‘COMPARATIVE EVALUATION OF FLEXURAL STRENGTH OF HEAT POLYMERIZED POLYMETHYL METHACRYLATE DENTURE BASE MATERIAL REINFORCED WITH SILANIZED ALUMINIUM OXIDE NANOPARTICLES AND SILANIZED TITANIUM DIOXIDE NANOPARTICLES –AN *IN VITRO* STUDY.’

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List of Abbreviations Used

Graph No.	Abbreviation	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.	^{o}C	Degree Celsius
8.	^{o}	Degree
9.	mm	Millimetre
10.	i.e.	that is
11.	PMMA	Polymethyl methacrylate
12.	µ	Micron
13.	Al_2O_3	Aluminum oxide
14.	TiO_2	Titanium oxide
15.	ZrO_2	Zirconium oxide
17.	Rpm	Rotations per minute
18.	mins	Minutes
19.	gms	Grams

Introduction

The key to growth is the introduction of higher dimensions of consciousness into our awareness

Dentistry as a speciality is believed to have begun about 3000 BC and the first dental prosthesis was believed to have been constructed in Egypt about 2500 BC.[1] In order to restore a degree of function and appearance, caused due to loss of teeth by accident or disease, it has been necessary always to adapt contemporary materials to dental applications as they are available in one period of history.[2]

During 18th century, wood, bone, ivory, gold and porcelain were the materials used for the fabrication of denture base. French dentist **Etienne Bourdet (1775)**, made the first reference to the use of a gold base punctuated with small holes resembling like the sockets of teeth. [2]

During 19th century, tortoise shell, gutta percha, vulcanite, aluminium and celluloid were used for fabricating denture base. For 75 years, Vulcanite remained the principal material for fabrication of denture base.[2]

During the 20th century, Bakelite (1909), Stainless steel (1921), Cobalt Chromium (1930), Vinyl resin (1932), acrylic resin (1937), Self-cure acrylic resin, Epoxy resin (1951), Polystyrene (1951), Nylon (1955), Polycarbonates (1967), High impact acrylic (1967), Polysulphones (1981), Visible L.C (1947), Acrylic (1986), and Pure Titanium (1998) were used for denture fabrication.[2]

Dr. Walter Wright (1937) introduced Polymethyl methacrylate as a denture base material which became the major polymer to be used [1] and most commonly for the fabrication of complete dentures. The popularity of PMMA as denture base material was attributed to its ease of processing, low cost, light weight, excellent aesthetic properties, low water sorption and solubility; and ability to be repaired easily. However, low thermal conductivity, inferior mechanical strength, brittleness, high coefficient of thermal expansion and relatively low modulus of elasticity makes it more prone to failure during the clinical service.[3]

Studies have shown that 68 % of the complete dentures fabricated, fractured within the first three years.[4] 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. [5]

Smith analyzed the practical situation with respect to the fracture of dentures and showed two types of failure.[6] Outside the mouth, caused by impact forces, i.e. a high stress rate and inside the mouth, usually in function; this is probably a fatigue phenomenon, i.e. a low and repetitive stress rate. [6,7]

Fracture of dentures in clinical service has been a concern and several attempts have been made to improve flexural and impact strength of PMMA by giving maximum bulk to the material in the regions most heavily stressed, by copolymerization and cross-linking, reinforcement with carbon fibres. [8]

Recent studies have demonstrated that specifically formulated metal oxide nanoparticles have good antimicrobial activity or mechanical strength and attempts has been directed towards the incorporation of inorganic nanoparticles into PMMA to improve its properties. The properties of polymer nanocomposites depend on the type of nanoparticles, their size and shape, as well as its concentration and interaction with polymer matrix.[9,10]

Clark & Plueddeman (1963), Rosen (1978) suggested that increase in the fracture resistance of an acrylic-fibre composite is dependent on the adhesion between the acrylic resin matrix and the fibres and silane compounds can be utilized to improve the adhesive properties of the fibres.[11] **Hero *et al.*, 1987; Naegeli *et al.*, 1988; Berzins, 1989; Kappert *et al.*, 1989** suggested that silanization of the surface of the metal with different techniques improved adhesion between the acrylic resin and enhance the fracture resistance of the denture base material construction. [12,13]

Aluminium oxide nanoparticles possess strong ionic inter-atomic bonding, have high hardness, good thermal properties, decrease warpage, make the material radio-opaque and inhibit growth of bacteria over the denture surface. They can exist in several crystalline phases, which all revert to the most stable hexagonal alpha phase at elevated temperature. [6, 7, 14] **Jasmin BS and Ismail JJ (2014)** concluded that addition of silanized Al_2O_3 nanoparticles increased the values of flexural strength with 1wt% significantly compared to control groups, then the flexural strength began to decrease with 3wt% in which the value of flexural strength was less than control. [10]

Titanium dioxide is a biocompatible material (non-toxic), corrosion resistance, has antimicrobial properties, increases the flexural and impact strength and aesthically acceptable. Several studies have investigated the effect of adding titanium dioxide (TiO_2) on the properties of PMMA. It was found that adding TiO_2 nanoparticles could improve the flexure strength, fracture toughness, hardness of PMMA, as well as thermal conductivity. [8,15] **Alwan SA and Alameer SS in 2015** [16] concluded that addition of silanized 3% titanium dioxide nanoparticles increases the value of flexural strength of PMMA when compared with the control group.

Therefore, an attempt is being made to evaluate and compare the flexural strength of heat polymerized acrylic resin denture base material reinforced with 3% silanized titanium dioxide nanoparticles and 1% silanized aluminium oxide nanoparticles with conventional heat polymerized polymethyl methacrylate denture base resin.

Aims and Objectives

AIM:

To evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles with that of heat polymerized polymethyl methacrylate denture base material.

OBJECTIVES:

1) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material.

2) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 1% silanized aluminium oxide nanoparticles.

3) To evaluate the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 3% silanized titanium dioxide nanoparticles.

4) To compare the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles with heat polymerized polymethyl methacrylate denture base material.

Review of Literature

Literature is one of the most interesting and significant expressions of humanity.

Polymethyl methacrylate (PMMA) acrylic resin is a preferred denture base material because of its low cost, ease of application, polish ability along with its reliance on simple processing equipment. But a major drawback of PMMA as a denture base material is its low flexural and impact strength that leads to common occurrences of the fracture of prosthesis in-situ and ex-situ. Many attempts have been made in the past to improve mechanical properties of acrylic resins including its chemical modification by the addition of a rubber graft copolymer and also by the

addition of various reinforcing materials like metals, metal fillers, carbon fibres, aramid fibres, glass fibres and ultra-high modulus polyethylene

Before 18th century, materials used for denture were wood, bone and ivory. **In 1678-1761**, **Pierre Fauchard** began with modern dentistry with the development of many prosthetic techniques by using human teeth or teeth made from hippopotamus or elephant ivory in the denture. He carved dentures from a single piece of ivory and bone where, bone displayed better dimensional stability than wood. Although, ivory was stable in the oral environment, and offered significant aesthetics, but its drawbacks were that it was not readily available, and was relatively expensive. [2] French dentist **Etienne Bourdet (1775)**, made the first reference to the use of a gold base punctuated with small holes resembling like the sockets of teeth. [2]

In **1774 Alexis Duchateau**, a Parisian apothecary, dissatisfied with his own stained hippopotamus ivory denture and inspired to use porcelain for denture fabrication. He teamed up with Parisian dentist Nicholas Dubois De Chemant.[1] Chemant's denture was popular until the introduction of individually baked porcelain teeth in **1808** by an **Italian dentist Giuseppangeio Fonzi**. In this, teeth were attached to the denture base by a small platinum hook. This pin was soldered to a gold denture base. It was one of the most important events in the history of dentistry. [2] **Loomis in 1854** fabricated the first porcelain denture with artificial teeth.

Charles H Land in 1890 made porcelain dentures with platinum bases known as continuous gum dentures. **Alexander Gutowski in 1962** from West Germany made dentures from one piece of porcelain [2]

During the latter part of the 19th century, polymers entered the field of denture base materials. **Charles Goodyear (1839)** developed the art of producing rubber and in 1851 his brother Nelson Goodyear invented a process for making hard rubber called vulcanite. Despite its displeasing appearance vulcanite dentures fitted the ridges of the patient more exactly, so that dentures could be worn with comfort.

In **1850 CF Harrington** introduce a **tortoiseshell base** that was first the thermoplastic denture base material.[2] In **1851 Edwin Truman** made a **base of Gutta percha**. However, the material was unstable and its use required complicated equipment. [2]

In **1856 Alfred A Blandy** made cheoplastic dentures by using low fusing alloy of silver, bismuth and antimony. He embedded a wax model of the denture in plaster of Paris and after melting the wax, he poured the metal compound. Although this metal denture was never accepted, molding and pouring technique was adopted for manufacturing of vulcanite dentures. [2]

Smith (1957)[7] investigated reinforcement of PMMA by mixing discrete glass fibres with the dough or by lamination with glass cloth and found that reinforcement of fibres did not improved the tensile strength. He also concluded that to strengthen polymer resins by glass fibres, the adhesion of polymer matrix and the fibres should be good because untreated fibres act as inclusion bodies and inhibits the homogenous mixture of acrylic resin and weaken the resin, inspite of strengthening it.

Grant A A and Greener E H in 1967 [14] evaluated the effect of whisker reinforcement of Polymethyl methacrylate denture base resin. Whiskers are microscopic single crystals which possess strengths of the order of 10^6 times that of

the same material in bulk form. The small size of single crystal whiskers reduces the large concentration of point, line and surface defects responsible for the relatively low strengths of polycrystalline aggregates. They used the principle that relatively soft ductile matrix is fully capable of transferring an applied load to short fibres via shear forces at the interface. In this instance the fibres would be the principal load-bearing constituents. In this study Al_2O_3 whiskers were used with diameter between 0.2 to 100µ, sapphire whiskers with diameter of 1 to 10µ and the length of 75 to 125 µ, stainless steel compacted fibres and low-density fibres, boron nitride filaments and silicon carbide whiskers. The results indicated that there is an enhancement of physical properties for all whiskers types studied but addition of Al_2O_3 whiskers in concentration of 10 to 13 % are most effective in reinforcement. The bending strength for sapphire whiskers approximately doubled and 25% changes were noted in the modulus and resiliency. On the basis of the results presented, the study strongly indicates that an enhancement of flexure of denture base polymethyl methacrylate is possible through the technique of whisker reinforcement with sapphire fibres. The results of this study indicate that a systematic investigation of the entire spectrum of physical and chemical properties of sapphire whiskers-polymethyl methacrylate composites may well be in order.

