COMPARATIVE EVALUATION OF IMPACT STRENGTH OF HEAT POLYMERIZED POLYMETHYL METHACRYLATE DENTURE BASE MATERIAL REINFORCED WITH NON-SILANIZED AND SILANIZED ZIRCONIUM OXIDE NANOPARTICLES – AN IN-VITRO STUDY

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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	Kj/m^2	Kilojoule/square meter
7	^0C	Degree Celsius
8	^{0}	Degree
9	mm	Millimetre
10	PMMA	Polymethyl methacrylate
11	µ	Micron
12	ZrO_2	Zirconium oxide
13	Rpm	Rotations per minute
14	mins	Minutes
15	gms	Grams

INTRODUCTION

Polymethyl methacrylate (PMMA) is one of the most widely used materials in Prosthodontics since its introduction in **1937** by **Walter Wright**.[1] It is used primarily as a denture base material, attributed to various desirable qualities such as excellent aesthetic property, ease of handling and repair, accurate reproduction of surface details, lack of toxicity and cost effectiveness.[2]

Through the ages, loss of teeth due to trauma, disease or any other cause has distressed mankind. The means of replacing missing teeth structures by artificial materials continues to account for a large part of the application of material science.[3] It was believed that in 2500 BC Egyptians constructed the first dental prosthesis. In around 700 BC dentures surfaced as a mode of treatment for replacing missing teeth.[2] During the 18th century, gold and porcelain were the material of choice for denture base materials. **Etienne Bourdet (1775**) made the first reference to the use of a gold base punctuated with small holes much like the sockets of teeth. In **1774 Alexis Duchateau,** a Parisian apothecary, dissatisfied with his own stained hippopotamus ivory denture, was inspired as an attempt the use of porcelain for denture fabrication.[4]

During the 19th century, Tortoise Shell, Gutta Percha, Vulcanite, Aluminum and Celluloid were used as denture base materials. Vulcanite remained the principal denture base material for the next 75 years. In the 20th century various materials like Bakelite, Stainless steel, Cobalt-chromium, Vinyl Resin, Acrylic Resin, Polystyrene and Nylon were utilized for fabrication of dentures.[2]

Dr. Walter Wright (1937), introduced polymethyl methacrylate (PMMA) which is one of the most widely used non-metallic material till date in Prosthodontics.[1] However, it also has relative inadequacies like residual monomer allergy, poor mechanical strength, low impact strength, brittle on fatigue, poor conductors of heat, low hardness, porosity, crazing, warpage, poor adhesion to metal and porcelain that can cause the denture base to fracture and affect its longevity.[5]

After years of use, dentures will deteriorate and the bases may fracture due to constant exposure to external stresses caused by poor occlusion or fit of dentures.[4, 5] Dentures may fail inside the mouth during function which is fatigue phenomenon and denture failure may also occur extra-orally when the dentures are dropped on hard surfaces which occurs due to impact force.

Several attempts have been made to improve the physical properties of the denture base material, such as the chemical modification of PMMA, development of an alternative material to PMMA, reinforcement with other materials, such as fibers, and use of macro and nano fillers.[6, 7]

There are many types of fibers that can be used as reinforcement for denture base resinssuch as; polyethylene fibers, aramid fibers and carbon fibers but the carbon has an adversely effect on denture aesthetic because of its black color, while the Kevlar fibers are also used to reinforce and strengthen the PMMA; but they are causing problems in aesthetics and difficulty in polishing.

Recently, studies have investigated the effect of incorporating inorganic nanoparticles into PMMA to improve its properties. It was found that the physical properties of acrylic resins are influenced by the addition of nanoparticles as the reinforcing agent. The shape and size as well as the concentration and interaction of these nanoparticles with a polymer matrix determines the properties of a polymer Nano-composite mixture. Many reports proved the dependence of acrylic resin properties on nanoparticle concentrations. Nanomaterials are known for their superior characteristics compared to the conventional ones.[8]

Zirconia is characterized by high fracture toughness and flexural strength as a result of a physical property known as transformation toughening. The biocompatibility of zirconia is also extensively studied.[9]

Tetragonal phase is found at temperature 2370°C and it is a catalytic phase and it is a mechanically interesting phase compared with the other phases. Tetragonal phase has positive mechanical properties like high strength and toughness, to stabilize this tetragonal yttria is added. Yttria (YSTZ) is the most widely used and studied stabilizer.[10]

Silanes have the ability to bond inorganic materials such as metal and metal oxides to organic resins resulting in improved mixing, better bonding and increased matrix strength.[11]

In general, dentures are subjected to a combination of tensile, compressive, and shear forces. Flexural fatigue occurs after repeated flexing of a material and is a mode of fracture whereby a structure eventually fails after being repeatedly subjected to loads that are so small that one application apparently does nothing detrimental to the component.

Impact failure is one of the most common cause of denture failure and this occurs dueto sudden dropping of the prosthesis and as a result fracture of the denture base occurs.[12, 13]

A survey reported that 63% of the dentures had broken within 3 years of their delivery. Fractures in dentures result from two different types of forces, namely flexural fatigue and impact. Another study showed that 68% of acrylic resin dentures break due to impact fracture within few years after fabrication.[14]

Any denture base material should have sufficient strength to withstand fracture in service. Many attempts have been made to enhance the strength of heat polymerized acrylic resin denture base material. Recently incorporation of zirconium oxide to PMMA have been successfully used to improve the mechanical properties of

PMMA.[15]

As limited amount of data is available in literature regarding effect of incorporation of silanized tetragonal zirconium oxide nanoparticles on impact strength of PMMA, it needs to be verified by scientific studies.

Therefore, the purpose of this study was to evaluate and compare the impact strength of heat polymerized polymethyl methacrylate denture base material reinforced with non-silanized tetragonal zirconium oxide nanoparticles and silanized tetragonal zirconium oxide nanoparticles.

