

**‘COMPARATIVE EVALUATION OF FLEXURAL STRENGTH  
OF HEAT POLYMERIZED POLYMETHYL METHACRYLATE  
DENTURE BASE MATERIAL REINFORCED WITH DIFFERENT  
PERCENTAGES OF SILANIZED ZIRCONIUM SILICATE  
NANOPARTICLES – AN *IN VITRO* STUDY’**

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# Contents

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Serial No .	Title	Page No.
1	Introduction	1
2	Aim and Objectives	7
3	Review of literature	9
4	Materials and Method	30
5	Results	42
6	Discussion	48
7	Summary	59
8	Conclusion	61
9	Bibliography	i
10	Tables	xiii
11	Graphs	xvi
12	Annexure	xvii

## List of photo plates

Fig No.	Title	Plate No.
1	Heat cure polymethyl methacrylate resin	I
2	Die stone	
3	Zirconium silicate nanoparticles	
4	Silane coupling agent	
5	Toluene	
6	Cold mould Seal (Separating medium)	
7	High accuracy balance	II
8	Ultrasonicator	
9	Magnetic stirrer	
10	Vacuum rotary evaporator	
11	Acrylizer with thermostat	
12	Universal testing machine	III
13	Rubber bowl, plaster spatula, lacron's carver and varsity flask & clamp	
14	Glass Beaker. Sterile Syringe, Petroleum jelly, camel hair brush, Sand paper, Porcelain jar, Dapen dish,	
15	Vernier caliper	
16	Brass metal dies	
17	Parafilm	
18	Hydraulic bench press	
19	Distilled water	
20	Preparation of gypsum mould to obtain specimens	IV
21	Silanization of zirconium silicate nanoparticles	
22	Testing of specimens	

<b>Fig No.</b>	<b>Title</b>	<b>Plate No.</b>
23	Group 1 : The control group Heat polymerized polymethyl methacrylate denture base material without reinforcement before and after testing of flexural strength	V
24	Group 2: Heat polymerized polymethyl methacrylate denture base material reinforced with 1.5% silanized zirconium silicate nanoparticles before and after testing of flexural strength	
25	Group 3: Heat polymerized polymethyl methacrylate denture base material reinforced with 2% silanized zirconium silicate nanoparticles before and after testing of flexural strength	
26	Group 4: Heat polymerized polymethyl methacrylate denture base material reinforced with 2.5% silanized zirconium silicate nanoparticles before and after testing of flexural strength	VI
27	Group 5: Heat polymerized polymethyl methacrylate denture base material reinforced with 3% silanized zirconium silicate nanoparticles before and after testing of flexural strength	
28	Group 6: Heat polymerized polymethyl methacrylate denture base material reinforced with 4% silanized zirconium silicate nanoparticles before and after testing of flexural strength	

## List of Tables

---

<b>Table No.</b>	<b>Title</b>	<b>Page No.</b>
1	Descriptive statistics for flexural strength	xiii
2	One-way analysis of variance for Flexural Strength across six groups	xiv
3	Individual pair –wise comparison of flexural strength between groups using Tukey’s post – hoc test	xv

## List of Graphs

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Graph No.	Title	Page No.
1	Mean and error bar for flexural strength according to study groups	xvi
2	Mean difference of Pair wise comparison of groups for flexural strength	

## List of Master Charts

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<b>Master Chart No.</b>	<b>Title</b>	<b>Page No.</b>
1	Flexural Strength test data	xvii

## List of Abbreviations Used

No.	Abbreviations	Full form
1	n	Number of specimens in each group
2	N	Newton
3	p value	Probability of happening of an event
4	S.D.	Standard deviation
5	ANOVA	Analysis of variance
6	MPa	Mega Pascal
7	<sup>0</sup> C	Degree Celsius
8	<sup>0</sup>	Degree
9	mm	Millimeter
10	i.e.	that is
11	PMMA	Polymethyl methacrylate
12	μ	Micron
13	Al <sub>2</sub> O <sub>3</sub>	Aluminium oxide
14	ZrSiO <sub>4</sub>	Zirconium Silicate
15	TiO <sub>2</sub>	Titanium dioxide
16	ZrO <sub>2</sub>	Zirconium Oxide
17	SiO <sub>2</sub>	Silicon dioxide
18	TMSPM	Trimethoxysilylpropylmethacrylate
19	Rpm	Rotations per minute
20	mins	Minutes
21	gms	Grams
22	Nps	Nanoparticles
23	ZS (in graph)	Zirconium silicate

# *Introduction*

*“To raise new questions, new possibilities, to regard old problems from a new angle, requires creative imagination and marks real advance in science.”*

**-Albert Einstein**

Edentulism is the result of loss of all permanent teeth. Tooth loss is regarded as mutilating, terminal outcome of a multifactorial process involving biologic processes (caries, periodontal diseases, pulpal pathology, trauma, oral cancer) as well as non-biological factors related to dental procedures (access to care, patient’s preferences, treatment options etc).<sup>1</sup>

Even though over a past few decades, there has been a steady decline in the rates of tooth loss, more than one-third (33.1 %) of those aged  $\geq 65$  years are edentulous. The number of edentulous people are expected to increase as a result of the strong increase in the aging population.<sup>2</sup> The need and demand for complete dentures will increase over the next 2 decades as this generation matures into the upper age groups.<sup>3</sup>

The treatment options for edentulous patients ranges from conventional complete dentures to fixed implant-supported restorations. In many cases conventional complete dentures still remain the treatment of choice due to medical and financial reasons. As the missing tooth structure is replaced by artificial materials it is very important that there has to be continuous research and development in the field of dental materials.<sup>4</sup>

Dentures are believed to have surfaced as a mode of treatment for replacing missing teeth since around 2500 BC.<sup>5</sup> In 8<sup>th</sup> century the Japanese mastered the art of woodcarving. Dentures carved from a single piece of wood were readily available and inexpensive.<sup>4</sup> Natural teeth were secured with the help of screws. George Washington had a set of dentures which were fabricated from wood.<sup>5</sup>

The pace of further development in the field of dental materials was slow until 17<sup>th</sup> century. Modern dentistry had been said to begin with **Pierre Fauchard** (1678-1761) who fabricated dentures from bone by measuring individual arches and shaped the bone to fit the arches. Dentures fabricated from ivory were stable in the oral environment and offered better esthetics but they were not readily available and were expensive.<sup>6</sup>

Although the art of firing porcelain was practiced in China in the 9<sup>th</sup> and 10<sup>th</sup> century, it was not until 1774 that **Alexis Duchateau**, a Parisian apothecary, dissatisfied with his own stained hippopotamus ivory denture, teamed up with Parisian dentist **Nicholas Dubois De Chemant** to fabricate a baked porcelain complete denture in a single block.<sup>5</sup> The advantages were that it could be shaped easily, ensured intimate contact with the underlying tissues, was stable, had minimal water sorption, smooth surfaces after glazing, less porosity, low solubility and could be tinted but its drawbacks were brittleness and difficulty in grinding and polishing. The first porcelain denture with artificial teeth was fabricated by **Loomis** (1854).<sup>4</sup>

Materials used in 19<sup>th</sup> century were Tortoise Shell (1850), GuttaPercha (1851), Vulcanite (1851), Cheoplastic (1856), Rose Pearl (1860), Aluminium (1867), Celluloid (1870).<sup>4</sup>

Materials used in 20<sup>th</sup> century were Bakelite (1909), Stainless steel (1921), Cobalt Chromium (1930), Acrylic Resin (1937), Self-cure Acrylic Resin, Epoxy Resin (1951), Polystyrene (1951), Nylon (1955), Polycarbonates (1967), High impact acrylic (1967), Visible L.C (1947), Pure Titanium (1998).<sup>4</sup>

During the latter part of the nineteenth century, polymers entered the field of denture base materials. With the introduction of polymethyl methacrylate (PMMA) by **Dr. Walter Wright** in 1937, PMMA has been the most widely used material for the fabrication of complete dentures. It has been the material of choice because of its biocompatibility, stability in the oral environment, superior esthetics, favorable working characteristics, processing ease, accurate fit, and inexpensive equipment's required for fabrication process.<sup>7,8</sup> However, because of certain drawbacks like poor

mechanical strength, low fatigue strength, brittleness, poor thermal conduction and low hardness, it is still far from ideal in fulfilling the mechanical requirements of the denture base material.<sup>9</sup>

Studies have shown that 68 % of the complete dentures fabricated, fractured within the first three years.<sup>10</sup> The fracture of acrylic resin dentures is an unresolved problem in removable prosthodontics. The midline fracture of a maxillary denture is most common and is often the result of flexural fatigue and deep incisal notching at the labial frenum.<sup>11</sup>

**Smith**<sup>7,12</sup> studied the practical situation with respect to the fracture of dentures and concluded that there are two types of failures.

- I. Outside the mouth which was caused by impact forces e.g accidental dropping of the denture while cleaning, insertion and removal.
- II. Inside the mouth, usually in function; this is probably due to a fatigue phenomenon, i.e. a low and repetitive stress rate.

Denture base material should have sufficient strength to withstand fracture while in service. Different physical properties can be used to assess the strength of denture base materials. The most common test is flexural strength i.e. the amount of force needed to deform the material to fracture or yield irreversibly.<sup>8</sup>

**Replacing PMMA with alternative material, chemically modifying it and reinforcing PMMA with other materials** are the three ways to improve the mechanical properties of PMMA.<sup>9</sup> Many attempts have been made to enhance the strength of heat polymerized acrylic resin denture base material. **Sehjpai and**

**Sood(1989)**<sup>13</sup> stated that PMMA reinforced with metal fillers like silver, copper and aluminium not only increases the strength but also provides radio-opacity to the heat polymerized denture base material.

Much attention has been directed towards the incorporation of inorganic nanoparticles into PMMA to improve its properties.<sup>14</sup> Recent developments of composite materials of great strength and low mass has made significant contribution in the field of dental material science. Ceramic fillers have low filler density when compared to metal fillers. Also they have the advantage of being white and therefore are less likely to alter the finished appearance of the denture base material.<sup>7</sup>

In nature, zirconia does not occur in a pure state. It can be found in conjunction with silicate oxide with the mineral name Zircon ( $ZrO_2 \times SiO_2$ )/ Zirconium silicate or as a free oxide ( $ZrO_2$ ). Zirconia possesses strong ionic inter-atomic bonding, giving rise to its desirable material characteristics. The high flexural strength and fracture toughness of zirconia is because of a physical property known as transformation toughening. The biocompatibility of zirconia has also been extensively studied.<sup>15</sup> The high surface area, fine size and homogenous distribution of nano-fillers improved the mechanical properties of PMMA and also increased its thermal stability when compared to pure PMMA. The properties of polymer nanocomposites depends on the type, size, shape, concentration of incorporating nanoparticles and their interaction with the polymer matrix.<sup>14</sup>

Silanes have the ability to bond inorganic particles to organic matrix resulting in improved mixing, better bonding and increased matrix strength.<sup>16</sup> Treating the surface (Silanization Process) of an inorganic particle with an organic substance is a

useful way to reduce its surface energy, increase dispersion homogeneity and thus improve the properties of the polymer nanocomposites.<sup>14</sup>

As limited amount of data is available in literature regarding the effect of different percentages of silanized zirconium silicate nanoparticles on flexural strength of heat polymerized PMMA, the purpose of this study is comparative evaluation of flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with different percentages of silanized zirconium silicate nanoparticles.

# *Aim and Objectives*

*“A goal properly set is halfway reached.” —Zig Ziglar*

## **Aim:**

‘To evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with different percentages of silanized zirconium silicate nanoparticles.’

## **Objectives:**

- 1) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material without reinforcement.

- 2) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 1.5% silanized zirconium silicate nanoparticles.
- 3) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 2% silanized zirconium silicate nanoparticles.
- 4) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 2.5% silanized zirconium silicate nanoparticles.
- 5) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 3% silanized zirconium silicate nanoparticles.
- 6) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 4% silanized zirconium silicate nanoparticles.
- 7) To compare the flexural strength of heat polymerized polymethyl methacrylate denture base material without reinforcement and reinforced with different percentages of silanized zirconium silicate nanoparticles.