Hargreaves AS in 1969 [17] conducted a survey at Dundee Dental Hospital for 6 months to study the prevalence of fractured denture. She stated that there were 113 denture repairs out of which 68% denture fractured at the end of three years, greater proportion being of partial dentures than complete dentures. 40% of dentures fractured during mastication. The survey concluded that upper denture fractured in the

midline during mastication and lower denture encountered fracture after being dropped.

Schreiber CK in 1971[18] studied the effect of carbon fibres to reinforce polymethyl methacrylate by using untreated carbon fibres and untreated chopped carbon fibres. Bundles of fibres were wet with monomer and incorporated into the polymer to form a thin shit within the matrix. The results showed greatest flexural strength by the reinforcement of surface treated carbon fibres which exceeded acrylic by 50%. While adverse effects were shown by untreated carbon fibres.

In 1971, Berry HH and Funk OJ [19] constructed a strengthener which would reduce or eliminate the lower denture breakage while retaining the properties of the acrylic resin denture base. They quoted that midline fracture of lower denture is common and it might be due to difficulty in cleaning, coughing which leads to pushing out of the denture from the mouth, lack of denture base material at the midline, greater than average biting force, dropping the denture accidently and the like. **Wasserman** suggested the advantages of inserting wire in the lingual crescent area of the acrylic resin base. Other metallic inserts as well as cast reinforcements adds strengths to the denture base. In this study vitallium was inserted in acrylic resin denture base. There was no denture breakage after the strengthener was inserted in the lower denture. The results showed that patients with chronic denture breakage problem can fracture their lower dentures on an average of three times per year. Denture flange and tooth fractures are also common among chronic denture breakers. [20]

Wylegala (1973) [21] investigated the addition of three types (untreated, untreated chopped and surface treated) of carbon fibre for the reinforcement of acrylic resin denture base material. He reported an increase in flexural strength with the addition of surface treated carbon fibre but a decrease in flexural strength with untreated fibres.

In 1981, Beyli MS, Dent M and Fraunhofer JA. [5] studied and analysed the cause of fracture of acrylic resin dentures where the ratio of fracture of upper denture to lower denture is 2:1 with the most common cause of fracture being poor fit and lack of balanced occlusion. They studied stress distribution within dentures using variety of techniques and stated that maxillary dentures are subjected to bending deformation with tensile stresses occurring at labial aspect and lingually to the incisors on the polished surface. He stated that, incisal notch represents a point of weakness which raises the stress and contributes to midline fracture of maxillary dentures resulting from cyclic deformation of the base during function. Any factor that exacerbates deformation of the base or alters its stress distribution will result in denture fracture. A survey of denture fractures, has indicated that most failures occurred when there was deep notching at the midline labial frenum. To prevent the midline fracture they suggest the use of higher strength polymers, notably impact-resistant materials, which will reduce the tendency to fracture

Carroll CE and Fraunhofer JA in 1984 [22] study was conducted 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 and one of four diameters of orthodontic wires: 0.016, 0.025, 0.036, and 0.051 inch

(0.41, 0.64, 0.91, and 1.30 mm). The specimens were subjected to flexural testing under three-point loading at a crosshead speed of 5mm/min. No statistically significant difference was found between the unreinforced and the unlopped 0.016-inch wire specimens, but the looped 0.016-inch specimens were stronger ($p < .05$) than the unreinforced acrylic resin. All other reinforced specimens were significantly stronger ($p < .00l$) than the unreinforced acrylic resin specimens. When stainless steel wire is used as a reinforcing system for autopolymerizing resin, greater flexural strength is obtained with the use of wires of larger dimension.

Yazdaine N and Mahood M in 1985 [23] carried out an investigation to evaluate and compare the flexural strength of standard-sized acrylic resin specimens reinforced with varying amounts of carbon fibre in two different lay-ups. Strands and woven mat fibres were used and both fibres were silane treated to enhance the bonding ability to acrylic resin. The fibres of the woven mat were at right angles to each other and had been held together by passing a barbed wire of 1 mm diameter through them. The strands were oriented along the long axis and the woven mat fibres were arranged parallel & at right angle to the long axis. The modulus of rupture and modulus of elasticity were measured for each composite resin prepared. This investigation has confirmed that carbon fibre acrylic resin composites are stronger and stiffer than unfilled acrylic resin. The experiments show clearly that strands are more efficient strengtheners than are woven mats.

Grave, Chandler & Wolfaardt (1985) [21] compared the flexural strength of samples of cross linked acrylic resin with samples containing various percentages of aramid fibres. All of the reinforced specimens were significantly weaker. A possible

explanation is the failure of adhesion between the fibre and the matrix resulting in the layers of fibre separating the matrix into layers of narrow cross-section. Contrary to this finding **Berrong, Weed & Young (1990)** reported a significant improvement of impact strength with a fibre content up to 2%. **Mullarky (1985)** also reported an increase in the strength and fatigue resistance of acrylic resin appliances reinforced with unidirectional aramid fibre.

Ruyter, Ekstrand & Bjork (1986), [21] in a search for an alternative to a gold framework on titanium implants, discussed the development of a carbon graphite fibre reinforced PMMA and compared the flexural properties in wet and dry conditions to unreinforced PMMA. Fracture stress and flexural modulus were higher for the reinforced than for the unreinforced material.

Solnit G in 1991 [24] studied the effect of methyl methacrylate reinforcement with various types of glass fibres which were silane treated and untreated. The fibres used were cloth form fibres, loose form yellow fibres and loose form white fibres. When the results were compared, samples containing silane treated loose form glass fibres strengthened the PMMA, whereas silane treated cloth form fibre weakened the PMMA when added to the monomer/polymer mixture.

Vallitu PK and Lassila VP in 1992 [25] aimed to study the effect of surface roughness of various metal wires on the fracture resistance of the acrylic resin. The metal wires used were semicircle wire (0 l-0x2-0mm), braided wire plate (0 0-8 x 2-4mm) and clasp wire (0 1-0 mm). Five roughness stages of each wire type were used and sandblasting was done with aluminium oxide (Al_2O_3) with the grain size of 50 and 250µm and the air pressure applied was 5-5 bar. The study concluded that all

metal wires increased the fracture resistance of the acrylic resin significantly when compared with the control group A (P< 0-001), where semi-circular wire had the most marked effect on the fracture resistance of the resin. The braided wire plate did not have as favourable an effect as the semicircle wire. When the different methods of treating the wire surfaces were compared, it was noticed, that the sandblasting was the most effective method with all the metal wires used in this study.

Vallitu PK and Lassila VP in 1992 [26] studied the reinforcement of acrylic resin denture base material with metal or fibre strengtheners where Remanium's spring hard clasp wire (0,1-0 mm), semi-circular wire (0,1-0 x 2-0 mm and l-25x2 50mm) and braided wire plate (0, 0-8x2-4mm) were used as metal strengtheners. All metal strengtheners were divided into two major groups: glossy and sandblasted and fibres were divided into silanized and untreated groups. Sandblasting was done by using 100µm sand. The study concluded that the specimens with the metal wire strengthener were clearly stronger than the specimens in other group and the best strengthener in this study was the semicircular metal wire (0, 1-25 X 2-50 mm), which doubled the fracture resistance of specimens

Vallitu PK in 1993[12] carried out an investigation to study the effect of metal wire bonding to acrylic resin on the fracture resistance of an acrylic denture base material construction with two different bonding methods Silicoating and Eudicolle. Semicircle metal wire (1-0 X 2.0mm) were used and pressed into an aluminium mould. The metal wires were sandblasted with 250 µm grain size aluminium oxide (Al_2O_3) and the air pressure applied was 5.5 bar. The semicircle metal wires were placed in three different positions with respect to the fracture load in the test

specimens. The fracture resistances of the test specimens reinforced with sandblasted metal wires were higher than the resistances of the control specimens. The bonding method of Silicoater increased the resistance significantly when compared to Eudicolle. No significant difference in fracture loads was observed when the effect of different positions of the metal strengtheners in the acrylic resin were compared.

Vallitu PK in 1993 [11] compared the effect of two different silane compounds on the adhesion between glass fibres, carbon fibres and aramid fibres reinforced with acrylic resin, where silane treated and untreated forms of each fibres were tested. 7.39% for glass fibres, 2.08% for carbon fibres and 2.30% for aramid fibres were measured by the weight percentage of the mass of the acrylic resin. The results showed that the silanized glass fibres used as reinforcement significantly increased the fracture resistance due to the good adhesion between fibres and acrylic resin. The glass fibre silanized with compound AP133 did not enhance the fracture resistance significantly, but when the fibres were treated with compound A174 the resistance was enhanced significantly. The aramid fibres treated with silane compounds AP133 or A174 increased the fracture resistance of the test specimens when compound A174 was used.

Ladizesky NH, Cheng YY and Chow TW in 1993 [27] studied the reinforcement of acrylic resin with chopped high-performance polyethylene fibre properties. The purpose of this investigation was to evaluate the mechanical properties of the new system, including the effect of notches. For straight bars the results indicated that incorporation of 2.4 vol% chopped fibres had negligible effect on the mechanical properties of the resin, whereas, the flexural modulus and impact strength

were greatly enhanced when the fibre loading was increased to 37 vol%. Reinforcement with 37 vol % chopped increased the flexural modulus of the resin by over 100%, but did not have a substantial effect on the flexural strength. In summary, the incorporation of at least 30 vol % chopped HDLPE fibre greatly increased the flexural stiffness and impact strength of acrylic denture base resin, while removing the weakening effect of anatomical features such as the frenal notch

Vallitu PK in 1997 [28] studied the effect of reinforcement of ultra-high modulus polyethylene ribbon fibre on the flexural strength of denture polymethyl methacrylare and evaluated the adhesion between denture polymethyl methacrylate and UHMP. Cold gas plasma-treated woven UHMP fibre ribbon that was 4 mm in width was first wetted with methyl methacrylate (MMA) liquid and then dusted thoroughly with polymer powder. One or two layers of ribbon were placed into the silicon mold, which was filled with the mixed PMMA powder and MMA liquid. The acrylic resin was polymerized in a pneumatic curing unit with an air pressure of 0.2 MPa for 15 min at a temperature of +40°C. The bending caused a fracture on the tension side of the test specimen, but the UHMP fibre ribbon held the fractured parts together. The improvement in flexural strength of the test specimens reinforced with UHMP fibre ribbon is modest and its clinical significance is doubtful. The SEM examination of the fibre surfaces of the test specimens suggests that the fracture toughness of these specimens was actually reduced and not increased due to inadequate fibre-matrix coupling.