AIMS AND OBJECTIVES

Aim of the study

To evaluate and compare the impact strength of heat polymerized polymethyl methacrylate denture base material reinforced with non-silanized tetragonal zirconium oxide nanoparticles and silanized tetragonal zirconium oxide nanoparticles.

Objective of the study

1. To evaluate the impact strength of heat polymerized polymethyl methacrylate without reinforcement.

2. To evaluate the impact strength of heat polymerized polymethy methacrylate denture base material reinforced with non-silanized tetragonal zirconium oxide nanoparticles.

3. To evaluate the impact strength of heat polymerized polymethy methacrylate denture base material reinforced with silanized tetragonal zirconium oxide nanoparticles.

4. To compare the impact strength of heat polymerized polymethy methacrylate denture base material reinforced with non-silanized tetragonal zirconium oxide nanoparticles, silanized tetragonal zirconium oxide nanoparticles and heat polymerized polymethy methacrylate denture base material without reinforcement.

REVIEW OF LITERATURE

Smith DC (1961)[6] 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 acts 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 A and Greener EH (1967)[16] studied the effect of Whisker reinforcement of polymethyl methacrylate denture base resins. They used Al_2O_3 whiskers of diameters between 0.2 and 10 microns, sapphire whiskers 1-l0 microns in diameter and 75-125 microns in length, stainless steel compacted fibers and low-density fibers, boron nitride filaments and silicon carbide whiskers. The study concluded that Al_2O_3 whiskers when added in concentrations in the order of 10-13 per cent are most effective in reinforcement. For stainless steel fibers, boron nitride filaments and silicon carbide whiskers, no dramatic changes were registered in any of the physical properties and for sapphire whiskers, the ultimate bending strength approximately doubled and 25 per cent changes in the modulus and resilience were noted. Thus, the evidence presented in their study strongly indicated that an enhancement of flexural strength of denture base polymethyl methacrylate is possible through the technique of whisker reinforcement with sapphire fibers.

Hargreaves AS (1969)[7] studied the prevalence of fractured dentures. She conducted a survey at Dundee Dental Hospital for 6 months and stated that during that period, there were 113 denture repairs. 68 per cent denture fracturedat the end of three years after provision, there being a greater proportion of partial dentures than complete dentures. 40 per cent dentures fractured during mastication, there being twice as many complete dentures as partial dentures. Women tended to have fractured the denture during eating. She found out that habits such as pipe smoking, nail biting or pencil chewing did not play a significant part in the general pattern of fractures. Her 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.

Schreiber CK (1971)[17] evaluated the effect of the use of carbon fibers to reinforce polymethyl methacrylate. Specimens were made containing untreated carbon fibers, untreated chopped carbon fibers, surface treated carbon fibers and the control group. Acrylic resin composites were made by wetting the bundles of fibers with monomer and then incorporating them into the polymer forming a thin sheet within the matrix. The results showed that surface treated carbon fibers showed the greatest transverse strength that exceeded plain acrylic by 50% however, untreated carbon fibers had just the opposite effect.

Berry HH and Funk OJ (1971)[18] said that midline fracture of the denture is common. Breakage is due to: (1) Difficulty in cleaning, (2) Coughing which pushes the denture out of the mouth, (3) Lack of denture base material at the midline, (4) Greater than average biting force, (5) Dropping the denture accidentally. Breakage is prevalent among neuropsychiatric patients, especially those having neuromuscular disorders such as Huntington's chorea, hemi paralysis, muscular dystrophy, and Parkinson's disease. They incorporated vitallium in acrylic resin denture base and concluded that the denture strengtheners used in this study were designed to retain all the qualities of the acrylic resin denture in addition to adding the needed strength to prevent lowerdenture breakage.

Beyli MS, Dent M and Fraunhofer JA (1981)[19] analyzed the causes of fracture of acrylic resin dentures. The ratio of upper to lower denture fractures was about 2:1, with the most common causes of fracture appearing 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. They suggested use of higher strength polymers, notably impact- resistant materials, constructing dentures with metal palates for patients with heavy occlusions, palatal relief in the anterior portion of the palate and reinforcement in the anterior part of the palate as a means to reduce fractures.

Carroll CE and Fraunhofer JA (1984)[20] conducted a study to determine the effect of the use of commonly available materials i.e., brass wire and stainless-steel wire to reinforce 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. The reinforcing wires and braid were located accurately 0.46 mm from the bottom of the specimens by resting them on l8 gauge wires placed in the wells of the mold before they were filled with acrylic resin. They concluded that when stainless steel wire was used, greater transverse strength was obtained with the use of wires of larger dimension. The increase in strength obtained with the braid was shown to be statistically significant, but it was questionable if that increase (17%) would prove to beclinically significant.

Yazdanie N and Mahood M (1985)[21] investigated the transverse strength of acrylic resin reinforced with varying amounts of carbon fiber in two different lay-ups. The fibers used were strands and woven mat. Both had been silane coated to improve their bonding to acrylic resin. The strands were in the form of long filaments, woven mat was cut according to the size of the test pieces. The orientation of strands was along the long axis of the specimens, whereas the fibers of the mats were parallel with and at right angles to the long axis andsandwiched between thin sheets of acrylic resin dough. They concluded that carbon fiber acrylic resin composites are stronger and stiffer than unfilled acrylic resin and strands are more efficient strengtheners than woven mats.

D.L. Gutteridge et al (1988)[22] conducted a study to know the effect of including 0.5%, 1%, 2%, 3% and 4% by wt. of 6 mm lengths of ultra-high modulus polyethylene fibers on the impact strength of acrylic denture base resin. He concluded that the inclusion of 1% by wt. of 6 mm length of ultra- high modulus polyethylene fibers provided an effective means of reinforcing acrylic resin with respect to impact strength.