## *Review of Literature*

*'You can't connect the dots looking forward; you can only connect them looking backwards. So you have to trust that the dots will somehow connect in your future.'*

*- Steve Jobs*

Polymethyl Methacrylate (PMMA), introduced by **Dr Walter Wright** in 1937, is still one of the most widely used material in the field of prosthetic dentistry. Despite its popularity which satisfies esthetics, simple processing and easy repair, its major drawback as a denture base material is low impact and flexural strength. This material is not ideal in every aspect and it is not one single desirable property but the combination of different properties that accounts for its popularity and wide usage.<sup>8,9</sup>

The clinical problems often experienced due to low flexural strength of heat polymerized acrylic resin denture base material has led to many scientific studies to improve mechanical properties of polymethyl methacrylate.

Studies have shown that 68% of the dentures fabricated, fractured within the first three years of its use.<sup>10</sup> Many trials have been made to enhance mechanical properties of denture base materials. Ceramic materials are biocompatible and also improve the mechanical properties.<sup>7</sup> Few of these materials have obtained promising results in improving the fracture resistance of heat polymerized acrylic resin denture base (PMMA). The reinforcement of polymers used in dentistry with these metal-composite systems has been of prime interest.

**Smith DC (1961)**<sup>12</sup> stated that one of the most practical deficiencies of a denture is fracture. Fracture can occur both; inside as well as outside the mouth. The most common are the midline fractures which act as fatigue fractures. It was found that, whether the denture fractures from accidental or masticatory cause, the strength of the denture has been inadequate in each case. It was also observed that the strength of a denture depends on the shape, residual stress, mechanical properties of the material and condition of loading. It is seen that the incisal notch in a maxillary denture acts as a crack initiator.

**Grantt AA and Greener EH (1967)**<sup>17</sup> evaluated the flexural property of polymethyl methacrylate by incorporating sapphire ( $\text{Al}_2\text{O}_3$ ) whiskers of various diameters. Two types of sapphire whiskers were used; one sapphire whisker of 1-10 $\mu$  in diameter, silanized and non-silanized was incorporated in a concentration of 8.3% by weight, the other type consisted of sapphire mixed grade fibres incorporated at 10%,

11% and 27% by weight of sapphire. Addition of sapphire whiskers to heat cured polymethyl methacrylate improved the physical properties. With large additions of mixed grade and smaller additions of graded sapphire whiskers the ultimate bending strength was approximately doubled and 25 % changes in the modulus and resilience were noted. The addition of silane increases the surface activity of whiskers permitting better transfer of stresses from polymethyl methacrylate to the whisker. They concluded that an enhancement of the flexural strength of denture base polymethyl methacrylate was possible through the whisker reinforcement with sapphire fibres.

**Hargreaves AS (1969)<sup>18</sup>** studied the prevalence of dentures fractures. She conducted a survey at Dundee Dental Hospital for 6 months and stated that during that period, there were 113 denture repairs cases, 68% of the dentures fabricated, fractured within the first three years. Women tend to have fractured the denture during eating. She found that habits such as pipe smoking, nail biting or pencil chewing did not play a significant part in denture fracture. This survey showed that upper dentures lost more teeth and fractured in the midline during mastication whereas lower dentures encountered a midline fracture after being dropped.

**Berry HH and Funk OJ (1971)<sup>19</sup>** stated that midline fracture of the denture is a commonly encountered problem. Breakage may be due to difficulty in cleaning, coughing which pushes the denture out of the mouth, lack of denture base material at the midline, greater than average biting force and dropping the denture accidentally. Breakage is more commonly seen in neuropsychiatric patients, especially those having neuromuscular disorders such as Huntington's chorea, hemiparalysis, muscular dystrophy and Parkinson's disease. They incorporated vitallium as a denture

strengthened. The reinforcements used were designed to retain all the qualities of the acrylic resin denture in addition to adding the needed strength to prevent denture breakage.

**Shreiber CK (1971)<sup>20</sup>** evaluated and compared the transverse strength of acrylic resin denture base materials, with and without carbon fibre reinforcement. Carbon fibres were in the form of untreated fibres, untreated chopped carbon fibres and surface treated carbon fibres. It was concluded that polymethyl methacrylate reinforced with surface treated carbon fibres had the greatest increase in transverse strength that exceeded the control group by 50%.

**Beyli MS and Fraunhofer JA (1981)<sup>11</sup>** analyzed the causes of fracture of acrylic resin dentures. 20 laboratories specialized in denture construction and repair were consulted for this survey. The result of this survey showed that the ratio of fracture of upper to lower denture was about 2:1. According to this study the most common causes of fracture appeared to be poor fit and lack of balanced occlusion. Midline fracture of a denture base is a flexural fatigue failure, resulting from cyclic deformation of the base during function. Any factor that exacerbates deformation of the base or alters its stress distribution will predispose the denture to fracture. A survey of denture fractures indicated that most of the failures occurred when there was deep notching at the midline in the labial frenum region and cracks initiate at the tip of the notch where there is high local stress concentration. Various methods to prevent denture fracture i.e. good processing technique, dentures with metal plates for patients with heavy occlusions, impact resistant polymers were suggested. Many midline fractures can be avoided by the application of established prosthodontic principles in constructing and

maintaining dentures. The most promising approach for preventing or reducing the incidence of this problem appears to be reinforcement in the anterior part of the palate of the denture.

**Carroll CE, Fraunhofer JA (1984)**<sup>21</sup> conducted a study to determine the effect of the use of commonly available materials to reinforce autopolymerizing acrylic resin. The acrylic resin was reinforced with flat, braided, two-strand brass wire (Great Lake Ortho Products, Inc., Buffalo, N.Y.) and one of four diameters of orthodontic wires (Unitek Corp., Monrovia, Calif.): 0.016, 0.025, 0.036, and 0.051 inch (0.41, 0.64, 0.91, and 1.30 mm). Statistically significant increase in strength were not obtained consistently until wires of 0.025 inch diameter were used. Clinically significant increase in strength may not be obtained until a wire of at least 0.036 inch diameter was used. The 0.051 inch diameter wire imparted increase in transverse strength that was clinically significant.

**Gutteridge DL (1988)**<sup>22</sup> studied the effect of including ultra-high-modulus polyethylene fibre on the impact strength of acrylic resin. He concluded that when concentration of fibre used was 1% there was significant increase in impact strength, significant increase was observed upto the concentration of 3% but there was no beneficial effect on impact strength beyond this concentration.

**Sejpal SP and Sood VK (1989)**<sup>13</sup> studied the effect of metal fillers on thermal conductivity, tensile strength, compressive strength and radio-opacity of PMMA. Silver, copper and aluminum particles of 10 microns were used as fillers. They were added to PMMA in percentages of 5, 10, 15, 20 and 25. The study concluded that with the increase in metal fillers concentration there was a progressive increase in

compressive strength of polymethyl methacrylate and decrease in tensile strength. The maximum increase in thermal conductivity was seen with aluminum, silver and copper fillers in 25% by volume concentration.

**Berrong JM, Weed RM and Young JM (1990)<sup>23</sup>** conducted a study on the fracture resistance of polymethyl methacrylate denture base resin reinforced with kevlar fibres embedded in the ratio of 0% (control), 0.5%, 1% and 2% by weight of PMMA resin specimen. All the specimens were subjected to impact testing. The result showed that all reinforced sample groups had greater fracture resistance than the unreinforced control group. They concluded that the use of upto 2% kevlar fibres as reinforcement might increase resistance of the resin denture base.

**Solnit GS (1991)<sup>24</sup>** concluded that:

1. Cloth-form glass fibers will strengthen PMMA when added to the monomer/polymer mixture cured in a pressure device.
2. Glass fibers in the loose form will significantly weaken PMMA when added to the monomer/polymer mixture cured in a pressure device.
3. Cloth-form glass fibers pretreated with silane coupling agent will significantly weaken PMMA when added to the monomer/polymer mixture cured in a pressure device.
4. Glass fibers in the loose form that are pretreated with silane coupling agent will strengthen PMMA when added to the monomer/polymer mixture cured in a

pressure device. Further investigation is necessary to ensure more predictable results.

5. Unreinforced PMMA cured in a pressure device results in the least variance in strength.

**Vallittu PK, Lasilla VP (1992)<sup>25</sup>** studied the effect of surface roughness of various metal wires on the fracture resistance of the acrylic test specimens. Metal strengtheners used were semicircle wire (0.1-0x2.0mm), braided wire plate (0.8x2.4mm) and clasp wire (1.0 mm). The sandblasting was done using aluminium oxide particles with grain sizes, 50 and 250 $\mu$ m, and the air pressure applied was 5.5 bar. Roughening of wires was done by grinding them with a heatless stone and with a 0.6 mm separating disc. All the wires were cleaned in water with an ultrasonic cleaning device. The semicircular wire had maximum effect on the fracture resistance of the specimens. The best results were obtained by sandblasting procedure. The resistance was not influenced by the grain size of the sand (50 $\mu$ m or 250  $\mu$ m).

**Vallittu PK and Lasilla VP (1992)<sup>26</sup>** studied the effect of different types of commonly used metal wires and glass fibres, as well as carbon and aramid fibres on the fracture resistance of polymethyl methacrylate. Metal strengtheners used were remanium's spring hard clasp wire, semi-circular wire, braided wire plate, stainless steel mesh and they were further divided into: glossy and sandblasted. Fibres were divided into 2 groups untreated and silanized. They concluded that all metal strengtheners increased the fracture resistance except stainless steel mesh. They found that the unsilanized glass fibres slightly weakened the test specimens. However, the

weakening was not statistically significant when compared to the silanized glass fibres, which had a significant strengthening effect.

**Ladizesky NH, Cheng YY, Chow TW, Ward IM (1993)<sup>27</sup>** evaluated 3 mechanical properties i.e. flexural strength, flexural modulus and impact strength of acrylic resins reinforced with woven highly drawn linear polyethylene fibres (HDLPE). They concluded that by incorporation of the polyethylene fibres in woven form, substantial improvements in impact strength was obtained.

**Ladizesky NH, Cheng YY, Chow TW, Ward IM (1993)<sup>28</sup>** concluded that when chopped high performance polyethylene fibres were incorporated 30% by volume in acrylic denture base resins, it resulted in substantial improvement in several important properties

- (1) Flexural stiffness and impact strength were higher;
- (2) Water sorption, polymerization shrinkage, dimensional changes during water immersion significantly decreased,
- (3) It also removed the weakening effect of anatomical features such as the frenal notch.

**Vallittu PK (1993)<sup>29</sup>** studied the effects of two different silane compounds on the adhesion between different fibres and acrylic resin. The fibres used were glass, carbon and aramid fibres and each type of fibres were either untreated or treated with silane agents. The fibres were studied by a scanning electron microscope (SEM) to establish the adhesion between the fibres and acrylic resin and the fracture resistance

of the specimens were tested. The results showed that the adhesion between the fibres and acrylic resin improved with silanization of glass and aramid fibres. SEM photographs taken confirmed these findings.

**Vallittu PK (1993)<sup>30</sup>** studied the effect of metal wire bonding to acrylic resin on the fracture resistance of an acrylic denture base material construction. Two different bonding methods were tested: Silicoating (group B19) and Eudicolle (group B18). The fracture resistance of the test specimens reinforced with sandblasted metal wires were higher than the resistance of the control specimens. The bonding method of Silicoater increased the resistance significantly whereas the bonding compound of Eudiclle did not increase the fracture resistance when compared with unsilanized strengtheners. The different positions of the wires had no effect on the fracture resistance.

**Marie M et al (1994)<sup>31</sup>** evaluated four physical and mechanical properties: thermal conductivity, impact strength, compressive strength and warpage by adding tin or aluminium powder with particle size of 10µm to heat cure acrylic resin in a concentration of 5% by volume. The addition of 5% by volume of both the metal powders to polymethyl methacrylate (PMMA) improved the four tested properties. However, aluminium powder was superior to tin powder in improving thermal conductivity of PMMA and decreasing its warpage. Tin powder was superior to aluminium powder in improving the impact and compressive strength of PMMA. The use of these metal-filled resin is recommended in the areas where it is not displayed because both metal powders caused undesirable discoloration to the heat cure acrylic resin.

**Vallittu PK, Vojtkova H, Lassila V (1995)**<sup>32</sup> compared the impact strength of heat-cured acrylic resin specimens reinforced with 1.0-mm-diameter steel wire and continuous E-glass fibres. When compared to unreinforced specimen it was found that both types of reinforcements increased the impact strength of the resin. They concluded that concentrations of glass fibre greater than 25 wt% yield better impact strength than steel wire 1.0 mm in diameter.