Stipho HD in 1998 [29] investigated the strength and deflection of repaired acrylic resin joints reinforced with 0%, 1%, 2%, 5%, 10%, and 15% of glass fibres.

Results indicated that acrylic resin reinforced with 1% glass fibres showed highest mean strength before and after repair and demonstrated 48% greater strength than specimens without reinforcement. Specimens treated with 15% of glass fibres showed least deflection pattern as higher concentration inhibits the homogenous matrix of the resin. All treated and untreated acrylic resin specimens demonstrated a significant drop in fracture load and maximum deflection after repair. Reinforcement of PMMA acrylic resin with low glass fibre concentrations was found to enhance the post repair yield and the fracture strength of the resin. No significant mechanical advantages were found by the incorporation of higher than 5% glass fibre contents.

Jagger DC and Harrison A in 1999 [21, 30] studied the flexural strength of reinforcement of acrylic resin with chopped polymethyl methacrylate fibres. 0.75 mm in diameter and 5 mm in length of polymethyl methacrylate fibres were added to the denture base material in 0%, 5%, 15%, 20% & 25% by weight. The study concluded that randomly arranged chopped polymethyl methacrylate fibres have no advantage on acrylic resin when compared to unmodified polymer in terms of strength and chopped polymethyl methacrylate cannot be mentioned as reinforcing agent for denture base material.

Jagger DC, Harrison A and Jandt KD in 1999 [21] reviewed the attempts made to improve the mechanical properties of denture base materials in last few years and found that developments of these materials possess a balance of impact resistance and flexural properties, however, most are not acceptable due to their processing characteristics. They concluded that most popular material to fabricate a denture, having a high impact strength, is a rubber modified acrylic polymer with handling

characteristics similar to conventional poly (methyl methacrylate). The flexural properties of these materials, however, are relatively poor and long-term failure due to fatigue can be a problem.

Vallitu PK in 1999 [31] aimed to study the effect of acrylic resin reinforced with unidirectional and woven glass fibres on flexural properties. The Stick (S) and Stick Net (SN) reinforcements composed of silanized Eglass fibers were preimpregnated with porous polymer. The S reinforcement was made from continuous unidirectional glass fibers with a diameter of 12 µm and SN reinforcement was a weave with fiber thickness of 5 µm. Thickness of preimpregnated woven SN reinforcement was 0.06 mm. Results showed that flexural strength of heat polymerizing acrylic resin polymer was 76 MPa and S reinforcement increased it to 341 MPa. They concluded that Novel glass fiber reinforcements may considerably enhance flexural properties of multiphase dental polymers, which is due to proper impregnation of fibers with polymer matrix.

Chen SY, Liang WM and Yen PS in 2001[32] investigated the mechanical properties of acrylic resin reinforced with Polyester fiber (PE), Kevlar fiber (KF), and Glass fiber (GF) in the concentration of 1%, 2% and 3% by weight. The fibres were cut into 2mm, 4mm and 6mm in length and incorporated in the resin mixture cured at 70°C in a water bath for 13 h, then at 90°C for 1 h. The results showed that the impact strength tended to be enhanced with fiber length and concentration, particularly PE at 3%and 6 mm length, which was significantly stronger than other specimens. Bending strength did not change significantly with the various formulations when compared to a control without fiber.

Foo SH et al in 2001 [33] investigated the effect of polyaramaid fibres on the flexural strength of intact and repaired heat-polymerized denture base acrylic resins where Acron MC, Lucitone 199, and Microlon were used. The treatment groups were intact heat-polymerized PMMA control, PMMA with unreinforced repair, PMMA with polyaramid reinforced repair, intact polyaramid reinforced heat-polymerized PMMA control, polyaramid reinforced PMMA with unreinforced repair, and polyaramid reinforced PMMA with polyaramid reinforced repair. The highest mean strength at fracture was recorded with intact polyaramid reinforced heat polymerized PMMA controls for all resins. Polyaramid fibers did not significantly increase strength to reinforce PMMA repairs.

Jacob John, Shivaputrappa A. Gangadhar, Shah (2001)[33] studied and compared the flexural strength of conventional PMMA resin reinforced with glass, aramid and nylon fibres in loose form. They concluded that glass and aramid fibres appeared to be suitable for long-term use in complete dentures and distal extension partial denture bases, which are considered prone to fracture. Glass fibre reinforcement may also help prevent fracture in provisional fixed partial dentures by strengthening them at the connector sites

T Kanie et al in 2003 [34] studied the flexural properties of denture base polymers reinforced with glass cloth–urethane polymer composite. The silanized glass cloth was sandwiched between two pieces of polyethylene film and pressed to form a sheet of 0.3 mm in thickness, which was light-cured and prepared using four different surface conditions: with or without the polyethylene film and with or without a bonding agent. The results showed that baseline flexural strengths of the self-, heat-,

and light-curing resins were 76.2, 68.6, and 55.6 MPa, respectively, and these values were increased to 271.7, 216.4, and 266.5 MPa by the reinforcement sheet.

Arundhati R and Patil NP in 2006 [35] carried out to evaluate and compare the flexural and impact strength of a new high - impact denture base resin and it was compared with DPI-TUFF, Lucitone 199 and DPI heat cure denture base resins. They concluded that DPI-TUFF high impact denture base resin appeared superior to other resins, with mean flexural strength of 115.0 MPa and impact strength of 18.95 kJ/m· The dry strength of the samples of the materials tested show that it is greater than after immersion of the samples in water at 37ºC for a week. The long curing cycle shows considerably higher values of flexural and impact strength as compared to short curing cycle.

Barbosa D B, Souza R F de, Pero A C, Marra J and Compagnoni M A in 2007, [36] studied the effect of different polymerization cycles on the flexural strength of acrylic resin. A conventional heat polymerized, a microwave-polymerized and a autopolymerizing acrylic resins were used. The microwave-polymerized groups showed the highest means (p<0.05) for flexural strength (MPa), and there were no significant differences among them. The heat-polymerized group (T) showed the lowest flexural strength means and differ significantly from all groups.

Ellakwa et al in 2008 [37] investigated the effect of Aluminium Oxide addition on the flexural strength and thermal diffusivity of heat-polymerized acrylic resin. Concentration of 5%, 10%, 15%, and 20% of aluminium oxide was used by weight and specimens were prepared. Results were analysed and presented that the mean flexural strength values of the heat-polymerized acrylic resin were (in MPa) 99.45,

119.92, 121.19, 130.08, and 127.60. The flexural strength increased significantly by incorporation of 10% Al_2O_3. Thermal diffusivities of the composites were found to be significantly higher than the unmodified acrylic resin. Thermal diffusivity was found to increase in proportion to the weight percentage of alumina filler, which suggested that the proper distribution of alumina powders through the insulating polymer matrix might form a pathway for heat conduction.

Ayad NM, Badawi MF and Fatah AA in 2008, [38] studied and evaluated the effect of reinforcement of high-impact acrylic resin (Metrocryl HI) with zirconia powder on physical and mechanical properties. 5% and 15% of zirconia powder was used by weight and specimens were fabricated and subjected to flexural strength test. The results showed that addition of zirconia significantly increased the flexural strength of high impact acrylic resin. Increase in flexural strength was directly proportional to the concentration of zirconia powder.

Yadav et al in 2012, [39] studied the effect of reinforcement of silane treated silver and aluminium microparticles on strength and thermal conductivity of polymethyl methacrylate. The study was carried out in two parts where in Part 1 the effect of addition of 10%, 20% and 30% of metal fillers by weight was carried out on the tensile, compressive, and flexural strength of PMMA. In part II, 10 edentulous patients were clinically evaluated by providing two sets of complete dentures, one without reinforcement and one with 20% aluminium particle filled on the palatal portion of the upper denture. The results showed mean tensile and flexural strength values among control and other groups were found to have statistically significant differences. all 10 participants reported higher perception of hot and cold sensations

in dentures with a metalized palatal portion. They concluded that compressive strength increased progressively on increasing the filler concentration for both silver- and aluminium-filled PMMA. Silane-treated metalized PMMA showed reduction in tensile and flexural strength at 30% concentration. Metalized dentures led to an appreciable increase in thermal perception by the participants of this study.

M Vojdani et al in 2012 [40] studied the effect of aluminium oxide reinforcement on the flexural strength, surface hardness and roughness of heat polymerized acrylic resin. 0.5%, 1%, 2.5% and 5% of aluminium oxide was measured and flexural strength was assessed with a three-point bending test using a universal testing machine. The results showed 2.5% of Al_2O_3 significantly increased the flexural strength compared to the control group. The Vickers hardness significantly increased after incorporation of 2.5 and 5% Al_2O_3. No significant difference was detected in surface roughness levels between the reinforced and control groups.

Jasim BS and Ismail IJ in 2014 [10] study was to evaluate the effect of addition of surface treated Aluminium oxide nano fillers on some properties of heat cured (PMMA) where silanized (Al_2O_3) nanoparticless was added to PMMA powder by weight in three different percentages 1wt%, 2wt% and 3wt% and mixed by probe ultrasonication machine. A highly significant increase in flexural strength was observed with the addition of (Al_2O_3) nanoparticless to (PMMA) at the percentage of 1wt%, the value was 117.72 Mpa and significant increase at 2wt%; while a significant reduction occurred in flexural strength at the percentage of 3% the value was 90.110 Mpa. They concluded that addition of Al_2O_3 nanoparticless to acrylic resin improves the thermal properties and flexural strength of acrylic resin at the same time this addition decreases water sorption and solubility.