Berrong JM, Weed RM and Young JM (1990)[23] studied the fracture resistance of polymethyl methacrylate denture base (PMMA) resin reinforced with Kevlar fibers. Kevlar, a synthetic aramid polymer fiber approximately 15 µm in diameter, was cut in 31-mm-long sections from a single continuous roll of bundled fibers and subdivided into groups representing ratios of 0.5%, 1% and 2% by weight of the PMMA resin specimens. After initial trial packing, each specimen was removed from the mold and then cut in two approximately equal portions. A bundle of Kevlar fibers representing one of the predetermined weight ratios (0.5%, 1 % and 2%) was then placed into the mold between the two layers of the resin. They concluded that the layering of Kevlar reinforcing fibers in the center of PMMA heat-processed denture resin significantly increased the fracture resistance of the resin. The use of up to 2%by weight of Kevlar reinforcement fibers in this manner may increase fracture resistance of the resin denture base.

Solnit GS (1991)[24] studied the effect of methyl methacrylate reinforcement with silane-treated and untreated glass fibers. Cloth form glass fibers, loose form yellow glass fibers and loose form white glass fibers were used. They were soaked in a silane coupling agent for 5 minutes and air dried before incorporation. This study suggests that glass fibers can be pretreated with a silane coupling agent to obtain a chemical bond between the fibers and the acrylic resin. Samples with untreated fibers tested weaker than samples without fibers. Samples with silane-treated tested stronger but the difference in strength was not statistically significant.

Ladizesky NH and Chow TW (1992)[25] studied the reinforcement of complete denture bases with continuous highly drawn linear polyethylene (HDLPE) fibers. Maxillary bases were reinforced with four layers of parallel fibers, with a modification of the preimpregnated "pre-preg" technique. Mandibular base was reinforced with pre-preg parallel fibers at right angles to the mandibular arch. It was found that the incorporation of approximately 48% volume of parallel fibers in acrylic resin bars produced increases of approximately 70% for flexural strength, 600 % for stiffness, and 1000 % for impact strength.

Vallittu PK and Lassila VP(1992)[26] evaluated the effect of different metal and fiber strengtheners on the fracture resistance of polymethylmethacrylate. Remanium's spring hard clasp wire, semi-circular wire, braided wire plate and stainless-steel mesh were used as metal strengtheners in addition to aramid and carbon fibers. The fibers were divided into: silanized and untreated and the metal strengtheners were divided into: glossy and sandblasted. They concluded that all of the metal wire strengtheners increased the fracture resistance of the specimens except the stainless-steel mesh which did not have very good strengthening properties and also the differences between sandblasted and glossy wires were not significant. All the fiber strengtheners reinforced the test specimens, with the exception of the unsilanized glass fiber, which slightly decreased the resistance.

Vallittu PK (1993)[27] studied the effect of two different silane compounds on the adhesion between the different fibers and acrylic resin. The silane compounds tested were A174 and AP133 and the different fibers used were glass, carbon and aramid fibers. The amount of fibers in a weight percentage of the mass of the acrylic resin was as follows: 7.39% for glass fibers, 2.08% for carbon fibers and 2.30% for aramid fibers. The results showed that the glass fibers and the aramid fibers when treated with A174 silane compound increasedthe fracture resistance of the specimens.

N.H. Ladizesky, Y.Y. Cheng, T. W. Chow and I.M. Ward et al (1993)[28] evaluated 3 mechanical properties i.e., flexural strength, flexural modulus and impact strength of acrylic resins reinforced with woven highly drawn linear polyethylene fibers (HDLPE) and concluded that the incorporation of three layers of woven HDLPE fibers substantially increases the impact strength of acrylic denture base resins.

N.H. Ladizesky, Y.Y. Cheng, T. W. Chow and I.M. Ward et al (1993)[29] incorporated over 30% by volume of chopped high performance polyethylene fibers into acrylic denture base resin. The reinforcement produced a substantial improvement in several important properties, namely (1) stiffness and impact strength were higher; (2) mechanical properties were insensitive to notches that mimic anatomical feature and (3) samples damaged during bending and impact did not break up into separate fragments.

Vallittu PK (1993)[30] investigated the effect of metal wire bonding to acrylic resin on the fracture resistance of an acrylic resin denture base material construction. He used metal wires which were semicircle wires 1.0 x 2.0 mm. These were sandblasted with 250 µm grain size aluminum oxide (Al_2O_3) under the air pressure of 5.5 bar and then bonded to the acrylic resin. Two different bonding methods were used: Silicoating and Eudicolle. The fracture resistances of the test specimens reinforced with sandblasted metal wires was higher than the resistance of the unreinforced specimens. The bonding compound of Eudicolle did not increase the fracture resistance compared with unsilanised strengtheners whereas the bonding method of Silicoater increased the resistance significantly.

P. K. Vallittu, H. Vojtkova and V. P. Lassila et al (1995)[31] compared the impact strength of heat-cured acrylic resin specimens reinforced with metal wire and continuous E-glass fibers. It was found that both types of reinforcement 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.

Vallittu PK (1995)[32] reviewed methods used to reinforce polymethyl methacrylate resin. He stated that Metal wires of various dimensions included within the resin increased the transverse strength from +5% to +85%, whereas metal mesh showed little effect (0% to +25%). The position of a non-chemically bonded strengthener relative to the loading force does not affect the strength of the PMMA-metal composite.

Strengtheners with macroscopic retention showed a slight increase in strength, and strengtheners with microscopic retention showed a clear increase in strength. The chemical bond between the resin and metal strengthener enhanced the strength of the PMMA metal composite when the metal was placed on the tension side of the specimen.