**Stipho HD (1998)**<sup>33</sup> investigated the transverse strength, maximum deflection, and modulus of elasticity of repaired acrylic resin joints reinforced with different concentrations of glass fibres to the weight of the powder/ liquid mix (0%, 1%, 2%, 5%, 10%, and 15%). Transverse strength, maximum deflection and the stiffness of all joints were significantly lower after the repair. Among the groups tested, the specimens treated with 1% glass fiber displayed the highest transverse strength before and after repair. Modulus of elasticity of the repaired 1% fiber concentration units was improved by approximately 25% when compared to specimens which were repaired but untreated with glass fiber (0% fiber).

**Vallittu PK (1999)**<sup>34</sup> evaluated the flexural properties of acrylic resin polymers reinforced with unidirectional and woven glass fibres. Heat-curing and autopolymerizing denture base polymers were reinforced with continuous unidirectional and woven pre-impregnated glass fibers. He concluded that within the limitation of this study,

- 1] Increase in transverse strength and flexural modulus of the polymers was seen with Stick reinforcement.

- 2] Increased strain at fracture of the polymers was seen with Stick Net reinforcement.
- 3] SEM examination revealed that fibers of Stick and Stick Net were well impregnated with the resin of polymer matrix.

**Kanie T, Fujii K, Arikawa H and Inoue K (2000)**<sup>35</sup> studied the reinforcing effect of woven glass fibres on deflection, flexural strength, flexural modulus and impact strength of acrylic denture base polymer. The flexural strength and deflection was significantly higher in specimens reinforced with silanized glass fibres of 1 mm thickness than those of unreinforced specimens. Also, the impact strength was significantly higher in specimens reinforced with silanized glass fibre of 2 mm thickness than that of unreinforced specimens.

**Jagger D et al (2001)**<sup>36</sup> studied the effect of the addition of surface treated chopped and continuous poly (methyl methacrylate) fibres on some properties of acrylic resin. Properties like transverse and impact strengths were investigated. The fibres were added in three arrangements (i) a single unidirectional layer (longitudinal in the direction of the specimen), (ii) two unidirectional layers and (iii) two layers in cross ply (an inferior layer at 0° and a superior layer at 90° to the direction of the length of the specimen). The addition of surface treated chopped or continuous fibres to acrylic resin did not improve the transverse or impact strengths over unmodified specimens, therefore cannot be recommended as a method of reinforcement.

**Foo H et al (2001)**<sup>37</sup> evaluated the effect of unidirectional poly-aramid fibre reinforcement on the transverse strength of intact and repaired heat-polymerized

denture base acrylic resins. The denture base resins tested were Acron MC (microwave polymerized resin), Lucitone 199 (butyl rubber reinforced high impact resin) and Microlon (conventional denture base resin). They concluded that poly-aramid reinforcement significantly increased the transverse strength of intact heat-polymerized PMMA resins when compared to the control group. Use of poly-aramid reinforcement in repair of unreinforced PMMA and poly-aramid reinforced PMMA did not significantly increase transverse strength.

**Chen S, Liang W, Yen PS (2001)**<sup>38</sup> evaluated the mechanical properties of acrylic resin reinforced with three types of fibre- Polyester fibres (PE), Kevlar fibres (KF), and glass fibres (GF) cut into 2, 4 and 6 mm lengths and incorporated at concentrations of 1, 2 and 3% (w/w). Polyester fibre and Kevlar fibre both improved the mechanical properties, but the polyester fibre was superior in aesthetics. There was significant increase in the impact strength of acrylic resin reinforced with 3% (w/w) of 6 mm polyester fibre when compared to other formulations. In this study they concluded that the optimum formulation to reinforce acrylic resin for improved strength is by incorporation of 3%, 6 mm length Polyester fibers.

**Aydin C, Yilmaz H, Çağlar A (2002)**<sup>39</sup> investigated the effect of a glass fibre reinforcement system on the flexural strength of three different denture base resins. Three denture base resins selected for testing: (1) heat-cured resin (QC 20 De Trey/Dentsply), (2) an autopolymerized resin (Vertex, Dentimex), and (3) a light-activated resin (Triad, Dentsply). Stick (S) and Stick Net (SN) (Stick Tech) were used to reinforce the three denture base polymers. The results showed that different denture base resins

when reinforced with the glass fibre may be a useful means of improving the flexural strength thereby improving the performance of dental acrylic resins.

**Kim SH, Watts DC (2004)**<sup>40</sup> studied the effect of glass fibre reinforcement on the impact strength of high impact acrylic resin maxillary dentures. They drew the following conclusion: at crack initiation and at complete fracture, the impact strength and crack propagation energy of high impact acrylic maxillary complete denture reinforced with woven E-glass fibres was higher than that of the unreinforced denture.

**Matinlinna J et Al (2004)**<sup>41</sup> reviewed silanes and their clinical application in dentistry. They stated silanes, hybrid organic-inorganic compounds, function as mediators and through dual reactivity promote adhesion between dissimilar, inorganic and organic matrices. They are called primers, coupling agents, or sizes, depending on their function and substrates. Methacryloxypropyltrimethoxysilane (or 3-trimethoxysilylpropyl methacrylate [MPS]) is most commonly applied silane in dental laboratories and chairside. MPS is used to optimize and promote the adhesion, through chemical and physical coupling, between metal-composite, ceramic-composite and composite-composite.

**Vojdani M, Khaledi AAR. (2006)**<sup>42</sup> conducted a study in which they observed that the transverse strength of heat polymerized denture base resin was considerably enhanced by incorporating either metal wires or glass fibres. S fibre reinforcement increased the transverse strength of PMMA upto 50%, SN fibre reinforcement increased the transverse strength upto 30% and reinforcing with wire increased the transverse strength of PMMA upto 14%. Moreover, the flexural strength of specimens

reinforced with continuous unidirectional glass fibres was significantly higher than that of metal wire or woven fibre reinforcements.

**Nakamura M, Takahashi H, Hayakawa I (2007)**<sup>43</sup> evaluated flexural strengths and moduli by reinforcing denture base resins with high short-rod fibre. A commercial PMMA (AC; average particle size, 150µm) and an industrial PMMA powder (MB; average particle size, 4µm) was used. Short-rod glass fibres were mixed with two powders at a mass ratio of 0–50%. The flexural strength of MB composites increased significantly at fibre contents exceeding 40%. The flexural moduli was significantly greater for AC and MB composites at fibre contents exceeding 20% than those of control group respectively.

**Dogan M et al (2008)**<sup>44</sup> evaluated flexural properties of a denture base resin material reinforced with esthetic fibres i.e. glass, rayon, polyester, nylon 6 and nylon 6,6 fibres at 3% concentration by weight and in 2, 4 and 6 mm length. The highest flexural strength was shown by specimens reinforced with nylon 6, 6 fibres of 6 mm length.

**Ellakwa AE, Morsy MA, El-Sheikh AM (2008)**<sup>8</sup> conducted a study to evaluate the effect on the flexural strength and thermal diffusivity of heat-polymerized acrylic resin on adding 0%, 5%, 10%, 15%, and 20% by weight aluminium oxide powder (Al<sub>2</sub>O<sub>3</sub>). The study concluded that incorporating Al<sub>2</sub>O<sub>3</sub> powder from 5% to 20% by weight into conventional heat-polymerized denture base resin resulted in an increase in both flexural strength and thermal diffusivity over control samples. The flexural strength increased significantly after incorporation of 10% Aluminium oxide powder. The highest mean flexural strength was seen with addition of 15% by weight of

aluminium oxide powder. Thus, increasing the flexural strength and heat transfer characteristics of the acrylic resin base material could result in more patient satisfaction.

**Dagar SR, Pakhan AJ, Thombare RU, Motwani BK (2008)**<sup>45</sup> evaluated the flexural and impact strength of commercially available heat polymerizing PMMA denture base resin reinforced with glass and nylon fibres. In this study it was concluded that fiber-reinforced specimens were more resistant to impact and flexural fatigue than conventional PMMA specimens. When compared with nylon fiber reinforcement it was found that glass fiber reinforcement considerably improves both impact and flexural strengths of denture base resin. Silane-impregnated glass fiber reinforcement suits best to increase the flexural and impact strengths of heat-polymerized PMMA denture base resin.

**Ayad NM, Badawi MF, Fatah AA (2008)**<sup>46</sup> evaluated the effect of reinforcing high-impact acrylic resin (Metrocyl HI) with zirconia powder in two different concentrations (5% and 15%) on the impact strength, transverse strength, water sorption, surface hardness and solubility. They concluded that the addition of zirconia resulted in a highly significant increase in transverse strength of high-impact acrylic resin when compared to control samples by a factor of 29% and 76% in a concentration of 5% and 15% respectively. This increase was directly proportional to the concentration of zirconia. There was no significant difference in impact strength, surface hardness and water solubility when compared to zirconia free high impact resin.

**Vojvodic et al (2009)**<sup>47</sup> studied the effect of different glass fibers (“dental” and “industrial” origin) on flexural strength values of dental base polymers. Flexural strength of the control specimens was 91.76 MPa, while there was a rise of flexural

strength values (103.10-163.88 MPa) in specimens reinforced with glass fibers irrespective of type (“dental” or “industrial”) fibers used but due to better investment to benefit ratio “industrial” glass fibers could be recommended for dental laboratory use.

**Abdulhamed AN, Mohammed AM (2010)**<sup>48</sup> evaluated the thermal conductivity, impact and tensile strength of alumina reinforced heat cure acrylic resin. Alumina powder was added to PMMA powder by weight in three different percentages 5%, 7.5% and 10%. The study concluded that the addition of Al<sub>2</sub>O<sub>3</sub> powder to acrylic resin improves the thermal conductivity, decreases both tensile and impact strength values. Further there was an increase in surface hardness. Water sorption and solubility were decreased while surface roughness was not affected with small percentages of alumina.

**Arora N, Jain V, Chawla A, Mathur VP (2011)**<sup>49</sup> studied the effect of adding silver filler particles and sapphire (aluminum oxide) on the flexural strength, thermal diffusivity and water sorption of polymethyl methacrylate (PMMA) resin. Silver filler particles and sapphire (aluminum oxide) were added in 25% by weight of acrylic resin. The study concluded that as compared to silver fillers, sapphire fillers were better for the reinforcement of polymethyl methacrylate resin. This is because they have low density, are highly esthetic and bring about an improvement in the mechanical properties (flexural strength and fatigue strength) and thermal properties (thermal diffusivity) of polymethylmethacrylate (PMMA) resin.

**Ihab NS, Moudhaffar M (2011)**<sup>50</sup> studied the effect of addition of modified nano-zirconium oxide (ZrO<sub>2</sub>) on strength and radio-opacity of heat cured acrylic

denture base material. The nanoparticles were coated with a layer of trimethoxysilypropylmethacrylate (TMSPM). They were sonicated in monomer (MMA) in different percentages 2%, 3%, 5% and 7% by weight. It was found that maximum increase in transverse strength, impact strength, and radio-opacity was observed in denture base reinforced with 5wt% of nano-ZrO<sub>2</sub>.

**Alhareb AO, Ahmad ZA (2011)**<sup>51</sup> evaluated the effect of 5wt% of Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> (80:20 ratio) reinforcement on the fracture toughness, flexural, and tensile properties of PMMA denture base. They concluded that the PMMA reinforced with Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> improved the tensile modulus, fracture toughness and flexural properties of the denture base material.

**Saritha MK, Shadakshari S, Nandeeshwar DB (2012)**<sup>7</sup> conducted an *in vitro* study to investigate the flexural strength of conventional heat polymerized denture base resin reinforced with 5%, 10% and 15% by wt. of aluminium oxide powder. They concluded that incorporation of 10% (group-C) and 15% (group-D) by wt. aluminium oxide powder to heat cure denture base resin significantly increased the flexural strength of denture base resin. The highest flexural strength was found with incorporation of 15% by wt aluminum oxide powder to heat cure denture base resin.

**Yadav P, Mittal R, Sood VK, Garg R (2012)**<sup>52</sup> studied the effect of incorporating metal filler particles on different strengths and thermal conductivity of polymethyl methacrylate (PMMA). The study was carried out in two parts. Part 1 was an *in vitro* investigation regarding the effect of incorporating metal fillers (aluminum and silver) in the concentrations of 10%, 20%, and 30%, by volume on the tensile, compressive and flexural strength of PMMA. Part 2 of the study comprised of clinical

evaluation of the thermal perception by 10 edentulous patients. Each patient was given two sets of complete dentures, one fabricated with unfilled PMMA and another with 20% aluminium particle filled PMMA on the palatal portion of the maxillary denture. They concluded that compressive strength increased progressively on increasing the filler concentration for both silver- and aluminum-filled PMMA. At 30% concentration silane-treated metalized PMMA showed reduction in tensile and flexural strength and incorporating metal filler particles led to an appreciable increase in thermal perception by the participants of this study.