Alwan SA and Alameer SS in 2015 [16] conducted a study the effect of addition of 3% weight of treated (silanized) Titanium oxide Nano filler on some physical and mechanical properties of heat cured acrylic denture base material. They concluded that addition of Titanium dioxide Nano particle to heat cure acrylic resin improve the impact strength, flexural strength and surface hardness of heat cure acrylic resin and at the same time this addition decreases water sorption and solubility.

Asopa V et al (2015) [42] and compared the flexural strength, impact strength; surface hardness and water sorption of 10% and 20% zirconia (ZrO_2) reinforced high impact acrylic resin with that of high impact acrylic resin. They concluded that the addition of zirconium oxide as a filler in the high impact acrylic resin increases their flexural strength. Impact strength and surface hardness of the zirconia reinforced specimens were found to have relatively lesser values as compared to the control specimens. Water sorption of the zirconia reinforced specimens was found to increase but was within the limit of ADA Specifications No. 12.

Gad et al in 2017 [15] reviewed the enhancement of acrylic denture base resin during the past few decadesby giving specific attention to the effect of fiber, filler, and nanofiller addition on poly(methyl methacrylate) (PMMA) properties. He concluded that

- Glass fiber reinforcement significantly increases the mechanical properties of PMMA. Natural fibers (OPEFB) and vegetable fiber can be used, but further investigations are needed.

- Obvious enhancement in the properties of denture base resin material properties was found with the addition of NPs and nanotubes, depending on the application and manipulation.
- Silane coupling agents play a central role in improving bonding between fillers and the resin matrix, and they subsequently improved the resin's properties. The newest reinforcement system is a hybrid one.
- Hybrid fiber, hybrid fillers, or hybrid fiber and filler may considerably enhance the properties of PMMA.

Arora et al in 2017 [43] evaluated the flexural strength, hardness, and impact strength of heat‑cured high‑impact denture base resins with different polymer/monomer ratios. The samples were divided into five groups based on different powder/liquid ratios (g/ml) with. The polymer/monomer ratio in Group 1 (Ratio ‑ 2.2:1) was the manufacturer's recommended ratio and was used as control. In Group 2, the ratio was 2.7:1, in Group 3, the ratio was 3.2:1, in Group 4, the ratio was 1.9:1, and Group 5 the ratio was 1.6:1. The results showed that the flexural strength values and VHN values showed a similar trend. The values decreased significantly as the ratio was increased or decreased from the control group. He concluded that for reinforcing resins or high impact resins, the manufacturer's recommended polymer/monomer mixing ratio should be used to obtain the appropriate strength of the material.

Material and Method

A necessary process for the artist is one which begins with a point of view, a reaction to the environment, to people, to whatever it is one is concerned with.

Flexural failure of denture base resins is considered the primary mode of clinical failure.[44] Hence, the ultimate flexural strength of a material reflects its potential to resist catastrophic failure under a flexural load. High flexural strength is crucial to the long-term success of dentures. Modifications in the composition of conventional acrylic resin denture base material can be done to achieve this purpose.

Recently, much attention has been directed toward the incorporation inorganic nanoparticles into PMMA to improve its properties. The properties of polymer nanocomposites depend on the type of incorporating nanoparticles, their size and shape, as well as the concentration and interaction with the polymer matrix. Nanoparticles were surface treated with silane coupling agent and embedded into PMMA. [9]

Hence this study was undertaken to evaluate and compare the flexural strength of heat polymerized acrylic resin denture base material reinforced with 3% silanized titanium dioxide nanoparticles and 1% silanized aluminium oxide nanoparticles with conventional heat polymerized polymethyl methacrylate denture base resin.

Material and methods are divided under following headings-

I. Materials

II. Armamentarium and equipments

III. Method

1. MATERIALS: -

SR. NO.	MATERIALS	MANUFACTURER	BATCH NO.
1	Heat polymerized acrylic resin	DPI Heat Cure™, (Dental products of India Ltd)	12152
2	Die stone	Ultrarock; Kalabhai Karson Pvt Ltd, India	161003
3	Aluminium oxide nanoparticle	Reinste	A-AlO-132
4	Titanium dioxide nanoparticle	Sigma Aldrich	MKBZ9326V
5	Silane coupling agent 3 methacryloxypropyltrimethoxy silane	Sigma Aldrich	SHBG2374V
6	Toluene	MERCK	ID21F62193
7	Cold mould seal (separating medium)	Pyrax	9151

II. ARMAMENTARIUM AND EQUIPMENTS: -

The required armamentarium and equipments are-

a) High accuracy balance

b) Ultrasonicator

c) Magnetic stirrer

d) Vacuum rotary evaporator

e) Acrylizer with thermostat

f) Universal testing machine

g) Rubber bowls and plaster spatula

h) Sand paper (No.120)

i) Varsity flasks and clamps

j) Camel hair brush

k) Vernier caliper

l) Porcelain jar

m) Glass beaker

n) Sterile syringe

o) Mixing spatula

p) Petroleum jelly

q) Para-film

r) Brass metal dies

s) Hydraulic bench press

t) Distilled water

III. METHODOLOGY:-

The basic methodology consisted of-

1. Die preparation

2. Silanization of aluminium oxide nanoparticles

3. Silanization of titanium dioxide nanoparticles
4. Preparation of gypsum mold for specimen fabrication
5. Preparation of heat polymerized polymethyl methacrylate denture base material specimen (Group C)
6. Preparation of heat polymerized polymethyl methacrylate denture base material specimen reinforced with silanized aluminium oxide nanoparticles (Group A)
7. Preparation of heat polymerized polymethyl methacrylate denture base material specimen reinforced with silanized titanium dioxide nanoparticles (Group T)
8. Testing of specimens for flexural strength.

90 specimens will be prepared in total with 30 specimens in each group.

Group C	The control group; heat polymerized polymethyl methacrylate denture base material without reinforcement. (n=30)
Group A	Heat polymerized polymethyl methacrylate denture base material reinforced with 1% Aluminium oxide nanoparticles. (n=30)
Group T	Heat polymerized polymethyl methacrylate denture base material reinforced with 3% Titanium dioxide nanoparticles. (n=30)

1. Die preparation:

Three metal dies of dimensions 65mm in length, 10mm in width, and 3mm in height were fabricated to prepare molds for fabrication of heat polymerized polymethyl methacrylate denture base material specimens. (ISO 1567 standard) [37, 40]

These metal dies have threaded holes, 8mm from the corner of the dies, which were 5mm in diameter and 3mm in depth. Screws were engaged through these holes for easy removal of the dies from the stone mold.

2. Silanization of aluminium oxide nanoparticles:

4gms of aluminium oxide nanoparticles were added to 100ml of toluene in a glass beaker and sonicated for 20mins at room temperature. After sonication, magnetic stirrer was placed in the beaker and kept on the stirring machine at room temperature for 30mins. After this 0.2ml of silane coupling agent (3-methacryloxy propyl trimethoxy silane) was added slowly, drop by drop under rapid stirring for homogenous mixing of silane coupling agent with aluminium oxide nanoparticles and toluene solvent. [45, 46]

Parafilm was used to cover the beaker and left standing for 2 days. Toluene solvent was then removed by rotatory evaporator under the vacuum at 60℃ and rotation of 150 rpm for 30 mins. The silanated aluminium oxide nanoparticles were dried in a vacuum oven at 60ºC for 10hrs. [47]

3. Silanization of titanium dioxide nanoparticles:

100 ml of ethanol aqueous solution (70 vol %) was prepared using 99.8 vol% ethanol and de-ionized water (30 % vol), and adjusted to pH of 4.5 using PH meter

through titrating with 99.9% acetic acid. To it 0.1125ml of silane coupling (5% wt to nano filler) [47] agent (3-methacryloxy propyl trimethoxy silane) was added to ethanol aqueous solution, and stirred using magnetic stirrer. After this 5g of titanium dioxide nanoparticles were added into the silane solutions and the mixture was stirred with magnetic stirrer for 20 minutes followed by sonication of the mixture with probe sonication apparatus for 30 minutes. The solution was left to dry at room temperature for 14 days. [16]

4. Preparation of gypsum mold for specimen fabrication:

Gypsum mold was prepared with the help of prefabricated brass metal dies. Carding wax was used to block the threaded holes on the dies and thin layer of petroleum jelly was applied and invested in the lower half of the flask. Die stone was used for base flasking where half the thickness of the metal dies were embedded in it carefully. After setting of the die stone, thin layer of petroleum jelly was applied on the metal dies & to the investment material and then counter flasking was done. [35]

After 1 hour, the flasks were opened and carding wax was removed from the threaded holes. The dies were then engaged with the screws and gently pulled out. [48] The molds were then treated with hot water to remove the traces of petroleum jelly and wax. This also facilitates application of separating medium. Thus, the mold cavities obtained was used for fabrication of specimens.

5. Preparation of heat polymerized polymethyl methacrylate denture base specimens (control group C):

30 samples were prepared using the conventional heat polymerized polymethyl methacrylate denture base material as per manufacturer's instruction, where monomer and polymer was mixed in ratio of 1:2.5 by weight. [49]

With the use of high accuracy balance to weigh the materials, 7.5gms of polymer powder and 3ml of monomer was used for fabricating 3 specimens (for 1 flasking). Packing was done at dough stage, following trial closure. Final closure was performed under hydraulic bench press at a pressure of 3000 psi for 3min. The flasks were clamped and maintained under pressure for 1 hour. [50] The flasks were immersed in water at room temperature and slowly raise the temperature up to 74ºC which was held for 2 hours. After the completion of 2 hours, temperature was raised to 100ºC and was maintained for 1 hour. After the completion of curing cycle, flasks were removed from water and allowed to bench cool at room temperature before deflasking. [50]

The polymerized specimens were retrieved carefully and finishing was done using sand paper (no. 120). The finished specimens were stored in distilled water for 1 week at room temperature. [6,40]

6. Preparation of heat polymerized polymethyl methacrylate denture base material reinforced with silanized aluminium oxide nanoparticles (group A):

30 samples were prepared using conventional heat polymerized polymethyl methacrylate denture base material reinforced with silanized aluminium oxide nanoparticles. 7.425gms of polymer, 3ml of monomer and 0.075gms of silanized aluminium oxide nanoparticles were used for fabrication of 3 specimens. [45,47]

High accuracy balance was used to weigh the material. By using ultra sonicator, silanized aluminium oxide nanoparticles was well dispersed in the monomer, which was done at 120W, 60KHz for 3mins.