Jagger D.C et al (1999)[33] stated that fractures in dentures occur due to impact force and flexural fatigue. There are continuous efforts to improve the strength, stiffness, dimensional stability and to achieve radio-opacity by incorporation of different fillers.

Uzun G, Hersek N and Tincer T (1999)[34] measured the effect of 5 fiber strengtheners on the fracture resistance of denture base acrylic resin material. Impact strength, transverse strength, deflection and elastic modulus values of a heat-polymerized denture base resin (Trevalon) reinforced with glass, carbon, thin kevlar, thick kevlar, and polyethylene fibers in woven form were studied. Polyethylene and glass-reinforced acrylic resin specimens were significantly more resistant to impact strength. Fiber reinforcement had no significant effect on the transverse strength. Carbon, thick Kevlar, and polyethylene-reinforced specimens showed significantly higher elastic modulus values.

Kanie T, Fujii K, Arikawa H and Inoue K (2000)[35] investigated the reinforcing effect of woven glass fibers on deflection, flexural strength, flexural modulus and impact strength of acrylic denture base polymer. Three silanized and unsilanized woven glass fibers were used. Specimens were made by heating the denture cure resin dough containing glass fibers, which were sheathed in the dough. Specimens with four different thicknesses and of five different types were made, incorporating the glass fiber. When specimens contained unsilanized glass fiber, the flexural strength in specimens of 1- and 2-mm thickness and the impact strength in specimens of 2 mm thickness were higher than those of specimens without glass fiber. On the contrary, the flexural strength and deflection in specimens reinforced with silanized glass fiber of 1 mm thickness were significantly higher than those of unreinforced specimens. Further, the impact strength in specimens reinforced with silanized glass fiber of 2 mm thickness was significantly higher than that of unreinforced specimens. Statistically significant differences were found in the flexural and impact strength when specimens of 4 mm thickness were reinforced with two or three unsilanized glass fibers.

Chen SY, Liang WM and Yen PS (2001)[36] studied the mechanical properties of acrylic resin reinforced with three types of fibers. Polyester fiber, Kevlar fiber, and glass fiber were cut into 2-, 4-, and 6-mm lengths and incorporated at concentrations of 1, 2, and 3% (w/w). The results showed that the impact strength enhanced with fiber length and concentration, particularly Polyester fiber at 3% and 6-mm length. They concluded that Polyester fiber and Kevlar fiber both improved mechanical properties, but the polyester fiber was superior in aesthetics and so it offers to be the best formulation for acrylic denture base resin reinforcement.

Matinlinna JP et al (2004)[37] reviewed silanes in prosthetic and restorative dentistry stating that the most commonly applied silane in dental laboratories is a monofunctional methacryloxypropyltrimethoxysilane (for 3- trimethoxysilyl propyltrimethoxysilane [TMPS]).

Franklin P, Wood DJ and Bubb NL (2005)[38] evaluated the effect of adding glass flake to denture base acrylic powder on the fracture toughness of the set material. Glass flake was added in 5, 10 or 20% w/w to Trevalon denture base powder. The addition of glass flake gave up to a 69% increase in fracture toughness compared to plain Trevalon material. The addition of 5% glass flake lead to an improvement in fracture toughness compared to both plain Trevalon and the 10 and 20% groups. They concluded that improvement in fracture toughness of a denture base acrylic material using glass flake is an extremely promising result but other mechanical properties would require testing before glass flake can be recommended as a reinforcing material.

Vojdani M and Khaledi AAR(2006)[39] conducted a study to measure the transverse strength of a heat polymerized acrylic resin after reinforcement with metal wire and two types of glass fibers. Heat-cured acrylic resin was reinforced with sandblasted metal wires, woven (Stick Net) and continuous unidirectional (Stick) fibers. They concluded that the transverse strength of heat-polymerized denture base resin was enhanced considerably by using metal wire and glass fiber reinforcements. However, the addition of unidirectional glass fibers was significantly more effective method to improve flexural strength of denture base acrylic resin.

Goyal S (2006)[40] reviewed the use of silanes in dentistry. They said that one reactive group of the silane (e.g., methoxy, ethoxy and silanolic hydroxy groups) reacts with various inorganic materials such as glass, metals, silica, sand to form a chemical bond with the surface of the inorganic material, while the other of the reactive groups (e.g., vinyl, epoxy, methacryl, amino and mercapto groups) is reactive with various kinds of organic materials or synthetic resins to form a chemical bond. As a result of possessing these two types of reactive groups, silanes are capable of providing chemical bonding between an organic material and an inorganic material. This unique property of silanes is utilized for the surface treatment of glass fiber products, performance improvement of fiber- reinforced plastics by the direct admixture to the synthetic resin, improvement of paints and other coating materials, and adhesives, modification of surface properties of inorganic fillers, surface priming of various substrate materials. When used as a coupling agent, silanes bind organic polymers to mineral or siliceous fillers, resulting in improved mixing, better bonding of pigment or fillers to resins, increased matrix strength, decreased water intake of composite and minimize wear. They stressed that silanization of E-glass and aramid fibers enhances the adhesion between the fibers and organic acrylic resin system in a denture material and long-range intraoral stain protection has been accomplished in a denture, when its surface was modified with a fluoro-carbon chain containing silane.

Tacir IH, JD Kama, M Zortuk, S Eskimez et al (2006)[41] compared the fracture resistance of unreinforced and glass fibre reinforced acrylic resin polymers prepared under both conventional heat curing and microwave curing techniques. They concluded that fracture resistance and impact strength of heatpolymerized acrylic resin improved with glass fibre reinforcement. It may be possible to apply these results to distal extension partial and complete denture bases.