**Ihab NS, Hassanen KA, Ali N.A (2012)<sup>14</sup>** evaluated impact strength, tensile strength and color stability of heat polymerized denture base reinforced with silanated and non-silanated zirconium oxide ( $ZrO_2$ ) nanofillers. Silanization was done by coating with a layer of trimethoxysilypropylmethacrylate (TMSPM) and nanoparticles were sonicated in monomer (MMA) in two percentages of 3% and 5% by weight of polymer. The maximum increase in impact strength was observed in PMMA reinforced with 5%wt of silanated  $ZrO_2$  nano-fillers. Silanized  $ZrO_2$  nano-fillers were effective in improving impact strength while it was not effective in improving the tensile strength. Also, significant color differences were seen between control group and specimens incorporated with zirconium oxide nano-fillers in different immersion solutions.

**Asar NV, Albayrak H, Korkmaz T, Turkyilmaz I (2013)<sup>53</sup>** evaluated the effect of addition of different types and amounts of the metal oxides on mechanical and physical properties of heat cured PMMA. They concluded that addition of  $Al_2O_3$ ,  $TiO_2$  and  $ZrO_2$  fillers resulted in significant increase in impact strength and fracture toughness and significant decrease in water sorption and solubility. Therefore,

reinforcing heat cure PMMA with certain amounts of metal oxides may be useful in preventing fracture of denture bases and unwanted physical changes resulting from oral fluids clinically.

**Sodagar A et al (2013)**<sup>54</sup> evaluated the effect of addition of TiO<sub>2</sub> and SiO<sub>2</sub> nano-particles on flexural strength of polymethyl methacrylate acrylic resins. They were divided into seven groups: control group and AR containing nano TiO<sub>2</sub> (2), SiO<sub>2</sub> (2) and TiO<sub>2</sub> (2) with SiO<sub>2</sub> (2) in two concentration of 1% and 0.5%. The maximum mean flexural strength (43.5 MPa) was seen with the control group. Incorporation of nano-particles into polymethyl methacrylate acrylic resin caused these particles to agglomerate and aggregate, adversely affecting the flexural strength of the final products and this effect was directly associated with the concentration of nano-particles.

**Ahmed MA, Ebrahim MI (2014)**<sup>55</sup> studied the effect of addition of zirconium oxide (ZrO<sub>2</sub>) nano-fillers powder in different concentrations (1.5%, 3%, 5% and 7%) on the flexural strength, fracture toughness, and hardness of heat-polymerized acrylic resin. Maximum increase in flexural strength was observed in heat-polymerized acrylic resin reinforced with 7% of zirconium oxide nanofillers.

**Asopa V et al (2015)**<sup>56</sup> evaluated and compared the transverse strength, impact strength, surface hardness and water sorption of 10% and 20% zirconia (ZrO<sub>2</sub>) reinforced high impact acrylic resin. They concluded that there was increase in transverse strength on addition of zirconium oxide as a filler in the high impact acrylic resin. On the other hand, impact strength and surface hardness of the zirconia reinforced specimens were found to have relatively lesser values when compared to control

specimens. Water sorption of the zirconia reinforced specimens was found to increase but was within the limit of ADA Specifications No. 12.

**Kareem S, Moudhaffer M (2015)**<sup>57</sup> studied the effect of silanized zirconium silicate nanopowder reinforcement on some mechanical and physical properties of heat cured poly (Methyl Methacrylate) denture base material. Silanization was done by coating zirconium silicate nanoparticles with a layer of trimethoxysilylpropylmethacrylate (TMSPM). Addition of silanized zirconium silicate nano fillers was done in two groups -1% and 1.5% by weight of polymer. They concluded that the maximum increase in impact strength, transverse strength, and surface hardness was observed in denture base nanocomposites containing 1.5%  $ZrSiO_4$  nanoparticles. In addition, highly significant decrease in water sorption and solubility and non-significant increase in surface roughness was also noticed.

**Gad et al (2017)**<sup>58</sup> reviewed the effect of fibers, fillers, and nanofillers addition on poly(methyl methacrylate) (PMMA) properties. He concluded that

- There was significant improvement in the mechanical properties of PMMA when reinforced with glass fibres. Natural fibers (OPEFB) and vegetable fibers can be used, but further studies are needed.
- Addition of nanoparticles and nanotubes to denture base materials results in enhancement of mechanical properties of PMMA, depending on the application and manipulation.
- Silane coupling agent improves the bonding between fillers and the resin matrix and thus bring about an improvement in mechanical properties of the resins.

- Hybrid fiber, hybrid fillers, or hybrid fiber and filler are the newest reinforcement system and they may considerably enhance the properties of PMMA.

**Tamore SH, Jyothi K S, Muttagi S, Gaikwad AM (2018)<sup>59</sup>** evaluated the flexural strength of repaired heat-polymerized acrylic resin with different percentages of aluminium oxide ( $\text{Al}_2\text{O}_3$ ) added to the repair resin and also the effect of two different surface treatments on the flexural strength of repaired heat-polymerized acrylic resin. Fifty specimens of heat-polymerized acrylic resin were prepared and  $\text{Al}_2\text{O}_3$  <50 nm particle size was silanized using metal alloy primer before incorporation in polymer. Autopolymerizing acrylic resin which was used as repairing material was reinforced with two different percentages of  $\text{Al}_2\text{O}_3$  nanoparticles 1% and 1.5%. They concluded that repair resin incorporated with 1.5%  $\text{Al}_2\text{O}_3$  in the group surface treated with silicon carbide paper improved the flexural strength of denture base resin. Scanning electron microscope showed proper filler distribution and deep penetration within the polymer matrix in the same group.

## *Materials and Method*

*“Our goals can only be reached through a vehicle of a plan, in which we must fervently believe, and upon which we must vigorously act. There is no other route to success.” — Pablo Picasso, painter*

Polymethyl methacrylate (PMMA) introduced by **Dr. Walter Wright** in 1937 is the most widely used material for fabrication of complete dentures. It has been the material of choice for fabrication of complete dentures because of its biocompatibility, favorable working characteristics, accurate fit, stability in the oral environment, processing ease, superior esthetics and use of inexpensive equipments. It has certain drawbacks like poor mechanical strength, low fatigue strength, brittleness, poor thermal conduction and low hardness.<sup>8,9</sup> Flexural Fatigue which occurs after repeated flexing when subjected to loads results in fracture of the denture.<sup>60</sup> Hence the ability to resist

catastrophic failure under a flexural load depends on the ultimate flexural strength of a material. High flexural strength is crucial to the long term success of dentures.<sup>10</sup> Modifications can be done in the composition of conventional acrylic resin denture base material to achieve this purpose.

This *in-vitro* study was done to evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with different percentages of silanized zirconium silicate nanoparticles.

**Material and methods have been divided under the following heads:**

- I) Materials**
- II) Armamentarium and equipments**
- III) Method**

**I) Materials (PLATE I)**

<b>SR. NO.</b>	<b>MATERIALS</b>	<b>MANUFACTURER</b>	<b>BATCH NO.</b>
1	Heat polymerized acrylic resin (Fig.1 )	DPI Heat Cure, (Dental products of India Ltd)	10111
2	Die stone (Fig.2 )	Ultrarock; Kalabhai Karson Pvt Ltd, India	161003
3	Zirconium silicate nanoparticles (Fig. 3)	Nanoshell	NS6130-01-180
4	Silane coupling agent	Sigma Aldrich	440159

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	(3- Trimethoxypropylsilylmethacrylate TMPSM) ( Fig 4)		
5	Toluene ( Fig 5)	Emplura R	108323
6	Cold mould seal (separating medium) (Fig. 6)	DPI(Dental products of India Ltd)	8117

## **II) Armamentarium and equipments (PLATE II & III)**

1. High accuracy balance (Fig.7 )
2. Ultrasonicator (Fig.8 )
3. Magnetic stirrer (Fig. 9)
4. Vacuum rotary evaporator (Fig10.)
5. Acrylizer with thermostat (Fig.11 )
6. Universal testing machine (Fig.12 )
7. Rubber bowls and plaster spatula ( Fig.13)
8. Varsity flasks and clamps (Fig13 )
9. Sand paper (No. 120) (Fig.14 )
10. Camel hair brush (Fig. 14)
11. Glass Beaker (Fig.14)
12. Sterile Syringe (Fig.14 )
13. Mixing spatula (Fig.14 )
14. Petroleum jelly (Fig.14 )
15. Porcelain jar and Dapen dish ( Fig. 14)
16. Vernier caliper (Fig.15)
17. Brass metal dies (Fig.16 )

18. Para-film (Fig.17 )
19. Hydraulic bench press (Fig.18 )
20. Distilled water (Fig.19 )

### **III) Methodology (PLATE IV)**

The basic methodology consisted of –

- a) Die preparation
- b) Silanization of zirconium silicate nanoparticles
- c) Preparation of gypsum moulds for fabrication of specimens
- d) Preparation of heat polymerized polymethyl methacrylate denture base specimens
- e) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 1.5% silanized zirconium silicate nanoparticles
- f) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 2% silanized zirconium silicate nanoparticles
- g) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 2.5% silanized zirconium silicate nanoparticles
- h) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium silicate nanoparticles
- i) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 4 % silanized zirconium silicate nanoparticles
- j) Testing of specimens for flexural strength.

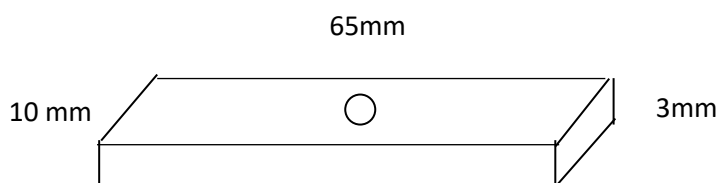
A total of 90 specimens were prepared with each group having 15 specimens.

The specimens were divided under the following groups:-

Group 1	The control group; heat polymerized polymethyl methacrylate denture base specimens without reinforcement. (n=15)
Group 2	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 1.5% silanized zirconium silicate nanoparticles (n=15)
Group 3	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 2% silanized zirconium silicate nanoparticles (n=15)
Group 4	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 2.5% silanized zirconium silicate nanoparticles (n=15)
Group 5	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium silicate nanoparticles (n=15)
Group 6	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 4% silanized zirconium silicate nanoparticles (n=15)

**a) Die preparation:**

Metal dies were fabricated to prepare moulds for the fabrication of heat polymerized polymethyl methacrylate denture base material specimens. Three brass metal dies of dimension 65 mm in length, 10 mm in width, and 3 mm in height (65×10×3) were fabricated. (ISO 1567 standard) .<sup>8,61</sup>



These fabricated metal dies had a threaded hole at the centre. These holes were of 5 mm in diameter and 3 mm in depth. Screws were used to engage these threaded holes to facilitate easy removal of dies from the stone mold.

**b) Silanization of zirconium silicate nanoparticles**

Pure toluene solvent in the amount of 175ml and 25gms zirconium silicate nanoparticles ( $ZrSiO_4$ ) were placed in the glass beaker of capacity of 250ml and sonicated by ultrasonic probe for 20 mins. Then magnetic stirrer was placed in the beaker. Then 1.25ml (5% wt to nano-filler) of silane (3-Trimethoxypropylsilyl methacrylate TMPSM) was added dropwise by using sterile syringe under rapid stirrer. The beaker was covered with parafilm and the slurry was left for two days. The slurry was placed in rotary evaporator under vacuum of 60°C, rotation of 150 rpm for 30 mins to remove toluene solvent. Finally, the silanated nanoparticles were made moisture free

by placing in vacuum oven (20 hours at 60°C) and then stored at room temperature before use.<sup>57</sup>

**c) Preparation of gypsum mould for fabrication of specimens**

Preformed brass metal dies were used to prepare gypsum moulds. Before investing them, the threaded holes on the dies were blocked with carding wax. A thin layer of petroleum jelly was applied on three metal dies which were then invested in the lower half of the varsity flask. Die stone was used for base flasking and care was taken to embed half the thickness of the metal die in it.<sup>62</sup> Once the investment material had set, a thin layer of petroleum jelly was applied to the metal dies and to the investment material and then the counter flasking was done. The flasks were closed to ensure metal to metal contact between the base of the flask and its counterpart. After the investment material had set (1 hour)<sup>63</sup> the flasks were opened and the carding wax within the holes was removed. The dies were engaged with a screw and gently teased out. The moulds were then immersed in hot water to remove any traces of petroleum jelly, wax and also to facilitate application of separating medium. These mould thus obtained were used for the fabrication of heat polymerized acrylic resin denture base material specimens (PMMA).

