situation. Hence further invivo studies are needed. Studies comparing the effect of thermocycling on the flexural and impact strengths of heat cured denture base resins are also required.

**Conclusion:-**
Within the limitations of this study it can be concluded that:
1. The greatest impact strength of high impact acrylic resin was seen in reinforcement with TiO$_2$ nanoparticles when compared to reinforcement with zirconium dioxide nanoparticles and its hybrid (ZrO$_2$-TiO$_2$) combination.
2. The greatest flexural strength of high impact acrylic resin was seen in reinforcement with hybrid combination (ZrO$_2$-TiO$_2$) when compared to reinforcement with zirconium dioxide and titanium dioxide nanoparticles.

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