Dagar S et al(2008)[42] compared the resistance to fracture properties of commercially available heat polymerizing PMMA denture base resin with those of the same material reinforced by glass and nylon fibers. On comparing the impact and flexural strength properties between conventional and fibre reinforced, it was found that fibre reinforced specimens were more resistant toimpact and flexural fatigue than conventional PMMA specimens and glass fibre reinforcement improved both impact and flexural strength denture base resin when compared with nylon fibers.

Ayad NM, Badawi MF and Fatah AA (2008)[43] investigated the effect of reinforcing high-impact acrylic resin (Metrocryl HI) with zirconia powder in two different concentrations on the transverse strength, impact strength, surface hardness, water sorption and solubility 5% and 15% zirconia- modified Metrocryl HI resin samples were prepared. They concluded that the addition of zirconia resulted in a highly significant increase in transverse strength of high-impact acrylic resin and this increase was in proportional to the concentration of zirconia. The impact strength, surface hardness as well as water solubility of the zirconia-reinforced high-impact resin were not significantly different from that of zirconia-free high-impact resin.

Elshereksi NW et al (2009)[44] investigated the effect of barium titanate ($BaTiO_3$) filler incorporation on the fracture toughness properties of denture base Poly(Methyl Methacrylate). The $BaTiO_3$ was treated by a silane coupling agent, 3-trimethoxysilylpropyl methacrylate (γ-MPS) before incorporating into PMMA. The samples were tested for fracture toughness before and after soaking for 28 days in simulated body fluid (SBF). They concluded that the dry samples provided higher fracture toughness than the wet samples.

Allareb OA and Ahmad ZA (2011)[45] evaluated the effect of 5wt% Al_2O_3/ZrO_2 reinforcement on the fracture toughness, flexural, and tensile properties of PMMA denture base. They concluded that the incorporation of Al_2O_3/ZrO_2 into PMMA improved the fracture toughness, tensile modulus and flexural properties of this denture base composite material.

Yadav P, Mittal R, Sood VK and Garg R (2012)[46] studied the effect of addition of metal filler particles on different strengths of polymethyl methacrylate

(PMMA) and to evaluate the thermal perception in vivo. The study was carried out in two parts. Part 1 of the study was an in vitro investigation regarding the effect of addition of metal fillers (aluminum and silver) in concentrations of 10%, 20% and 30%, by volume on the tensile, compressive, and flexural strength of PMMA. Part 2 of the study comprised the clinical evaluation of the thermal perception by 10 edentulous patients provided with two sets of complete dentures, one fabricated with unfilled PMMA and another with 20% aluminum 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. 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.

Ihab NS, Hassanen KA and Ali NA (2012)[8] assessed zirconium oxide nano-fillers incorporation and silanation on impact, tensile strength and color alteration of heat polymerized acrylic resin. The nanoparticles were silanated by coating with a layer of trimethoxysilypropylmethacrylate (TMSPM) and were added in a concentration of 3% and 5% by weight. Findings of the study showed that silanized ZrO_2 nano-filler was effective in improving impact strength while it was not effective in improving the tensile strength. The maximum increase in impact strength was observed in denture base nano composite containing 5% wt of silanated ZrO_2 nano-fillers.

Asar NV et al (2013)[47] studied the effect of various metal oxides on impact strength, fracture toughness of heat-cured acrylic resin. 1% TiO_2 and 1% ZrO_2, 2%

Al_2O_3, 2% TiO_2, and 2% ZrO_2 by volume were used as reinforcements. Thus, they concluded that 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, modification of heat- cured acrylic resins with certain amounts of metal oxides, especially with ZrO_2 may be useful in preventing denture fractures and undesirable physical changes resulting from oral fluids clinically.

Zhang XY et al (2014)[48] studied the hybrid effects of ZrO_2 nanoparticles (nano-ZrO_2) and aluminum borate whiskers (ABWs) on flexural strength andsurface hardness of denture base resin, polymethyl methacrylate (PMMA). ZrO_2 nanoparticles of an average granularity of 90 nm and an average surface area of 7±2 m^2/g and Aluminum borate whiskers of 5–30 μm length and a diameter of less than 1.5 nm, with a surface area of 2.0–2.5 m^2/g were used. Both nano-ZrO_2 and ABWs were modified by silane coupling agent i.e., Z- 6030 before being mixed with PMMA. They concluded that the flexural strength was maximum when 2 wt% of nano-ZrO_2 was mixed with ABWs at a ZrO_2/ABW ratio of 1:2, ß amounting to an increase of 52% when compared with pure PMMA.

Ahmed MA and Ebrahim MI (2014)[49] investigated the effect of Zirconium oxide (ZrO_2) nanofillers powder with 1.5%, 3%, 5% and 7% on the flexural strength, fracture toughness and hardness of heat-polymerized acrylic resin. Zirconium oxide powders with different concentrations (1.5%, 3%, 5% and 7%) were incorporated into heat-cure acrylic resin (PMMA) and processed with optimal condition of PMMA. PMMA specimen with 7% zirconium oxide nanofillers showed significantly highest mean flexural strength and hardness followed by PMMA specimen with 5% followed by specimen with 3% and then specimen with 1.5%. They concluded that the best mechanical properties were achieved by adding 7%wt ZrO_2 concentration.

Alnamel HA, Mudhaffer M (2014)[50] evaluated the effect of addition of surface treated silicon dioxide nano filler (SiO_2) on impact strength, transverse strength, and surface hardness of PMMA. In addition to controlled group, SiO_2 powder was added to PMMA powder by weight in three different percentages 3%, 5% and 7%, mixed by probe ultrasonication machine. A highly significant increase in impact strength and transverse strength was observed with the addition of SiO_2 powder at 3% and 5%; while a significant reduction occurred in both impact and transverse strength specimens tests at the percentage of 7% and a highly significant increase in surface hardness was observed at the percentage of 3%, 5% and 7%.