**d) Preparation of heat polymerized polymethyl methacrylate denture base specimens without reinforcement (n=15)**

15 specimens were prepared using conventional heat polymerized denture base material (PMMA).

Monomer and polymer were mixed in ratio of 1:2.5 by weight as per manufacturer's recommendations.<sup>64</sup> The materials were weighed using electronic balance of high accuracy. 7.5gms of polymer powder and 3ml of monomer was used for preparing 3 specimens. Packing was done at dough stage, following which trial closure was performed. Final closure was done under a hydraulic bench press at a pressure of 3000psi for 3 mins (according to the manufacturer). The flask was clamped and maintained under pressure for 1 hour.<sup>63</sup> It was then immersed in water in an acrylizer at room temperature. The temperature was raised slowly upto 74<sup>0</sup>C and was held for 2 hours. The temperature was then raised to 100<sup>0</sup>C and was maintained for 1 hour.<sup>65</sup> After completion of this short curing cycle, the flask was removed from the water bath and allowed to bench cool at room temperature prior to deflasking.<sup>65</sup>

The polymerized specimens were carefully removed and specimens with defects were discarded. Finishing of the specimens was done using sand paper (No. 120). The finished specimens were stored in distilled water for 1 week at room temperature.<sup>7,61</sup>

**e) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 1.5% silanized zirconium silicate nanoparticles (n=15)**

15 specimens were prepared using conventional heat polymerized denture base material (PMMA) reinforced with 1.5% silanized zirconium silicate nanoparticles.

For complete homogenous dispersion, 1.5% silanized zirconium silicate nanoparticles were added to the monomer.<sup>57</sup> As per group 1, same proportion was

followed for fabrication of specimens. 7.388gms of polymer powder, 3ml of monomer and 0.112gms of silanized zirconium silicate nanoparticles were taken for fabrication of 3 specimens. An electronic balance of high accuracy was used to weigh the materials.

Silanized zirconium silicate nanoparticles were well dispersed in monomer by using an ultrasonicator. The ultrasonication was done at 120W, 60KHz for 3 minutes. This allowed the homogenous dispersion of silanized zirconium silicate nanoparticles in the monomer.<sup>57</sup>

Immediately to this suspension, polymer powder was added gradually to reduce the possibility of particle aggregation and phase separation. Mixing was done according to manufacturer's instructions. Packing, curing, deflasking and finishing was done in the same manner as that for fabrication of heat polymerized polymethyl methacrylate denture base specimens (Group 1). Specimens with defects were discarded. The finished specimens were stored in distilled water for 1 week at room temperature.<sup>57</sup>

**f) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 2% silanized zirconium silicate nanoparticles (n=15)**

15 specimens were prepared using conventional heat polymerized denture base material (PMMA) reinforced with 2% silanized zirconium silicate nanoparticles.

For complete homogenous dispersion, 2% silanized zirconium silicate nanoparticles were added to the monomer.<sup>57</sup> As per group 1, same proportion was followed for fabrication of specimens. 7.35gms of polymer powder, 3ml of monomer and 0.15gms of silanized zirconium silicate nanoparticles were taken for fabrication of

3 specimens. As per group 1, same proportion and procedure was followed for fabrication of specimens reinforced with 2% silanized zirconium silicate nanoparticles.

The finished specimens were stored in distilled water for 1 week at room temperature.<sup>57</sup>

**g) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 2.5% silanized zirconium silicate nanoparticles (n=15)**

15 specimens were prepared using conventional heat polymerized denture base material (PMMA) reinforced with 2.5% silanized zirconium silicate nanoparticles.

For complete homogenous dispersion, 2.5% silanized zirconium silicate nanoparticles were added to the monomer.<sup>57</sup> As per group 1, same proportion was followed for fabrication of specimens. 7.313gms of polymer powder, 3ml of monomer and 0.187gms of silanized zirconium silicate nanoparticles were taken for fabrication of 3 specimens. As per group 1, same proportion and procedure was followed for fabrication of specimens reinforced with 2.5% silanized zirconium silicate nanoparticles.

The finished specimens were stored in distilled water for 1 week at room temperature.<sup>57</sup>

**h) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium silicate nanoparticles (n=15)**

15 specimens were prepared using conventional heat polymerized denture base material (PMMA) reinforced with 3% silanized zirconium silicate nanoparticles.

For complete homogenous dispersion, 3% silanized zirconium silicate nanoparticles were added to the monomer.<sup>57</sup> As per group 1, same proportion was followed for fabrication of specimens. 7.275gms of polymer powder, 3ml of monomer and 0.225gms of silanized zirconium silicate nanoparticles were taken for fabrication of 3 specimens. As per group 1, same proportion and procedure was followed for fabrication of specimens reinforced with 3% silanized zirconium silicate nanoparticles.

The finished specimens were stored in distilled water for 1 week at room temperature.<sup>57</sup>

**i) Preparation of heat polymerized polymethyl methacrylate denture base specimens reinforced with 4% silanized zirconium silicate nanoparticles (n=15)**

15 specimens were prepared using conventional heat polymerized denture base material (PMMA) reinforced with 4% silanized zirconium silicate nanoparticles.

For complete homogenous dispersion, 4% silanized zirconium silicate nanoparticles were added to the monomer.<sup>57</sup> As per group 1, same proportion was followed for fabrication of specimens. 7.2gms of polymer powder, 3ml of monomer and 0.3gms of silanized zirconium silicate nanoparticles were taken for fabrication of 3 specimens. As per group 1, same proportion and procedure was followed for fabrication of specimens reinforced with 4% silanized zirconium silicate nanoparticles.

The finished specimens were stored in distilled water for 1 week at room temperature.<sup>57</sup>

**j) Testing of specimens (PLATE IV)**

Testing of specimens was carried out at metallurgical laboratory. The specimens for each group were tested for flexural strength. The flexural three-point bending test is useful in comparing the flexural strength of denture base materials as it simulates the type of stress that is applied to the denture during mastication.

Flexural strength was tested with universal testing machine system, at a 5.0mm/minute crosshead speed.<sup>7</sup> The specimens were supported on the jig separated at a distance of 50 mm. Load was applied at the centre of the specimen. Stress- strain curves were recorded on a chart throughout the flexural tests. The maximum load during fracture was determined from the chart and recorded as fracture load in N (Newton) and the flexural strength was calculated in MPa.

Flexural strength (FS) was calculated using the formula.

$$FS = \frac{3Pl}{2bd^2}$$

Where, FS = flexural strength (N/mm<sup>2</sup>),

P = load at fracture (N),

I = distance between the supporting wedges (mm),

b = width of the specimen (mm) &

d = thickness of the specimen (mm).<sup>61</sup>

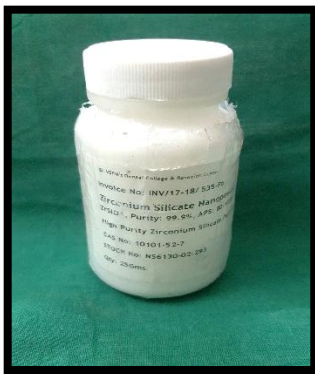
**PLATE I**  
**MATERIALS**



**Fig 1: Heat polymerized acrylic resin**



**Fig 2: Die Stone**



**Fig 3: Zirconium silicate nanoparticles**



**Fig 4: Silane coupling agent**



**Fig 5: Toluene**



**Fig 6: Cold mould seal**

**PLATE II**  
**ARMAMENTARIUM AND EQUIPMENTS**



**Fig 7: High accuracy balance**



**Fig 8: Ultrasonicator**



**Fig 9: Magnetic stirrer**



**Fig 10: Vacuum rotary evaporator**



**Fig 11: Acrylizer with thermostat**



**Fig 12: Universal testing machine**

**PLATE III**  
**ARMAMENTARIUM AND EQUIPMENTS**



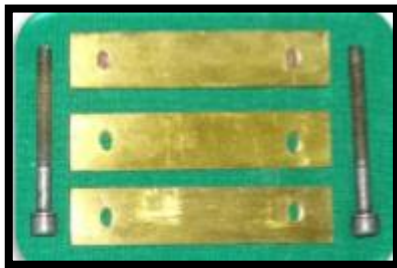
**Fig 13: Rubber bowl, plaster spatula, lacron's carver and varsity flask & clamp**



**Fig 14: Glass Beaker. Sterile Syringe, Petroleum jelly, camel hair brush, Sand paper, Porcelain jar, Dapen dish,**



**Fig 15: Vernier Caliper**



**Fig 16: Brass metal dies**



**Fig 17: Parafilm**



**Fig 18: Hydraulic bench press**



**Fig 19: Distilled water**

**PLATE IV**  
**METHODOLOGY**



**Fig 20: Preparation of gypsum mould to obtain specimens**

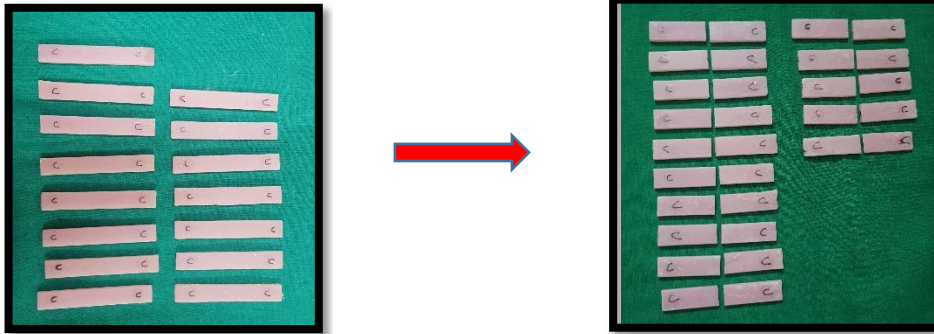


**Fig 21: Silanization process of zirconium silicate nanoparticles**

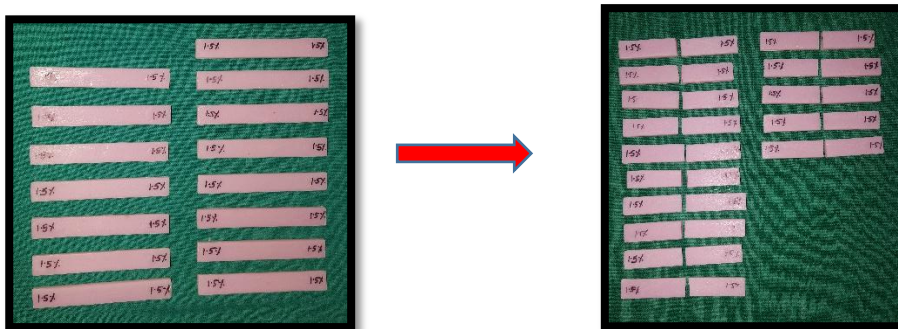


**Fig 22: Testing of specimens**

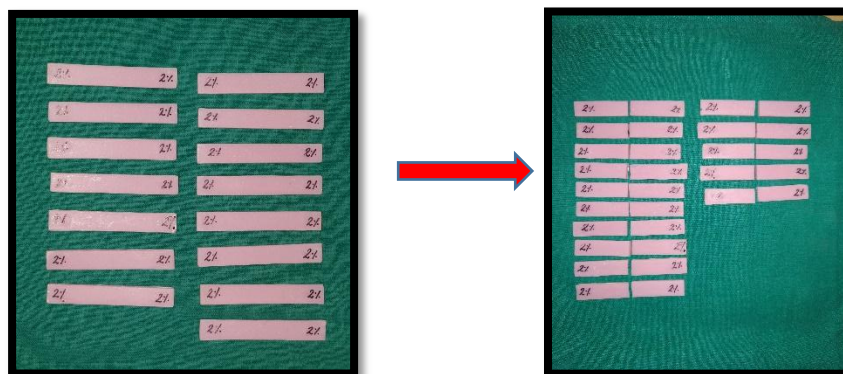
## PLATE V



**Fig 23: Group 1- The control group; Heat polymerized polymethyl methacrylate denture base specimens without reinforcement before and after testing of flexural strength.**