Polymer powder was then added gradually to reduce the possibility of particle aggregation. Mixing, flasking, packing and curing was done in the same manner as done for control group. The finished specimens were then stored in distilled water for 1 week at room temperature.

7. Preparation of heat polymerized polymethyl methacrylate denture base material reinforced with silanized titanium dioxide nanoparticles (Group T):

30 samples were prepared using conventional heat polymerized polymethyl methacrylate denture base material reinforced with silanized titanium dioxide nanoparticles. 7.275gms of polymer, 3ml of monomer and 0.225gms of slianized titanium dioxide nanoparticles were used for fabrication of 3 specimens.

High accuracy balance was used to weigh the material. By using ultra sonicator, silanized titanium dioxide nanoparticles were well dispersed in the monomer, which was done at 120W, 60KHz for 3mins.

Polymer powder was then added gradually to reduce the possibility of particle aggregation. Mixing, flasking, packing and curing was done in the same manner as done for control group. The finished specimens were then stored in distilled water for 1 week at room temperature. [45,47]

8. Testing of specimens:

Testing was carried out at metallurgical laboratory where each group of specimens were tested for flexural strength. It is a 3-point bending test which is useful in comparing flexural strength as it will stimulate the type of stress which is applied to denture during mastication.

Flexural strength was tested with universal testing machine at a 5mm/minute crosshead speed.[6] A jig was present to support the specimen which was separated at a distance of 50mm.

Flexural strength (FS) was calculated with the help of the formula-

$$FS = {}^{3PI}/_{2bd^2}$$

Where, FS= flexural strength

P= load

I= distance between supporting wedges (mm), b= width of the specimen(mm)

d= thickness of the specimen (mm) [40]

PLATE I

Fig 1: Heat polymerized acrylic resin

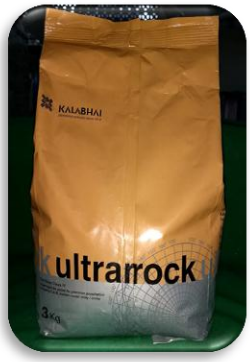

Fig 2: Die Stone

Fig 3: Aluminium oxide Nanoparticles

Fig 4: Titanium dioxide Nanoparticles

Fig 5: Silane coupling agent

Fig 6: Toluene

PLATE II

ARMAMENTARIUM AND EQUIPMENTS

Fig 7: High accuracy balance

Fig 8: Ultrasonicator

Fig 9: Magnetic stirrer

Fig 10: Hot air oven

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: Porcelain jar, petroleum jelly, dapen dish, sandpaper (No. 120), camel hair brush, sterile syringe, mixing spatula

Fig 15: Vernier caliper

Fig 16: Separating medium

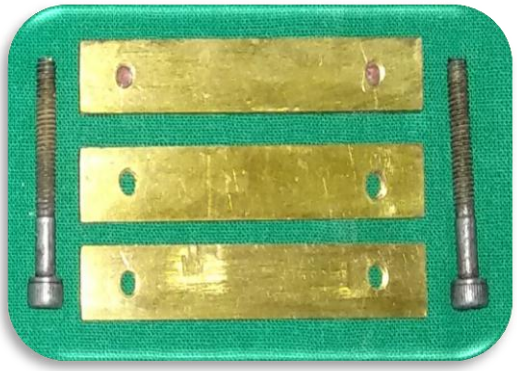

Fig 17: Brass metal dies

Fig 18: Parafilm

Fig 19: Hydraulic bench press

Fig 20: Distilled water plant

PLATE IV

METHODOLOGY

Fig 21: Silanization process of titanium dioxide nanoparticles

Fig 22: Silanization process of aluminium oxide nanoparticles

Fig 23: Preparation of gypsum mold to obtain specimens

Fig 24: Testing of specimens

PLATE V

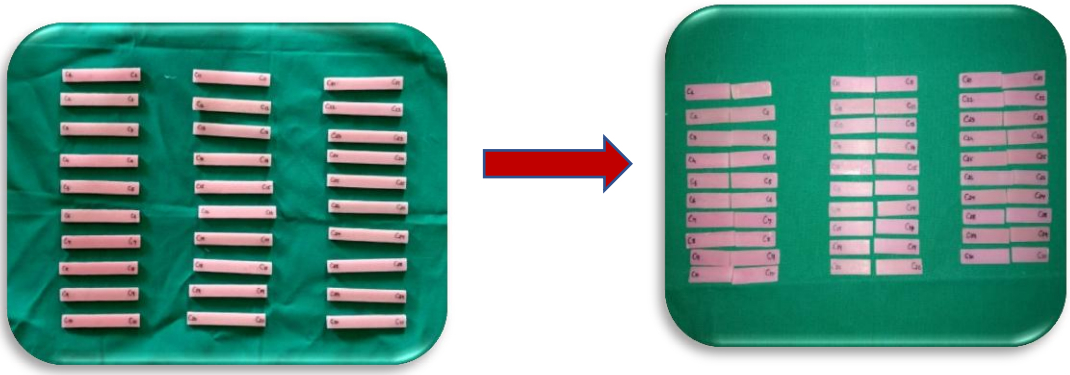

Fig 25: Group C. The Control group. Heat polymerized acrylic resin without reinforcement, before and after testing of flexural strength.

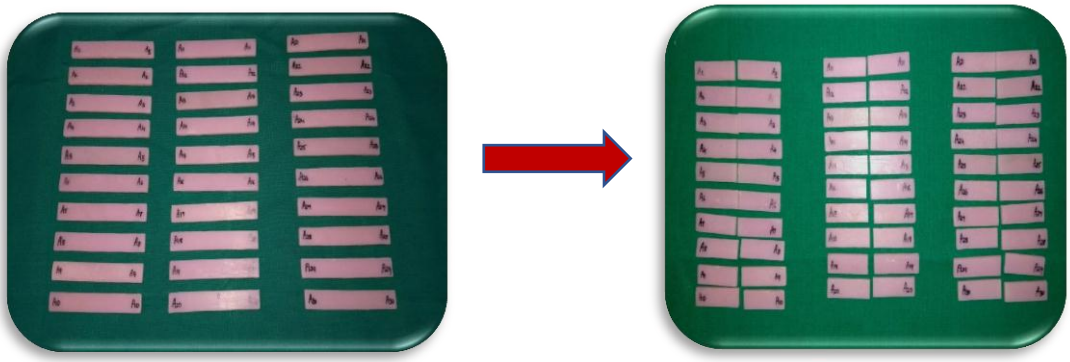

Fig 26: Group A. Heat polymerized acrylic resin reinforced with 1% silanized aluminium oxide nanoparticles, before and after testing of flexural strength.

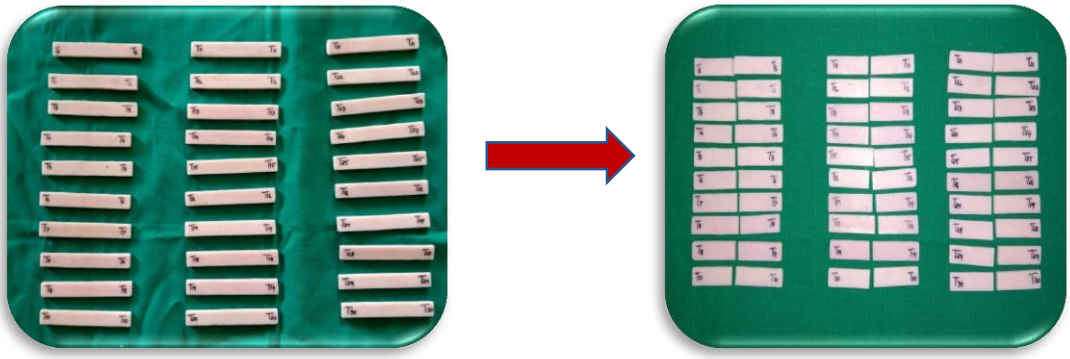

Fig 27: Group T. Heat polymerized acrylic resin reinforced with 3% silanized titanium dioxide nanoparticles, before and after testing of flexural strength.

Results

The achievements of an organization are the results of the combined effort of each individual.

In this study flexural strength of heat polymerized polymethyl methacrylate acrylic resin specimen reinforced with 3% silanized titanium dioxide nanoparticles and 1% silanized aluminium oxide nanoparticles was evaluated and compared with the conventional heat polymerized polymethyl methacrylate denture base resin specimens.

90 samples were prepared in total and were divided into three groups, including 30 samples in each group

DISTRIBUTUON OF SAMPLES INTO GROUPS

Sr. No	Code	Group	n= no. of samples
1.	**C**	The control group; heat polymerized polymethyl methacrylate denture base material without reinforcement. (n=30)	n=30
2.	**A**	Heat polymerized polymethyl methacrylate denture base material reinforced with 1% Aluminium oxide nanoparticles. (n=30)	n=30
3.	**T**	Heat polymerized polymethyl methacrylate denture base material reinforced with 3% Titanium dioxide nanoparticles. (n=30)	n=30

Specimens of each group were subjected to flexural strength test on Universal Testing Machine at a crosshead speed of 5.0mm/min. The maximum load was determined from the chart and recorded as fracture load in N (Newton). The flexural strength was calculated in MPa and the results were statistically analysed.

STATISTICAL ANALYSIS:

The data was analysed using SPSS statistical software. The *p* value was taken as significant when less than $p < 0.05$

The statistical test used for the analysis of the results were:

1. One-way ANNOVA by K. Wallis test
2. Tukey's post-hoc test

The mean, median and standard deviation were calculated by ANOVA (one-way analysis of variance) and pair wise comparison of means was carried out and tested for statistical significance used Tukey's post-hoc test.

Mean is sum of all observations and divided by number of observations.

Median is value of the variable that divides the distribution in two equal parts i.e. 50% of the observations will be below and above it.

Standard Deviation is summarized as the amount of variation (change) in the observation from their average value (mean).

Standard Deviation: It is the most frequently used measure of deviation. It is defined as root mean square deviation and is denoted by s or SD.