Alwan SA and Alameer SS (2015)[51] evaluated the effect of addition of 3% wt of treated (silanized) Titanium oxide Nano filler on physical and mechanical properties of heat cured acrylic denture base material. TMSPM silane coupling agent was used. Results showed that a highly significant increase in impact strength and transverse strength was observed with the addition of (TiO_2) nanoparticles to PMMA and a significant increase in surface hardness and surface roughness. The water sorption and solubility were significantly decreased when compared with the control group but there was an increase in surface roughness.

Asopa V et al (2015)[52] evaluated and compared the transverse strength of 10% and 20% zirconia (ZrO_2) reinforced high impact acrylic resin with that of high impact acrylic resin (Trevalon). A total of 30 specimens were fabricated.10 specimens fabricated from high impact acrylic resin; 10 specimens fabricated from 10% zirconia and 10 specimens fabricated from 20% zirconia reinforced high impact acrylic resin. Results revealed an increase in values of transverse strength when compared to the control group and higher transverse strength with the use of 10% zirconia when compared to 20%.

Gad M et al (2018)[53] in their study on Effect of zirconium oxide nanoparticles addition on the optical and tensile properties of polymethyl methacrylate denture base material, it was found that the addition of nano- ZrO2 increased the tensile strength of the denture base acrylic. The increase was directly proportional to the nano-ZrO2 concentration. The translucency of the PMMA was reduced as the nano-ZrO2 increased.

Somani MV et al (2019)[54] in their study on effect of incorporating various reinforcement materials on flexural and impact strength of polymethyl methacrylate, showed that maximum data available in literature on FS and IS of reinforced PMMA and converting all data to a common metric. Although a detailed mechanistic review was beyond the scope of this review, the following conclusions were drawn:

A. Fourteen reinforcement techniques out of 15 available data showed better results for IS of reinforced PMMA resin when compared to their respective control group

B. Eleven reinforcement techniques out of 25 available data showed better results for FS of reinforced PMMA resin when compared to their respective control group

C. Out of all the reinforcement materials included in the studies, zirconium dioxide, glass fibers, and titanium oxide showed increased values with respect to both IS and FS when compared to the unreinforced PMMA.

D. Hence, to increase the clinical life of PMMA, these reinforcement materials can be taken into consideration according to clinical requirement, patient's need, and clinician and laboratory personnel's skill.

MATERIALS AND METHODS

This in-vitro study was done to evaluate and compare the impact strength of heat polymerized acrylicresin denture base material reinforced with non-silanized and silanized zirconium oxide nanoparticles with that of conventional heat polymerized denture base material.

Material and methods are divided under the following heads:

1) Materials
2) Armamentarium and equipment
3) Method

MATERIALS

Sr. No.	Materials	Manufacturers	BATCH NO.
1.	Heat polymerized acrylic resin	DPI Heat CureTM, (Dentalproducts of India Ltd)	10183
2.	Die stone	Ultrarock; Kalabhai KarsonPvt Ltd, India	190603
3.	Silane coupling agent i.e., 3-methacryloxy propyl trimethoxy silane	Alpha Aesar (A JohnsonMatthey Company)	M0725
4.	Cold mold seal (separating medium)	DPI Heat CureTM, (Dentalproducts of India Ltd)	SL-63
5.	Zirconium oxide nanoparticles	Reinste (Nano Ventures)	DCZrOAK41
6.	Toluene	Emplura R	1811610317

ARMAMENTARIUM AND EQUIPMENTS

1. High accuracy balance (Fig 7)
2. Ultrasonicator (Fig 8)
3. Magnetic stirrer (Fig 9)
4. Vacuum rotary evaporator (Fig 10)
5. Acrylizer with thermostat (Fig 11)
6. Charpy's impact testing machine (Fig 12)
7. Rubber bowls and plaster spatula (Fig 13)
8. Sand paper (No. 120) (Fig 14)

9. Varsity flasks and clamps (Fig 13)
10. Camel hair brush (Fig 14)
11. Vernier caliper (Fig 15)
12. Glass Beaker (Fig 20)
13. Sterile Syringe (Fig 14)
14. Porcelain jar and Dapen dish (Fig 14)
15. Mixing spatula (Fig 13)
16. Petroleum jelly (Fig 14)
17. Para-film (Fig 17)
18. Brass metal dies (Fig 16)
19. Hydraulic bench press (Fig 18)
20. Distilled water (Fig 19)

METHODOLOGY:

The basic methodology consists of -

a) Die preparation.

b) Silanization of zirconium oxide nanoparticles.

c) Preparation of gypsum mold for fabrication of specimens.

d) Preparation of heat polymerized acrylic resin denture base specimens. (Group C)

e) Preparation of heat polymerized acrylic resin denture base specimens reinforced with non-silanized zirconium oxide nanoparticles. (Group

N)

f) Preparation of heat polymerized acrylic resin denture base specimens reinforced with silanized zirconium oxide nanoparticles. (Group S)

g) Testing of specimens for impact strength.