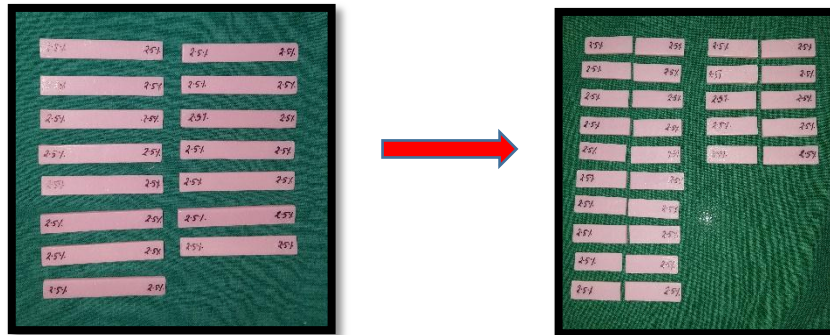


**Fig 24: Group 2- Heat polymerized polymethyl methacrylate denture base specimens reinforced with 1.5% silanized zirconium silicate nanoparticles before and after testing of flexural strength.**

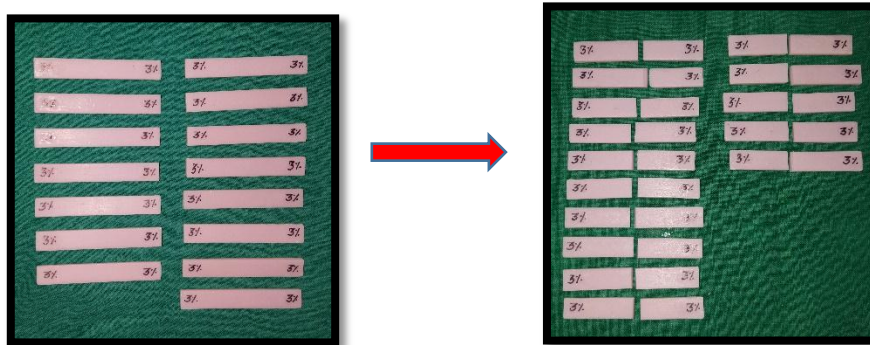


**Fig 25: Group 3- Heat polymerized polymethyl methacrylate denture base specimens reinforced with 2% silanized zirconium silicate nanoparticles before and after testing of flexural strength.**

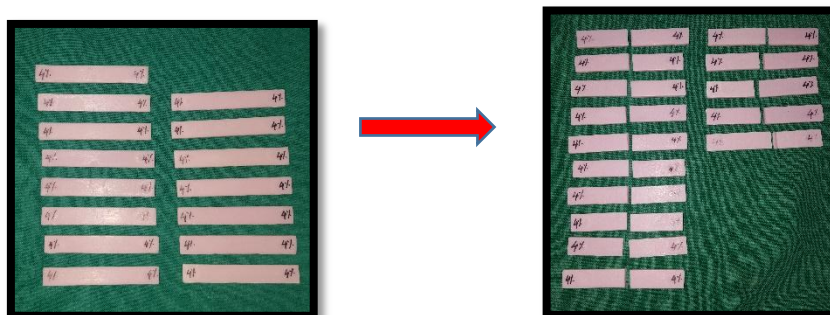
## PLATE VI



**Fig 26: Group 4- Heat polymerized polymethyl methacrylate denture base specimens reinforced with 2.5% silanized zirconium silicate nanoparticles before and after testing of flexural strength.**



**Fig 27: Group 5- Heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium silicate nanoparticles before and after testing of flexural strength.**



**Fig 28: Group 6- Heat polymerized polymethyl methacrylate denture base specimens reinforced with 4% silanized zirconium silicate nanoparticles before and after testing of flexural strength.**

## *Results*

*The science of today is the technology of tomorrow - Edward Teller*

In this study the flexural strength of heat polymerized polymethyl methacrylate denture base material without reinforcement and reinforced with different percentages of silanized zirconium silicate nanoparticles was evaluated and compared.

A total 90 specimens were prepared and were divided into six groups. Each group included 15 specimens.

### Distribution of samples into groups

Sr no.	Group	Code	n = no. of samples
1	The control group; heat polymerized polymethyl methacrylate denture base specimens without reinforcement	Group 1	15
2	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 1.5% silanized zirconium silicate nanoparticles	Group 2	15
3	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 2% silanized zirconium silicate nanoparticles	Group 3	15
4	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 2.5% silanized zirconium silicate nanoparticles	Group 4	15
5	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium silicate nanoparticles	Group 5	15

6	Heat polymerized polymethyl methacrylate denture base specimens reinforced with 4% silanized zirconium silicate nanoparticles	Group 6	15
	Total number of samples		90

15 specimens of each group were tested for flexural strength. Flexural strength was tested with universal testing machine at a 5.0 mm/minute crosshead speed. The maximum load was determined from the chart and recorded as a fracture load in N (Newton) and the flexural strength was calculated in MPa and the results were then statistically analyzed.

## STATISTICAL ANALYSIS

The statistical calculations were performed using the software SPSS for Windows (Statistical Presentation System Software, SPSS Inc. 1999, New York) version 19.0. The following statistical methods were employed in the present study.

- Descriptive statistics including mean , standard deviation.
- **Mean** is sum of all observations divided by the no. of observations.
- **Median** is value of the variable that divides the distribution into two equal parts i.e. 50 % observations will lie below and above it.
- **Standard Deviation** is summarized as the amount of variation (change) in the observation from their average value (mean).

- The formula used for calculating standard deviation:

$$SD = \sqrt{\frac{\sum(\bar{X} - X)^2}{n-1}}$$

Where:

$\bar{X}$  = Mean

X = Values of the variables

$\Sigma$  = Sum of the value

n = Number of observations

Min = Minimum Value

Max = Maximum Value

- **Null Hypothesis:** There is no significant difference in the flexural strength between the groups i.e. Group 1 = Group 2 = Group 3 = Group 4 = Group 5 = Group 6
- **Alternate Hypothesis:** There is a significant difference in the score recorded between the groups i.e. Group 1  $\neq$  Group 2  $\neq$  Group 3  $\neq$  Group 4  $\neq$  Group 5  $\neq$  Group 6
- **Level of Significance:**  $\alpha=0.05$

### One-way ANOVA

- The One-Way ANOVA test produces a one-way analysis of variance for a quantitative dependent variable by a single factor (independent) variable. Analysis of variance is used to test the hypothesis when several means are equal. This technique is an extension of the two-sample t test.
- In addition to determining that differences exist among the means, we may require to know which means differs and post hoc tests was used. In the present study one- way ANOVA was applied to find out the mean difference.

#### **Tukey's multiple post hoc Test**

- Once it is determined that differences exist among the means, post hoc range tests and a pair-wise multiple comparisons aid in determining which means differ. Range tests identify homogeneous subsets of means that are not different from each other. Pairwise multiple comparisons test the difference between each pair of means, and yield a matrix where asterisks indicate significantly different groups means at an alpha level of 0.05.
- Tukey's HSD (Honestly significant Difference) is one of the widely used post hoc tests. In the present study Tukey's HSD post hoc Test was applied to make pairwise multiple comparisons to find out the difference between each pair of mean values of the six groups included in the study.

**Table 1** provides the descriptive statistics for flexural strength of test specimens in six study groups. The mean of **Group 1** was **95.7** MPa and the strength ranged between 82.16 to 105.43 MPa. For **Group 2**, the mean flexural strength was **106.53** MPa and ranged between 88.87 to 118.54 MPa. In **Group 3**, the mean flexural strength

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was **104.13** MPa and ranged between 95.58 to 118.70 MPa. For **Group 4**, the mean flexural strength was **101.95** MPa and ranged between 77.58 to 117.58 MPa. In **Group 5**, the mean flexural strength was **99.16** MPa and ranged between 85.45 to 113.50 MPa. For **Group 6**, the mean flexural strength was **97.49** MPa and ranged between 86.54 to 111.08 MPa. A graphical visualization of mean strength along with error bar is given in Graph 1.

**Table 2** reveals that using ANOVA F test, the mean flexural strength across groups differed significantly across six groups, as indicated by *p*-value =**0.002** (*p* <**0.05**). In order to determine, which groups contributed to overall significance, a pair-wise comparison of mean strength was performed using *Tukey's HSD test*.

**Table 3** shows individual pair-wise comparison of flexural strength between groups using Tukey's post-hoc test. It shows significant statistical difference (*p* <**0.05**) exist on comparison of flexural strength between **Group 1** (control) and **Group 2** (1.5% silanized zirconium silicate nanoparticle) (*p* = **0.004**). Also, statistical significant difference (*p* <**0.05**) was found between **Group 2** (1.5% silanized zirconium silicate nanoparticle) and **Group 6** (4% silanized zirconium silicate nanoparticle) (*p* =**0.029**). While, no statistical significant difference (*p* >0.05) was observed between rest of all groups.

## *Discussion*

*Science is beautiful when it makes simple explanations of phenomenon or connection between different observations- Stephen Hawking*

The use of dental prosthesis is essential in order to restore function and esthetics in edentulous patients. There has been continuous development in the field of material science from using naturally occurring materials to use of synthetic resins for fabricating dentures.<sup>4</sup> **Dr. Walter Wright** in 1937, introduced polymethyl methacrylate as a denture base material. Since its introduction, polymethyl methacrylate is still the most predominantly used denture base material because of its excellent esthetics, being economical, ease of processing and repair.<sup>7,8</sup> It is a combination of the benefits mentioned above rather than one excellent aspect that accounts for its popularity and wide usage. However, this material is not ideal in every aspect, especially when meeting

the mechanical requirements of a prosthesis. It has certain drawbacks like poor mechanical strength, residual monomer allergy, low fatigue strength, poor conduction of heat, low hardness, thermal shrinkage, porosity, high coefficient of thermal expansion, crazing and warpage.<sup>9</sup>

The denture base resin is subjected to various stresses during function, which include compressive, tensile, shear, and impact stresses.<sup>11</sup> A study by **Johnston et al**<sup>12</sup> observed that 68% of acrylic resin dentures fractured within the first three years after fabrication.

**Smith (1961)**<sup>12</sup> analyzed the practical situation with respect to fracture of dentures and showed that there are two types of failure.

1. Outside the mouth which is caused by impact forces, i.e. a high stress rate and
2. Inside the mouth, usually in function; probably due to a fatigue phenomenon, i.e. a low and repetitive stress.

Fracture of acrylic resin denture base happens because of the fatigue and chemical degradation of denture base material.<sup>9</sup> Any factor that exacerbates deformation of the base or alters its stress distribution will predispose the denture to fracture.<sup>11</sup> During chewing, denture base material undergoes flexural deformation. Flexural strength of denture base resin is considered as the primary mode of clinical failure.<sup>10</sup> For this reason, flexural strength was selected as the most relevant mechanical property to evaluate the strength of denture base resins.

Studies have shown that the average values of flexural strength of heat polymerizing acrylic resins are near to 78-92 MPa.<sup>11</sup> There are three ways to improve the properties of PMMA: **development of an alternative material** to PMMA; the **chemical modification of PMMA** such as by the addition of a rubber graft copolymer and the **reinforcement of PMMA with other materials** such as carbon fibres, glass fibres and ultra-high modulus polyethylene.<sup>9</sup> Various substitutes for polymethyl methacrylate have been introduced such as polystyrene, poly vinyl acrylic, polyamides (nylons) and light activated urethane dimethacrylate resins. Although these materials exhibited desirable properties, none have been proven superior to polymethyl methacrylate (PMMA).<sup>7</sup>

Different attempts have been made to enhance the properties of PMMA including the addition of metal wire to the resin. Metals in various forms such as wires, plates and fillers have been incorporated into PMMA to improve thermal conductivity and radiopacity, as well as mechanical properties. Various studies have added metal fillers to improve the thermal conductivity of the acrylic resins.<sup>13</sup> The drawbacks of metal fillers are that they do not chemically bond to the resins and produce negative effects on esthetics and stress concentration.<sup>9</sup>

The fibre-reinforced plastics are commonly used in many fields in the industry because of their better mechanical properties, which can be tailored to specific needs. Reinforcement of denture base resins with short or long fibres have been described in the literature for nearly half a century. The polymer matrix forms a continuous phase that surrounds the fibers, thus the applied loads are transferred from the polymer matrix to the fibers.<sup>7</sup> In some studies, reinforcement of denture PMMA

resin has failed because of the stress concentrations around the embedded fibers, this phenomenon may be the result of poor distribution of the reinforced fibers and poor adhesion between resin matrix and fibers.<sup>61</sup>

**Larson et al.**<sup>20</sup> have concluded that the use of carbon fibers improves the strength of denture bases. Use of carbon fibers has improved the mechanical properties of the matrix because of its inherent high strength and optimal combination of the carbon fibers and matrix. Mainly, carbon fibers have been used to enhance fatigue and impact strength. Despite good mechanical properties, cytotoxicity of carbon fibers is problematic.