The formula for calculating standard deviation:

$$\boldsymbol{SD = \sqrt{\frac{\Sigma(\bar{x} - x)^2}{n - 1}}}$$

$\bar{x}$	=	Mean
x	=	The values of variable
Σ	=	Sum of the value
n	=	Number of the observations
Min	=	Minimum value
Max	=	Maximum value

RESULTS:

Table 1 provides the descriptive statistics for flexural strength of test samples in three study groups. The mean of **Group C** was minimum i.e. **89.18 MPa** and the strength ranged between 60.43 to 112.61 MPa. For **Group A,** the mean flexural strength was **107.59 MPa** and ranged between 100.76 to 120.21 MPa. In **Group T**, the mean flexural strength was **105.73 MPa** and ranged between 99.90 to 112.82 MPa. A graphical visualization of mean strength along with error bar is given in the Graph I.

Table 2 reveals that the mean flexural strength across groups differed highly significantly across three groups, as indicated by p-value < 0.000 (1.4123E-14). 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 highly significant difference in the means of Group C and Group A ($p < 0.000001$). Also, the difference in the means of Group C and Group T was statistically significant ($p < 0.000001$).

However, the difference between Group A and Group T was statistically insignificant as revealed by p-value of **0.647** ($p > 0.05$). The same has been graphically shown through bar chart in Graph II

Discussion

"Good directors don't answer questions with their work. They generate debate and create discussion."

Acrylic polymers were utilized as denture base materials since 1937 and by 1946, 98% of all denture bases were based on PMMA.[2] In 1942, vinyl acrylic copolymer and in 1948 polystyrene, a styrene polymer developed by **Charles Dimmer**, were introduced as denture base materials which had greater flexural strength and high residual stresses. [2] **Faber (1957)** described the technique for fabrication of lower cast metal bases. According to **Faber, Peyton (1943) and Skinner (1951**) suggested the use of metal base as it causes fewer tissue changes.[2]

Poly (methyl methacrylate) (PMMA) is frequently used to fabricate denture bases due to its low cost, biocompatibility, ease of processing, stability in the oral environment, and acceptable aesthetics. However, having all these advantageous properties, PMMA is not considered as an ideal denture base material because of its inferior physical and mechanical properties. [15] The denture base material should have sufficient strength and resilience to withstand normal masticator forces; withstanding sudden shock caused by impact forces is an important property. Such material should not creep under masticatory loads for long-term use if good occlusion is to be maintained and potential irritant effects are kept at minimum.[13]

An ideal denture base material should have adequate mechanical and physical properties, besides biocompatibility and aesthetics. Several studies have been conducted with the goal of enhancing the properties of PMMA by using different curing methods and/or incorporating fillers in its composition. Addition of fillers and fibres to PMMA is a commonly used method to improve both its physical and mechanical properties.[15]

Although PMMA has been commonly utilized in the fabrication of removable denture bases, a number of polymeric materials, such as high-density polyethylene (HDPE), polyamide (PA), and poly(*L*-lactide) (PLLA), have been studied for their prosthodontic applications. Furthermore, polystyrene polyvinyl acrylic and light-activated UDMA have also been used in the construction of denture bases. These materials must be durable and strong enough to withstand masticatory forces, particularly for patients with parafunctional habits. However, none of these polymers provides the unique combination of physical and aesthetic properties exhibited by

PMMA. Despite these excellent properties, improvement in the fracture resistance of PMMA is needed.

The ultimate flexural strength of a denture base material is directly proportional to its potential to resist catastrophic failure under a flexural load. As a foundation, the acrylic resin materials should exhibit a high proportional limit to resist plastic deformation and also exhibit fatigue resistance to endure [4,51,52] repeated masticatory loads. An acrylic resin capable of sustaining higher flexure in combination with high resistance to cyclic loading may be less prone to clinical failure.[6]

Smith DC analyzed the fracture of dentures and showed two types of failure which are outside the mouth, caused by impact forces, i.e. a high stress rate and inside the mouth which is probably a fatigue phenomenon, i.e. a low and repetitive stress rate. These considerations led to conclusion that denture fracture occurs through flexural fatigue under the respective conditions. **Hargreaves** in his study concluded that at the end of 3 years provision 68% of dentures fractured and 40% fractured at the time of mastication.

Beyli et al stated that the ratio of upper denture fracture to the lower denture fracture is 2:1 with the common cause of poor fit and lack of balance in the denture. He suggested that midline fracture of a denture base is caused due to flexural fatigue failure which is a result of cyclic deformation of the base during function. He stated that any factor that exacerbates deformation of the base or alters its stress distribution will predispose the denture to fracture.[5] Some of the factors responsible for denture fracture include stress intensification, increased ridge resorption leading to an

unsupported denture base, deep incisal notching at the labial frenum, sharp changes at the contours of the denture base, deep scratches, and induced processing stresses. [5,13] The most promising approach to preventing or reducing the incidence of this problem appears to be reinforcement in the anterior part of the palate of the denture. [5]

Huggett and Harrison conducted a questionnaire survey and reported that 29% of midline fracture occurred due to flexural fatigue failure and mostly in upper dentures. These considerations led to a general conclusion that denture fracture occurs due to flexural fatigue under respective conditions and due to this reason flexural strength test were selected as most relevant to evaluate the strength of denture base resin. Studies have shown that the average values of flexural strength of heat polymerizing acrylic resins are near to **78-92 Mpa**.[5.] The mean flexural strength of conventional heat polymerized polymethyl methacrylate without any reinforcement recorded in the present study was **89.18MPa** which was comparable with the previous studies.

As the fracture resistance of a denture base resin is important, attempts have been made to improve the mechanical properties of acrylic resin by giving maximum bulk to the material in the regions most heavily stressed by copolymerization and cross linking, reinforcement with glass fibers, aluminium and sapphire whiskers, polycarbonates, carbon fibers, and the addition of metal strengtheners. These reinforcing methods were done to improve the mechanical properties of the denture base resins and to overcome the problem of denture fractures.[23,53]

Yazdaine N and Mahood M studied the flexural strength of acrylic resin reinforced with various amounts of carbon fibres. They confirmed that carbon fibre

acrylic resin composites are stronger and stiffer than unfilled acrylic resin and strands are more efficient strengtheners than are woven mats.[23]

Mullarky (1985) and Berrong, Weed and Young (1990)[54] studied the reinforcement of acrylic resin with aramid fibers. They were successful in enhancing the fatigue resistance of the aramid fiber reinforced acrylic resin denture base material. But the incorporation of aramid fibers produced similar problems with respect to colour. The yellow appearance of the fibers was difficult to mask within the denture, necessitating thick layers of acrylic resin that added significantly to the bulk of the denture.

The concept of self-reinforcement (with a material that is chemically identical to the matrix holding the fiber in place) has been studied by **Jagger, Harrison and Jandt (2000)**. [37] Unfortunately, the effect of the addition of untreated and surface treated chopped PMMA fibers did not produce a significant improvement in either the flexural strength or impact strength of acrylic resin. They found that the fiber arrangement in terms of fiber displacement and inter fiber spacing, may be important factors in the success of the reinforcement. [6]

It has hypothesized that the addition of synthetic fibers to the monomer/polymer mixture may strengthen the resultant acrylic resin. Many attempts, however, to strengthen acrylic resin in this way failed because stress concentrations occurred around embedded materials, and the net effect of embedding fibres or metals resulted to weaken the polymer. This is due to poor adhesion between the fibre, metal inserts and acrylic resin matrix.[37] **Hero *et al.*, 1987; Naegeli *et al.*, 1988; Berzins, 1989; Kappert *et al.*, 1989** suggested that silanization of the surface of the metal with

different techniques improves the adhesion between acrylic resin and the filler particle which enhances the fracture resistance of the denture base material.[12] These considerations led to the process of silanization of the filler particle before incorporating to the acrylic resin. [11]

Solnit G studied the strength of polymethyl methacrylate after reinforcing with silanated and non-silanated glass fibres. And concluded that silane-treated glass fibers in a loose form significantly strengthened PMMA when compared to the samples with untreated fibers, but not when compared to the control samples.[24]

He stated that untreated fibers act as inclusion bodies in the acrylic resin mixture and, instead of strengthening, it actually weakens the resin [14, 24]. The fibers may break up the homogeneous matrix. Silane coupling agents chemically bonds the glass fibers to the resin matrix and make the mixtures more homogeneous, resulting in stronger PMMA. [24, 55]

Clark & Plueddeman (1963), Rosen (1978) suggested that increase in the fracture resistance of an acrylic-fibre composite **is** dependent on the adhesion between the acrylic resin matrix and the fibres and silane compounds can be utilized to improve the adhesive properties of the fibres.[11]

Vallitu PK evaluated and compared the effect of two different silane compounds on the adhesion between glass fibres, carbon fibres and aramid fibres reinforced with acrylic resin. He concluded that acrylic resin reinforced with silanized glass fibres significantly increased the fracture resistance due to the good adhesion between fibres and acrylic resin.[11]

Ihab (2011) [45] gave a method for homogenous dispersion of filler particles in polymer matrix. It plays a major role in the mechanical properties of particulate-filled polymer composites. The reason for increase in flexural strength with addition of aluminium oxide powder was attributed to proper distribution of alumina spheres within denture base powder which acts as potential fillers in the resin matrix. These considerations led to the process of silanization of the filler particle before incorporating to the acrylic resin.[11]

In methacrylic resin based dental composites, adhesion between the polymeric matrix and the reinforcing filler is usually achieved by the use of silane coupling agent, 3-methacryloxypropyltrimethoxysilane (MPTMS). It is a bifunctional molecule capable of reacting via its alkoxysilane groups with the filler and itself, and with the resin through its methacrylate functional group. The overall degrees of reaction of the silane with the glass filler (oxane bond formation), with itself (by siloxane formation), and with the resin system (by graft copolymerization) determine the efficacy of the coupling agent. For a given resin/filler system, the physical-chemical nature of the silane agent, (e.g., chemical structure, molecular size, degree of hydrophobicity, reactivity, functionality), the silanization procedure employed, the silane layer orientation that develops and the extent of filler coverage are important parameters that determine many of the physicochemical and mechanical properties of the interphase. [56,57]

In this study, aluminium oxide nanoparticles and titanium dioxide nanoparticles were silanized with 3-methacryloxypropyltrimethoxysilane (MPTMS) silane coupling agent.