A total of 96 specimens were prepared with each group having 32 specimens. The specimens were divided under the following groups:-

Group C	The control group; heat polymerized acrylic resin (PMMA) without reinforcement. (n=32)
Group N	Heat polymerized acrylic resin (PMMA) reinforced with 3% (by weight) non-silanized zirconium oxide nanoparticles (n=32)
Group S	Heat polymerized acrylic resin (PMMA) reinforced with 3% (by weight) silanized zirconium oxide nanoparticles (n=32)

a. Die preparation:

Metal dies were fabricated to prepare molds for the fabrication of heat polymerized acrylic resin specimens. Three brass metal dies of dimension 80 mm in length, 10 mm in width, and 4 mm in height were fabricated. (ISO 179 standard)[55]

These fabricated metal dies had a threaded hole at the center. These holes were of 5 mm in diameter and 4 mm in depth. Screws were used to engage these threaded holes to facilitate easy removal of dies from the stone mold.

b. Preparation of gypsum mold for fabrication of specimens:

Gypsum molds were prepared with preformed brass metal dies. The threaded holes on the dies were blocked with carding wax before investing them. A thin layer of petroleum jelly was applied on three metal dies which was then invested in the lower half of the varsity flask. Investment material (die stone) was used for base flasking taking care to embed half the thickness of the metal die in it. 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 was set (1 hour), the flasks were opened and the carding wax within the holes was removed. The dies were then engaged with a screw and gently teased out.[56]

The molds formed were then immersed in hot water to remove any traces of petroleum jelly, wax and also to facilitate application of separating medium. These mold cavities thus obtained were used for the fabrication of heat polymerized polymethyl methacrylate denture base material specimens.

c. Silanization of zirconium oxide nanoparticles:

Zirconium oxide nanoparticles were added 4 gms to 100ml of pure toluene solvent in a glass beaker. The beaker containing toluene solvent and zirconium oxide nanoparticles was sonicated at room temperature for 20 mins. magnetic stirrer was placed in the beaker. This was then kept on a stirring machine and stirred at room temperature. 0.2 ml silane coupling agent i.e., 3- methacryloxypropyltrimethoxysilane (3% weight to nano Zr2O3) was added drop wise to it with the sterile syringe under rapid stirring. This was done for homogenous mixing of silane coupling agent with zirconium oxide nanoparticles and toluene solvent.

The beaker was covered by a parafilm and left standing for 2 days. The toluene solvent was removed by a rotary evaporator under vacuum at 60 C and rotation of 150 rpm for 30 mins. The silanated zirconium oxide nanoparticles were dried in a vacuum oven at 60 degree Celsius for 10 hours.

Preparation of heat polymerized acrylic resin denture base specimens (Control Group C):

32 samples were prepared using conventional heat polymerized denture base material (PMMA). As per manufacturer's recommendation, monomer and polymer were mixed in ratio of 1: 2.5 by weight. An electronic balance of high accuracy was used to weigh the materials. 7.5 gms of polymer powder and 3 ml of monomer were used for preparing 3 specimens. Packing was carried out at dough stage, following which trial closure was performed. Final closure was done under a hydraulic bench press at a pressure of 3000 psi for 3 mins (according to the manufacturer). The flask maintained for 1 hour. After completion of this short curing cycle, the flasks were removed from the water bath and allowed to bench cool at room temperature prior to deflasking.

The polymerized specimens were carefully removed. 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.

Preparation of heat polymerized acrylic resin denture base specimens reinforced with non-silanized zirconium oxide nanoparticles (Group

N):

32 samples were prepared using conventional heat polymerized denture base material (PMMA) reinforced with non-silanized zirconium oxide nanoparticles. For complete homogenous dispersion, non-silanized zirconium oxide nanoparticles were added to the monomer. As per group C, same proportion was followed for fabrication of specimens. 7.425 gm of polymer powder, 3 ml of monomer and 0.075 gm of non-silanized zirconium oxide nanoparticles were taken for fabrication of 3 specimens. An electronic balance of high accuracy was used to weigh the materials.

Non-silanized zirconium oxide nanoparticles were well dispersed in monomer by using an ultra-sonicator. Theultra-sonication will be done at 120 W, 60 KHz for 3 minutes. This allowed the homogenous dispersion of non-silanized zirconium oxide nanoparticles in the monomer.

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 acrylic resin denture base (Control group C). Specimens with defects were discarded. The finished specimens were stored in distilled water for 1 week at room temperature.

Preparation of heat polymerized acrylic resin denture base specimens reinforced with silanizedzirconium oxide nanoparticles (Group S):

32 samples were prepared using conventional heat polymerized denture base

material reinforced with silanized zirconium oxide nanoparticles. As per group N, same proportion and procedure was followed for fabrication of specimens reinforced with silanized zirconium oxide nanoparticles. The finished specimens were stored in distilled water for 1 week at room temperature.[56]

a) Testing of specimens

Testing of specimens were carried out at metallurgical laboratory. The specimens for each group were tested for impact strength. Impact strength Test was Conducted Following the Procedure Given by The ISO 179 **Charpy Type Impact Testing Machine**.[55]

The Specimen was Supported Horizontally and its ends & Struck by a Free-Swinging Pendulum that Releases from Fixed height in the Middle. The Charpy's impact strength of unnotched specimen was calculated in KJ/m^2 using the following formula :

$$\text{Impact strength} = E/BD\text{x}10^3$$

Where,

E = Impact energy

B = width of the specimen

D = Thickness of the specimen

PLATE I - MATERIALS

Fig 1: Heat polymerized acrylic resin

Fig 2: Die Stone

Fig 3: Tetragonal Zirconium oxide nanoparticles

Fig 4: Silane coupling agent

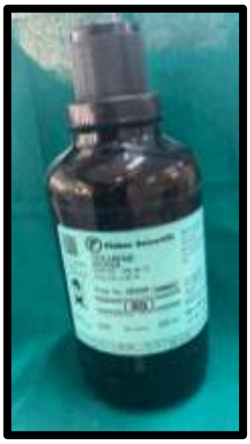

Fig 5: Toluene

Fig 6: Cold mold 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: Charpy's Impact testing machine

PLATE III

ARMAMENTARIUM AND EQUIPMENTS

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

Fig 14: SterileSyringe, Petroleum jelly, camel hair brush, Sand paper, Porcelain jar, Dapendish,