In 1959, **Feynman** introduced the concept of nanotechnology. Since then, nanotechnology has been widely used in many applications, including medical sciences where it plays an important role in diagnosis, treatment and regenerative medicine. A nanomaterial is an object, in which at least one of its dimensions is at the nanometer scale (approximately 1 to 100 nm). Nanomaterials are categorized according to dimension – those with all 3 dimensions less than 100 nm [nanoparticles (Nps) and quantum dots]; those that have 2 dimensions less than 100 nm (nanotubes, nanofibers, and nanowires); and those that have one dimension less than 100 nm (thin films, layers, and coatings).<sup>66</sup> Recently, researchers have used nanofillers for reinforcement of denture base resins. Size, shape, surface area, concentration and dispersion of nanofillers into resin matrix all have an effect the mechanical properties of the filler/resin composite. Nanoparticles of Alumina, titanium (TiO<sub>2</sub>), silver, zirconia (ZrO<sub>2</sub>), gold, platinum, silicon dioxide (SiO<sub>2</sub>) are among the fillers that have been incorporated to enhance the mechanical properties of denture base resins.<sup>58</sup>

**Korkmaz et al.**<sup>67</sup> suggested that the small size of filler particles ensures proper processing. The average particle size of PMMA beads used in denture base resins is around 100 $\mu$ m. The particle size of Ag nanoparticles (80-100 nm) used is much smaller than that of powder resin particles. Silver nanoparticles fills the interstitial spaces of polymer particles to give a heterogenous mixture and does not force the displacement of the segments of polymer chain. Also, low percentage of nanoparticles should be used to ensure that they will be embedded in resin. Due to high surface area of the nanoparticles, the applied stress is transformed from the matrix onto the silver nanoparticles, resulting in an enhancement of the mechanical properties.

Denture base resins reinforced with TiO<sub>2</sub> nanoparticles have also been of interest to researchers because of its unique properties. The properties like biocompatibility, excellent mechanical properties, low cost, high stability and appropriate antimicrobial effect makes TiO<sub>2</sub> a favorable additive for biomaterials.<sup>68</sup> TiO<sub>2</sub> nanoparticles have been used as an additive to improve both mechanical and antibacterial properties of different dental materials.<sup>69</sup>

Recent development of composite materials of great strength and low mass has made significant contribution in the field of dental material science. The incorporation of the ceramic nano-filler into the more flexible and lower thermal resistance polymer improves its stiffness and thermal stability.<sup>70</sup> Ceramic fillers were used for reinforcements as opposed to metal fillers because of its lower filler density.<sup>7</sup>

**Asopa et al.**<sup>56</sup> concluded that zirconium oxide when used as a filler in the high impact acrylic resin resulted in increase in transverse strength as compared to the

control group. Zirconium oxide, commonly referred to as zirconia ( $ZrO_2$ ), possesses strong ionic inter atomic bonding, giving rise to its desirable material characteristics. Addition of zirconia nano-fillers to acrylic resin was found to improve mechanical properties. In addition to that  $ZrO_2$  is known to have excellent biocompatibility and white color which was less likely to alter esthetics.

In nature Zirconia does not occur in a pure state. It has been found in conjunction with silicate oxide with the mineral name Zircon ( $ZrO_2 \times SiO_2$ )/ Zirconium silicate or as a free oxide ( $ZrO_2$ ) with the mineral name Baddeleyite.<sup>71</sup>

**Kareem S**<sup>57</sup> concluded that highly significant increase in the impact strength, transverse strength and surface hardness occurred with the addition of 1.5% wt zirconium silicate nano-filler. Non-significant increase in the impact strength, significant increase in transverse strength and highly significant increase in surface hardness occurred with the addition of 1% wt  $ZrSiO_4$  nano-filler. Non-significant increase in surface roughness was seen with both 1% and 1.5% wt  $ZrSiO_4$  nano-filler. Highly significant decrease in water sorption and solubility was seen with 1.5% wt  $ZrSiO_4$  nano-filler and non-significant decrease in water sorption and solubility occurred with 1% wt  $ZrSiO_4$  nano-filler when compared with the control group.

The highly significant increase in the impact strength when polymethyl methacrylate was reinforced with zirconium silicate nanoparticles was due to this new compound ( $ZrSiO_4$ +PMMA) which not only potentiates the internal resistance but also significantly affects the compound's stress-strain behavior due to the particle size and

bonding interaction. Also, forces applied are transferred to the nanoparticles which improves the impact strength.

Addition of nanoparticles fills the free spaces between the chains and attract resin molecules, hence polymer chains during the curing process create more complicated network chains thereby increasing the transverse strength.

The increase in hardness of PMMA reinforced with zirconium silicate nanoparticles can be explained with several factors; it may be attributed to the intrinsic hardness characteristic of the  $ZrSiO_4$  nanoparticles, zirconium silicate has tetragonal crystal structure which looks like small prism shaped structure separated or may give the impression of double pyramids connected from the bottom resulting in very hard and heavy properties of the polymer nano-composite. Another factor for increase in hardness may be the good distribution of nanoparticles in the resin matrix.

Zirconium silicate nano filler has the property of being insoluble in water. When it was incorporated into PMMA resin matrix it led to decrease in diffusivity of the water molecule which resulted in decrease in water sorption and solubility.<sup>57</sup>

**Abdulkareem M, Hatim N**<sup>72</sup> concluded that microwave radiation of PMMA powder and the addition of  $Al_2O_3$  and Ag nanoparticles is effective in increasing the flexural strength of denture base resins. Increase in the flexural strength may be explained based on the property of transformation toughening. When sufficient stress develops and micro cracks begin to propagate, a transformation phenomenon of Nps occurs, which depletes the energy of crack propagation. Hence, proper distribution of

the nano-filler within the matrix can stop or deflect the cracks. Another possible reason for increase in flexural strength of the denture base with the addition of nanoparticles was due to transfer of stress from more flexible polymer to the higher modulus, more rigid and stiffer nanoparticles.

**Chandler H et al (1971)**<sup>42</sup> suggested that one of the disadvantage of the available denture base resin materials is their radiolucency. Thus there are chances of ingestion or aspiration of either broken parts or portions of ill-fitting complete dentures by the patient. **Sehgal and Sood(1989)**<sup>13</sup> stated that reinforced PMMA with metal oxide fillers like silver, copper, aluminium not only increases the strength but also provides radiopacity to the heat polymerized denture base material.

A study by **Ihab et al**<sup>50</sup> concluded that increase in the transverse strength occurred with addition of 2-5wt% ZrO<sub>2</sub> nanoparticles due to good distribution of the very fine size of nanoparticles. But increasing the percentage of modified nano-ZrO<sub>2</sub> to 7wt% lowered the impact strength and transverse strength due to agglomeration nano-ZrO<sub>2</sub>. Hence 1.5%, 2%, 2.5%, 3%, 4% percentages were selected for this study.

The inorganic filler particles usually display high surface energy because of hydrophilic ionic nature. But due to difference in surface energy, the hydrophobic polymer does not wet or interact with the filler particles.<sup>53</sup> Therefore it is important to modify the filler surface for better dispersion and improve surface wetting, thereby improving the physical properties of the composites.<sup>73</sup> Hence in this study, Zirconium silicate nanoparticles were treated with trimethoxysilylpropylmethacrylate (TMSPM) to improve adhesion of nanoparticles to the resin matrix.<sup>57</sup>

According to our study the average values of flexural strength of heat polymerized acrylic resins is 95.7 MPa. The ultimate goal of this study was to evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate denture base material without reinforcement and reinforced with 1.5%, 2%, 2.5%, 3%, 4% of silanized zirconium silicate nanoparticles. Thus the mean flexural strength obtained were Group 1 (95.7 MPa), Group 2 (1.5% Zirconium silicate) (106.53 MPa), Group 3 (2% Zirconium silicate) (104.13MPa), Group 4 (2.5% Zirconium silicate) (101.95 MPa), Group 5 (3% Zirconium silicate) (99.16 MPa) and Group 6 (4% Zirconium silicate) (97.49 MPa). This study shows that maximum increase in the flexural strength was obtained when PMMA was reinforced with 1.5% of zirconium silicate nanoparticles.

These results obtained are similar to the study done by **Kareem S**<sup>57</sup> who concluded that highly significant increase in the impact strength, transverse strength and surface hardness occurred with the incorporation of 1.5%wt zirconium silicate nano-filler.

**Vojdani M**<sup>61</sup> concluded that addition of 2.5 wt% Al<sub>2</sub>O<sub>3</sub> powder significantly increased the flexural strength and hardness of heat-polymerized acrylic resin. In their pilot study addition of 5–20 wt% Al<sub>2</sub>O<sub>3</sub> significantly reduced flexural strength when compared to the control group. Possible explanations for reduction in strength with increasing in percentage could be: a decrease in the cross-section of the load-bearing matrix; stress concentration as a result of too many filler particles; increased amount of fillers may also alter the modulus of elasticity of the resin and mode of crack

propagation through the specimen; entrapped air and moisture may lead to void formation and incomplete wetting of the fillers by the resin.

## **CLINICAL IMPLICATION**

When the entire spectrum of this study is analysed, it becomes evident that the heat polymerized acrylic dentures reinforced with silanized zirconium silicate nanoparticles increase the flexural strength of the denture base material and thus, reduces the probability of occurrence of fracture. According to the literature zirconium silicate nanoparticles are also insoluble in water therefore decreases water sorption and solubility. It also improves the impact strength thereby reducing fracture of dentures due to accidental dropping.<sup>57</sup>

## **SCOPE FOR FURTHER STUDIES**

1. Fatigue testing of these materials under dynamic loading using the denture base configurations in simulated oral conditions, using saliva or its substitutes is an area for further research.
2. Further research is needed to evaluate the effect of aging on the new reinforced denture base material before clinical application.
3. Other physical and mechanical properties like thermal diffusivity, abrasion resistance, color stability and disinfectant property can be studied.

4. The heat polymerized acrylic dentures can be reinforced with even further different sized nanoparticles and various physical and mechanical properties can be evaluated.
5. Further research is also needed to quantify the filler distribution in the polymer matrix.

## **LIMITATIONS OF THE STUDY**

The study was designed and carried out with utmost accuracy, however certain limitations encountered in the study can be enlisted as follows:

1. In the oral cavity, reinforced denture base is exposed to forces of varying magnitudes acting in different directions. The same situation could not be simulated in this in vitro study.
2. Scanning electron microscopy (SEM) examination of the samples to evaluate the adhesion of zirconium silicate nanoparticles to the surface of PMMA was not performed.

## *Summary*

The heat cure denture base resins are extensively used because of their excellent properties such as ease of handling, polishing and esthetics.<sup>7,8</sup> However, the mechanical strength is not sufficient to maintain the longevity of the denture.<sup>9</sup> The fracture of acrylic resin denture is a common occurrence.<sup>11</sup>

This study was conducted to evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate denture base material without reinforcement and reinforced with different percentages of silanized zirconium silicate nanoparticles. Standard heat cured acrylic resin specimens were fabricated according to ADA specification no. 12 and total 90 specimens were fabricated with 15 specimens in each group. Group 1- The control group; heat polymerized polymethyl methacrylate denture base specimens without reinforcement. Group 2, 3, 4, 5, 6-Heat polymerized

polymethyl methacrylate denture base specimens reinforced with 1.5%, 2%, 2.5%, 3%, 4% silanized zirconium silicate nanoparticles respectively.

Flexural strength was tested using universal testing machine at a crosshead speed of 5mm/min. The findings were statistically analyzed and the flexural strength was calculated in MPa.