Recently, much attention has been directed toward the incorporation of inorganic nanoparticles into PMMA to improve its properties [16, 58]. The properties of polymer nano composites depend on the type of incorporated nanoparticles, their size and shape, as well as the concentration and interaction with the polymer matrix. Nanoparticles have been increasingly used in material science for its wear and tear resistance and anti-corrosion abilities. The principle behind the usage of nanoparticles is that alteration of filler size is considered responsible for the performance of the material (PMMA) in aspects of both polishability and fracture resistance. Many metals such as aluminium oxide, cobalt-chromium, silver, zinc oxide, zirconia and most commonly titanium dioxide have been used in experiments to improve the mechanical properties of PMMA.[59]

TiO_2 particles are preferred in dentistry because of their pleasing colour and high biocompatibility. TiO_2 nanoparticles with the elastic modulus approximately 230 GPa. Other characteristics such as white colour, biocompatibility, and high stability and efficiency, as well as availability and low cost, have made TiO_2 an appropriate antimicrobial additive for dental materials. Titania nanoparticles have been used as an additive in dental materials to match the opaque properties of teeth and to enhance the mechanical properties of dental resins.

Thorat et al. prepared and characterized bis-GMA resin dental restorative composites with glass, silica, and titanium fillers. The researchers concluded that TiO_2 fillers could be useful in future applications because their photocatalytic effects promote local antibacterial or remineralization reactions. Likewise, studies have been performed by **Asar NV et al, Alwan SA and Alameer SS** to modify dental

composites by incorporating TiO_2 nanoparticles into a standard dental acrylic. Such studies have reported that the most available commercial product for dental restorations could be improved through the addition of nano TiO_2. [16,41]

In addition, the mechanical behaviour of TiO_2 nanoparticles reinforced resin-based dental composites was investigated by **Hua et al**. This study demonstrated that the mechanical advantage of nanocomposites over microcomposites could be found in the reinforcing effect of nano TiO_2 with 3% volume fraction on the stiffness, which is the same as a glass fiber with twice the volume fraction. This result is consistent with previous studies, which indicated that dental composites reinforced with 3% nano TiO_2 exhibited superior mechanical properties compared to the control, with minimal effects on flowability and radiopacity. [13,16]

Several studies have investigated the effect of adding titanium dioxide (TiO_2) on the properties of PMMA. It was found that adding TiO_2 particles could improve the flexure strength, fracture toughness, hardness of PMMA, as well as thermal conductivity. In addition, a significant increase in impact strength and a significant decrease in water sorption and solubility were found upon addition of TiO_2 to PMMA.[15]

Safi et al found that modifying PMMA with TiO_2 nanoparticles has an effect on its thermal and mechanical stability, while a reduction in flexural strength and toughness was reported. Adhesion between the resin matrix and filler particles is very important in order to enhance the composite's properties. Accordingly, a titanium coupling agent could be useful for improving the properties of titanium-reinforced PMMA. Incorporation of silanized TiO_2 nanoparticles in PMMA improved the impact

strength, flexural strength, and surface hardness of the resin and decreased its water sorption and solubility.[15]

Titania (TiO_2) was used in the present study because it is a biocompatible material (non-toxic) and with pleasing colour. [60,61] Due to high interfacial shear strength between nano fillers and matrix, it resulted in an increase in the value of flexural strength, and decrease in the crack propagation by good bonding between nano filler and matrix [62.] This resulted because, the total particle/matrix interfacial surface area available for energy dissipation increase and the critical stress for particles/matrix debonding also increase.[63] There was an increase in the value of flexural strength when 3% of nano TiO_2 were added to PMMA compared with the control group with the mean value of **105.73MPa** which was comparable with the study performed by **Alwan SA and Alameer SS in 2015** with the mean value of **117.92MPa.**

Arora et al recently reviewed the effect of alumina addition and reported a positive impact on the properties of acrylic resin. Addition of alumina powder to acrylic resin improved its thermal conductivity and, accordingly, patient satisfaction was expected to increase. In addition, reinforcing PMMA with aluminium increased the flexural strength, impact strength, tensile strength, compressive strength, and surface hardness of the resin. Warpage also decreased significantly after addition of aluminium to PMMA. On the other hand, some studies found that adding aluminium decreases both the impact and tensile strength of PMMA. Treating aluminium oxide particles with a coupling agent increased the flexural properties of acrylic resin. Also, silane-treated aluminium particles significantly increased the compressive, tensile, and flexural strength and the wear resistance of reinforced denture base resin.[15]

Safi et al concluded that adding Al_2O_3 nanoparticles to PMMA increases its thermal stability compared with pure PMMA. Addition of silanized Al_2O_3 nanoparticles to acrylic resin improved the thermal properties and flexural strength of acrylic resin, and at the same time this addition decreased water sorption and solubility. A recent study by **Abdulkareem MM, Hatim NA** reported that alumina nanoparticles have a good level of biocompatibility when added to microwave-treated and untreated PMMA powder. **Jasim BS, Ismail IJ** stated that aluminium-reinforced PMMA causes discoloration of the resin, which limits its use to areas where it is not visible. Although **Kul E, Aladağ Lİ, Yeşildal R**. indicated the addition of Al_2O_3 to PMMA significantly increased thermal conductivity, the flexural strength values of PMMA were not significantly changed. [15]

Pisaisit et al [10,64.] added silanized and nonsilanized alumina to PMMA, and by SEM image they found there was a gap between nonsilanized alumina particles and resin matrix this could explain reduction in mechanical properties.

In this study, the addition of silanized Al_2O_3 nanoparticles showed the mean value of flexural strength was **107.59MPa** which was comparable with the study conducted by **Jasim BS, Ismail IJ** showed that there is an increase in the value of flexural strength with 1wt% significantly compared to control groups.

H. H. Chandler et al (1971) [65] suggested that one of the deficiencies of the available denture base resins is their radiolucency. Thus, there are chances of ingestion or aspiration of either broken or portions of ill-fitting complete dentures by the patient. **Sehjpal and Sood (1989)** [66] stated that reinforced PMMA with metal oxide fillers like silver, copper, aluminium, zirconium and titanium dioxide not only

increases strength but also provides radio-opacity to the heat polymerized denture base material.

The test specimens utilized in this study were prepared according to ISO 1567 standardization with the dimensions of 65 × 10 × 3 mm from heat polymerizing acrylic resin.[59, 67] The thickness and width of each specimen were measured with a digital caliper.[67] Specimens were tested for flexural strength using a three point-bending test with a universal testing machine. The distance between supports was 50 mm. A load was applied to the specimens by a centrally located rod until fracture occurred. The chart recorder on the testing machine produced a complete force versus deflection history of each test. The force and deflection at fracture were obtained from the data of the machine.[53]

The average values of flexural strength of heat polymerizing acrylic resins are near to 78-92 Mpa.[5] Therefore, ultimate goal of this study was to evaluate and compare the flexural strength of heat polymerized acrylic resin denture base material reinforced with 3% silanized titanium dioxide nanoparticles and 1% silanized aluminium oxide nanoparticles. The results showed that the mean flexural strength of **Group A (107.58MPa)** and **Group T (105.72 MPa)** was greater than **Group C (89.18 MPa).** This study shows that flexural strength can be increased by reinforcement with 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles. The reason for increase in flexural strength with addition of 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles was attributed to proper distribution of particles within the denture base powder and these act as potential fillers in the resin matrix. But, there was no statistical difference between the reinforced PMMA with 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles.

CLINICAL IMPLICATION

When the entire spectrum of this study is analyzed, it becomes evident that the heat polymerized acrylic dentures reinforced with silanized aluminium oxide nanoparticles and silanized titanium dioxide nanoparticles increases the flexural strength of the denture base material and thus, reduces the probability of occurrence of fracture. It also increases the thermal diffusivity of the denture base material, which enhances the patient's perception to hot and cold, hence improving the adaptability of the patient to the denture. This in turn, aids in better comfort and satisfaction with the prosthesis in place. In addition to this, it imparts radio-opacity to the material so that any fractured remnants can be detected radiographically.

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, hardness, 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.

Summary

Don't fear failure so much that you refuse to try new things. The saddest summary of a life contains three descriptions: could have, might have, and should have.

The heat cure denture base resins are extensively used for their excellent properties such as ease of handling, polishing and aesthetics. However, the mechanical strength is not sufficient to maintain the longevity of the denture. The fracture of acrylic resin denture is a common occurrence.

This study was conducted to evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles with that of conventional heat polymerized polymethyl methacrylate

denture base material. The specimens tested were fabricated according to ADA specification no. 12 with 30 specimens in each group.

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

Results of the study shows that the mean flexural strength of Group A was maximum which is **107.59MPa.** For **Group T**, the mean strength was **105.73 Mpa, and** for **Group C** the mean strength was **89.18 Mpa.** Statistical analysis shows highly significant difference in the means of Group C and Group A ($p < 0.000001$). Also, the difference in the means of Group C and Group T was statistically significant ($p < 0.000001$). However, the difference between Group A and Group T was statistically insignificant as revealed by p-value of **0.647** ($p > 0.05$).

Thus, the study results concluded that reinforcement of acrylic resin with 1% silanized aluminium oxide nanoparticles shows a significant increase in the flexural strength when compared with the unmodified acrylic resin and there was no statistical difference between reinforced PMMA with 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles.

Conclusion

The studio is a laboratory, not a factory. An exhibition is the result of your experiments, but the process is never-ending. So, an exhibition is not a conclusion.

Within the limitations of this study following conclusions were drawn:

1. Specimens with reinforcement increase the flexural strength.
2. Reinforcement with 1% silanized aluminium oxide nanoparticles showed highly significant increase in flexural strength.
3. There was no statistical significance between the heat polymerized acrylic resin denture base specimens reinforced with 1% silanized aluminium oxide nanoparticles (Group A) and 3% silanized titanium dioxide nanoparticles (Group T).