Results showed that the mean flexural strength for Group 1 (95.7 MPa), Group 2 (1.5% silanized zirconium silicate Nps) (106.53 MPa), Group 3 (2% silanized zirconium silicate Nps) (104.13MPa), Group 4 (2.5% silanized zirconium silicate Nps) (101.95 MPa), Group 5 (3% silanized zirconium silicate Nps) (99.16 MPa) and Group 6 (4% silanized zirconium silicate Nps) (97.49 MPa). The statistical analysis showed that significant statistical difference (**p <0.05**) exist on comparison of flexural strength between **Group 1** (control) and **Group 2** (1.5% silanized zirconium silicate Nps) (**p = 0.004**). Also, statistical significant difference (**p <0.05**) was found between **Group 2** (1.5% silanized zirconium silicate Nps) and **Group 6** (4% silanized zirconium silicate Nps) (**p =0.029**). While, no statistical significant difference ( $p >0.05$ ) was observed between rest of all groups.

Thus reinforcement with 1.5% of silanized zirconium silicate nanoparticles (Group 2) showed statistically significant increase in the flexural strength as compared to unreinforced specimens (Group 1). Specimens with reinforcement increased the flexural strength, but it was not statistically significant when compared to unreinforced specimens.

## *Conclusion*

*Science is organized knowledge. Wisdom is organized life-Immanuel Kant*

Within the limitations of this study following conclusions were drawn:

- Specimens with reinforcement increased the flexural strength.
- There was no statistically significant increase in the flexural strength of heat polymerized polymethyl methacrylate denture base specimens reinforced with 2% (Group 3), 2.5% (Group 4), 3% (Group 5), 4% (Group 6) silanized zirconium silicate nanoparticles when compared to unreinforced specimens.
- Reinforcement with 1.5% of silanized zirconium silicate nanoparticles showed statistically significant increase in flexural strength.

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*Tables and Graphs*

**TABLES**

**Table 1: Descriptive statistics for flexural strength**

<b>GROUPS</b>	<b>MEAN</b>	<b>Standard Deviation (S.D)</b>	<b>Minimum</b>	<b>Maximum</b>
<b>Group 1 (Control)</b>	95.7	7.88	82.16	105.43
<b>Group 2 (1.5% Zirconium silicate Nps)</b>	106.53	8.10	88.87	118.54
<b>Group 3 (2% Zirconium silicate Nps)</b>	104.13	5.80	95.58	118.70
<b>Group 4 (2.5% Zirconium silicate Nps)</b>	101.95	9.83	77.58	117.58
<b>Group 5 (3% Zirconium silicate Nps)</b>	99.16	8.4	85.45	113.50
<b>Group 6 (4% Zirconium silicate Nps)</b>	97.49	6.95	86.54	111.08

**Table 2: One-way analysis of variance for Flexural Strength across six groups**

<b>GROUPS</b>	<b>MEAN</b>	<b>Standard Deviation (S.D)</b>	<b>ANOVA F TEST</b>	<b>P value, Significance</b>
<b>Group 1 (Control)</b>	95.7	7.88	<b>F = 4.046</b>	<b>p = 0.002, significant difference</b>
<b>Group 2 (1.5% Zirconium silicate Nps)</b>	106.53	8.10		
<b>Group 3 (2% Zirconium silicate Nps)</b>	104.13	5.80		
<b>Group 4 (2.5% Zirconium silicate Nps)</b>	101.95	9.83		
<b>Group 5 (3% Zirconium silicate Nps)</b>	99.16	8.4		
<b>Group 6 (4% Zirconium silicate Nps)</b>	97.49	6.95		

p >0.05 – not significant

p <0.05 – significant

p <0.001 – highly significant

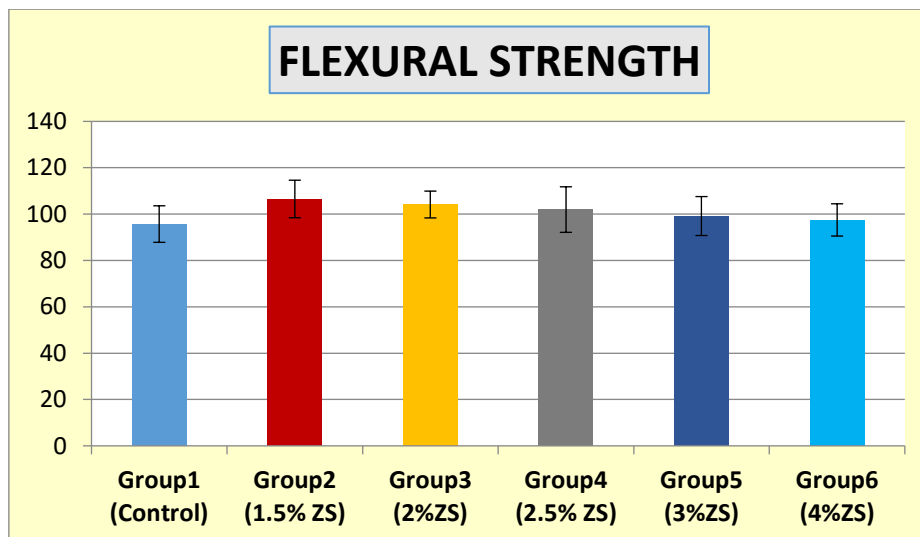
**Table 3: Individual pair –wise comparison of flexural strength between groups using Tukey’s post – hoc test**

Tukey’s post hoc test to find individual pair wise comparison			
GROUP	COMPARISON GROUP	MEAN DIFFERENCE	p value, Significance
<b>Group 1 (Control)</b>	<b>Group 2</b>	10.83	<b>p = 0.004*</b>
	<b>Group 3</b>	8.42	p = 0.051
	<b>Group 4</b>	6.24	p = 0.269
	<b>Group 5</b>	3.45	p = 0.838
	<b>Group 6</b>	1.79	p = 0.989
<b>Group 2 (1.5% ZrSiO<sub>4</sub> Nps)</b>	<b>Group 3</b>	2.40	p = 0.961
	<b>Group 4</b>	4.58	p = 0.612
	<b>Group 5</b>	7.37	p = 0.123
	<b>Group 6</b>	9.03	<b>p = 0.029*</b>
<b>Group 3 (2% ZrSiO<sub>4</sub> Nps)</b>	<b>Group 4</b>	2.18	p = 0.974
	<b>Group 5</b>	4.97	p = 0.525
	<b>Group 6</b>	6.63	p = 0.210
<b>Group 4 (2.5% ZrSiO<sub>4</sub> Nps)</b>	<b>Group 5</b>	2.78	p = 0.929
	<b>Group 6</b>	4.44	p = 0.642
<b>Group 5 (3% ZrSiO<sub>4</sub> Nps)</b>	<b>Group 6</b>	1.66	p = 0.992

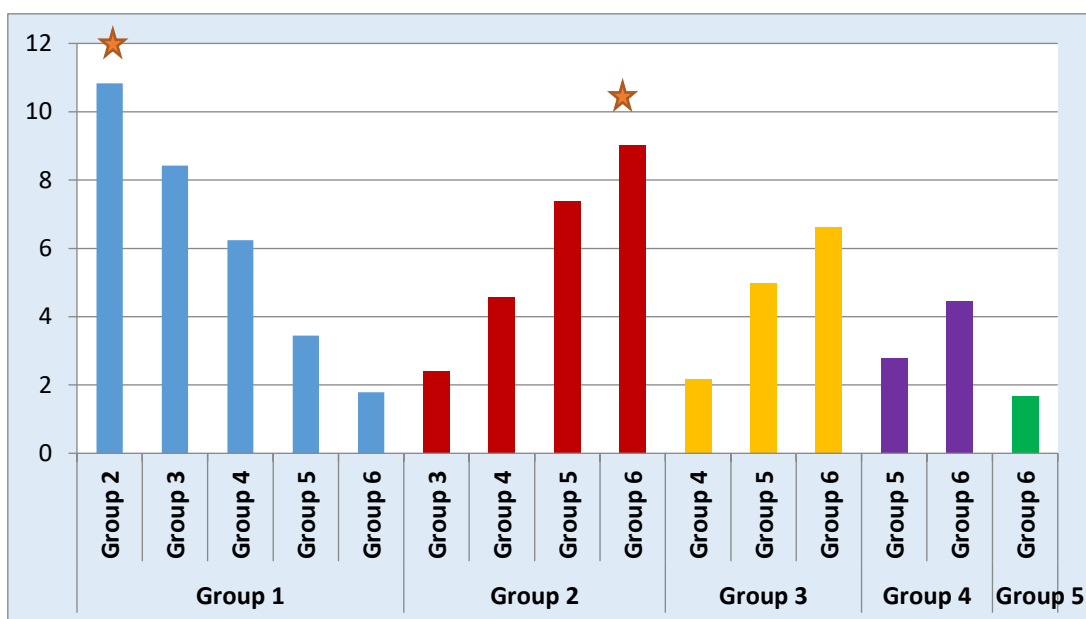
**p >0.05 – not significant \* p <0.05 – significant \*\*p <0.001 – highly significant**

## GRAPHS

**Graph 1: Mean and error bar for flexural strength according to study groups**



**Graph 2: Mean difference of Pair wise comparison of groups for flexural strength**



**ANNEXURE**

<b>Group 1 : Control</b>			<b>Group 2 : 1.5% Zirconium Silicate Nanoparticles</b>	
<b>Sr. No</b>	<b>Flexural Load (N)</b>	<b>Flexural Strength (MPa)</b>	<b>Flexural Load (N)</b>	<b>Flexural Strength (MPa)</b>
<b>1</b>	98.592	82.16	135.696	113.08
<b>2</b>	115.932	96.61	130.8	109
<b>3</b>	113.244	94.37	142.248	118.54
<b>4</b>	124.068	103.39	127.344	106.12
<b>5</b>	117.408	97.84	106.644	88.87
<b>6</b>	123.036	102.53	131.592	109.66
<b>7</b>	99.264	82.72	122.544	102.12
<b>8</b>	121.02	100.85	116.04	96.7
<b>9</b>	124.704	103.92	125.34	104.45
<b>10</b>	121.716	101.43	141.792	118.16
<b>11</b>	119.46	99.55	131.844	109.87
<b>12</b>	126.516	105.43	119.796	99.83
<b>13</b>	107.22	89.35	137.1	114.25
<b>14</b>	106.08	88.4	127.944	106.62
<b>15</b>	104.364	86.97	120.84	100.7
<b>Mean</b>		<b>95.7</b>		<b>106.53</b>

Group 3 : 2% Zirconium Silicate Nanoparticles			Group 4 : 2.5% Zirconium Silicate Nanoparticles	
Sr. No	Flexural Load (N)	Flexural Strength (MPa)	Flexural Load (N)	Flexural Strength (MPa)
1	118.992	99.16	118.14	98.45
2	129.396	107.83	127.5	106.25
3	129.792	108.16	118.14	98.45
4	122.544	102.12	121.248	101.04
5	128.34	106.95	119.796	99.83
6	118.5	98.75	93.096	77.58
7	117.9	98.25	125.796	104.83
8	114.696	95.58	112.74	93.95
9	127.14	105.95	110.1	91.75
10	122.04	101.7	124.596	103.83
11	121.692	101.41	138.9	115.75
12	129.9	108.25	122.544	102.12
13	121.44	101.2	129.648	108.04
14	142.44	118.7	141.096	117.58
15	129.54	107.95	131.7	109.75
<b>Mean</b>		<b>104.13</b>		<b>101.95</b>

<b>Group 5 : 3% Zirconium Silicate Nanoparticles</b>			<b>Group 6 : 4 % Zirconium Silicate Nanoparticles</b>	
<b>Sr. No</b>	<b>Flexural Load (N)</b>	<b>Flexural Strength (MPa)</b>	<b>Flexural Load (N)</b>	<b>Flexural Strength (MPa)</b>
<b>1</b>	118.5	98.75	118.644	98.87
<b>2</b>	119.4	99.5	126.096	105.08
<b>3</b>	136.2	113.5	116.196	96.83
<b>4</b>	108.444	90.37	133.296	111.08
<b>5</b>	117.792	98.16	130.44	108.7
<b>6</b>	108	90	121.44	101.2
<b>7</b>	127.392	106.16	114.24	95.2
<b>8</b>	122.196	101.83	111.648	93.04
<b>9</b>	129.444	107.87	115.44	96.2
<b>10</b>	106.596	88.83	117	97.5
<b>11</b>	112.848	94.04	103.848	86.54
<b>12</b>	127.548	106.29	106.692	88.91
<b>13</b>	102.54	85.45	117.84	98.2
<b>14</b>	131.7	109.75	115.14	95.95
<b>15</b>	116.292	96.91	106.992	89.16
<b>Mean</b>		<b>99.16</b>		<b>97.49</b>