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TABLES

Table 1: Descriptive statistics for Flexural Strength

GROUPS	N	Mean	Median	SD	Range
Group C (Control)	30	89.18	87.625	12.058	[60.43, 112.61]
Group A (Al2O3)	30	107.59	106.155	5.716	[100.76, 120.21]
Group T (TiO2)	30	105.73	104.98	4.289	[99.90, 112.82]

Table II

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

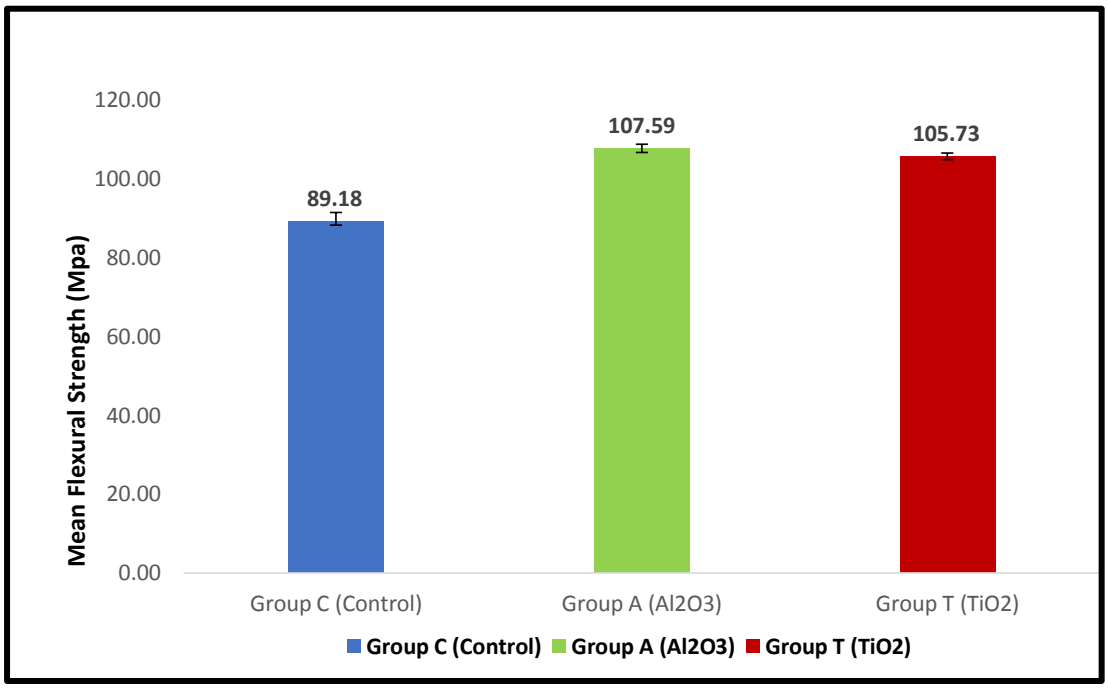
Source	Degrees of freedom	Sum Squares	Mean Square	F-value	P-value
Between Groups	2	6162.081	3081.041	47.049	**0.000 (1.4123E-14)**
Within Groups	87	5697.317	65.486		

Table III

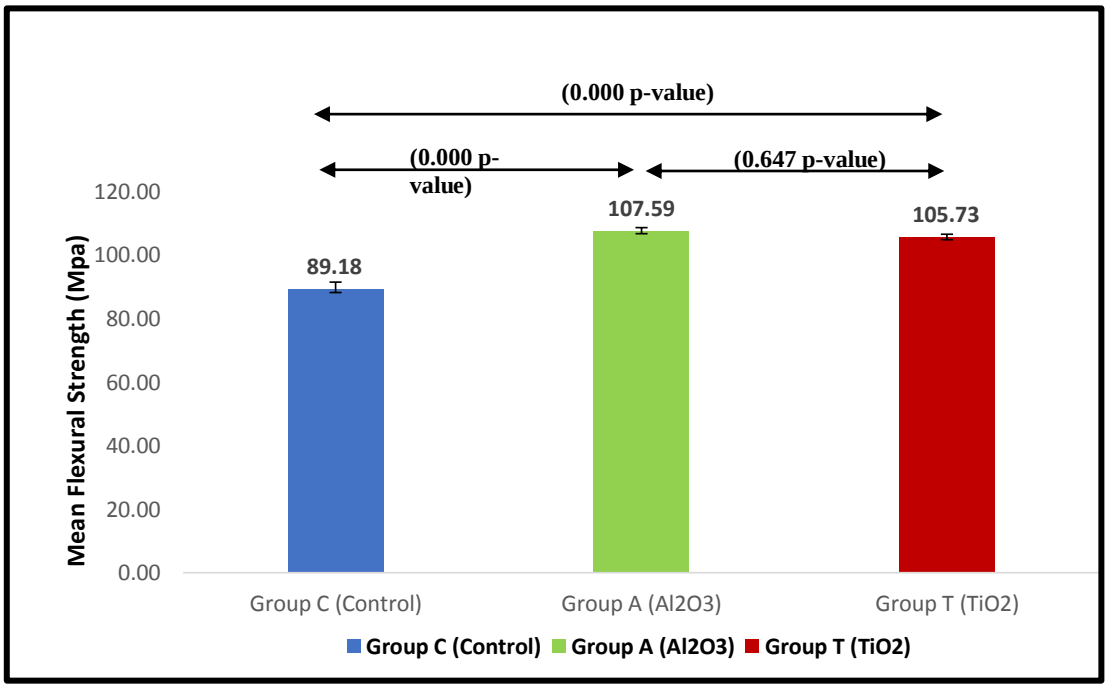
Table 3: Pair-wise comparison of flexural strength between groups using Tukey's post-hoc test

Comparison	P-values
Control vs Group A	0.000 (5.1008E-9)
Control vs Group T	0.000 (5.1215E-9)
Group A vs Group T	0.647

GRAPHS



Graph 1: Bar chart with error bars showing means flexural strength according to the study groups



Graph 2: Comparison of mean flexural strength between groups

ANNEXURE

GROUP C: CONTROL (Heat polymerized polymethyl methacrylate without reinforcement)								
Sr. No.	**Sample No.**	**Flexural Load (N)**	**Flexural Strength (MPa)**		**Sr. No.**	**Sample No.**	**Flexural Load (N)**	**Flexural Strength (MPa)**
1	No.1	123.87	103.22		**16**	No.16	102.50	85.42
2	No.2	112.89	94.08		**17**	No.17	105.44	87.87
3	No.3	123.77	103.14		**18**	No.18	101.92	84.93
4	No.4	84.96	70.80		**19**	No.19	106.13	88.44
5	No.5	92.80	77.33		**20**	No.20	98.88	82.40
6	No.6	126.02	105.02		**21**	No.21	125.93	104.94
7	No.7	116.71	97.26		**22**	No.22	128.77	107.31
8	No.8	111.52	92.93		**23**	No.23	99.76	83.13
9	No.9	121.12	100.94		**24**	No.24	91.23	76.03
10	No.10	72.52	60.43		**25**	No.25	95.64	79.70
11	No.11	114.46	95.38		**26**	No.26	108.38	90.32
12	No.12	97.51	81.25		**27**	No.27	87.02	72.52
13	No.13	102.50	85.42		**28**	No.28	104.86	87.38
14	No.14	102.80	85.66		**29**	No.29	98.78	82.32
15	No.15	135.14	112.61		**30**	No.30	116.62	97.18
							Average	**89.18**

GROUP A: Al_2O_3 (Heat polymerized polymethyl methacrylate reinforced with 1% silanized aluminium oxide nanoparticles)								
Sr. No.	**Sample No.**	**Flexural Load (N)**	**Flexural Strength (MPa)**		**Sr. No.**	**Sample No.**	**Flexural Load (N)**	**Flexural Strength (MPa)**
1	No.1	134.55	112.12		**16**	No.16	130.14	108.45
2	No.2	122.50	102.08		**17**	No.17	131.29	109.40
3	No.3	125.36	104.46		**18**	No.18	139.65	116.37
4	No.4	121.71	101.43		**19**	No.19	128.24	106.86
5	No.5	134.17	111.80		**20**	No.20	125.23	104.35
6	No.6	144.25	120.21		**21**	No.21	121.37	101.14
7	No.7	123.23	102.69		**22**	No.22	133.09	110.90
8	No.8	123.38	102.81		**23**	No.23	123.48	102.90
9	No.9	144.25	120.21		**24**	No.24	130.14	108.45
10	No.10	135.44	112.86		**25**	No.25	121.71	101.43
11	No.11	123.38	102.81		**26**	No.26	135.65	113.04
12	No.12	133.35	111.25		**27**	No.27	126.54	105.45
13	No.13	134.35	111.96		**28**	No.28	120.92	100.76
14	No.14	135.39	112.82		**29**	No.29	124.40	103.66
15	No.15	121.91	101.59		**30**	No.30	124.07	103.39
							Average	**107.58**

GROUP T: TiO_2 (Heat polymerized polymethyl methacrylate reinforced with 3% silanized titanium dioxide nanoparticles)								
Sr. No.	**Sample No.**	**Flexural Load (N)**	**Flexural Strength (MPa)**		**Sr. No.**	**Sample No.**	**Flexural Load (N)**	**Flexural Strength (MPa)**
1	No.1	122.79	102.32		**16**	No.16	121.37	101.14
2	No.2	133.22	111.01		**17**	No.17	125.93	104.94
3	No.3	135.31	112.75		**18**	No.18	125.00	104.16
4	No.4	120.80	100.66		**19**	No.19	128.77	107.31
5	No.5	119.89	99.90		**20**	No.20	123.87	103.22
6	No.6	127.51	106.25		**21**	No.21	123.77	103.14
7	No.7	121.60	101.33		**22**	No.22	126.02	105.02
8	No.8	125.46	104.55		**23**	No.23	121.16	100.96
9	No.9	133.60	111.33		**24**	No.24	135.14	112.61
10	No.10	128.05	106.70		**25**	No.25	128.97	107.47
11	No.11	135.39	112.82		**26**	No.26	126.02	105.02
12	No.12	121.37	101.14		**27**	No.27	127.30	106.08
13	No.13	123.38	102.81		**28**	No.28	121.12	100.94
14	No.14	134.55	112.12		**29**	No.29	135.14	112.61
15	No.15	122.50	102.08		**30**	No.30	131.29	109.40
							Average	**105.72**