Fig 15: Vernier Caliper

Fig 16: Brass metal dies

Fig 17: Paraffin

Fig 18: Hydraulic bench press

Fig 19: Distilled water

Fig 20: Glass beaker

PLATE IV

METHODOLOGY

Fig 21: Preparation of gypsum mold to obtain specimens

Fig 22: Silanization process of zirconium oxide nanoparticles

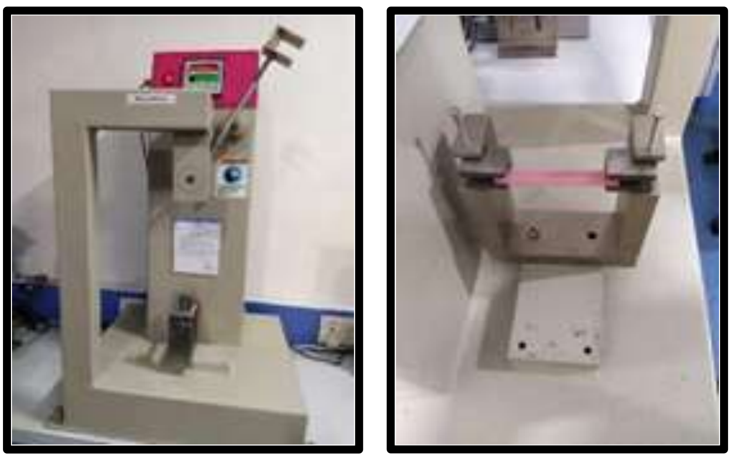

Fig 23: Testing of specimens

PLATE V

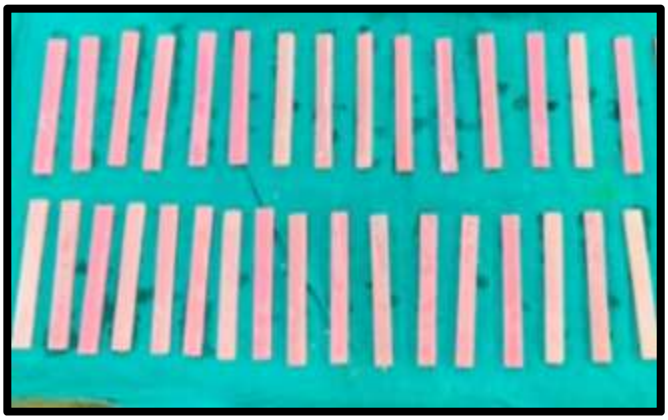

Fig 24: Group C- The control group; Heat polymerized polymethyl methacrylate denture base specimens without reinforcement before testing of impact strength.

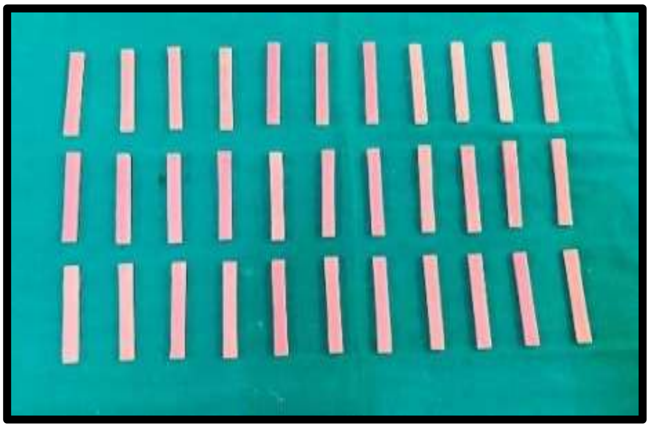

Fig 25: Group N- Heat polymerized polymethyl methacrylate denture base specimens reinforced with nonsilanized zirconium oxide nanoparticles before testing of impact strength.

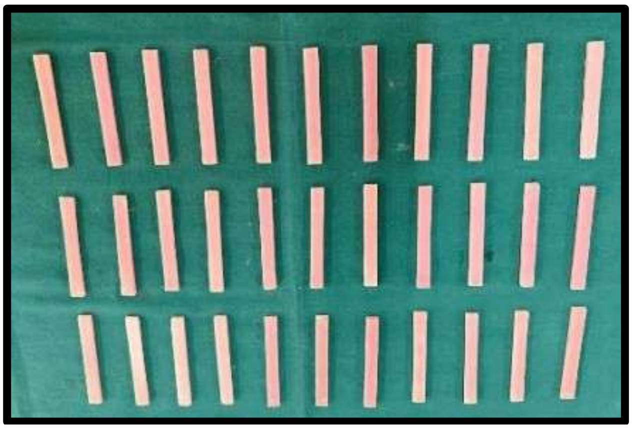

Fig 26: Group S- Heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium oxide nanoparticles before testing of impact strength.

PLATE VI

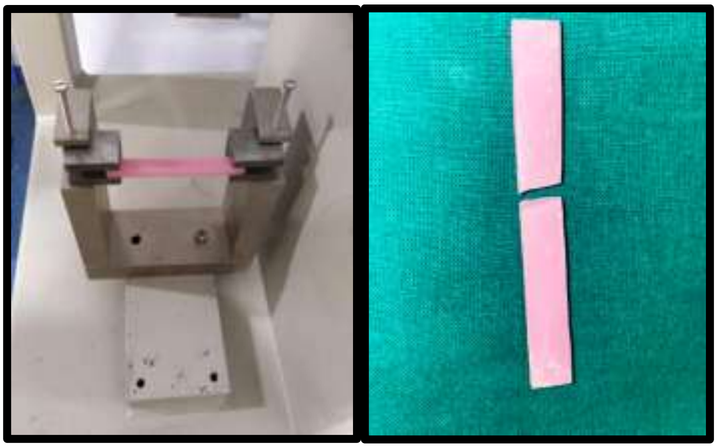

Fig. 27: Before and after testing of specimen

RESULTS

The mean values of impact strength were calculated along with using descriptive statistics amongst the following three groups:

Group S: Heat polymerized polymethyl methacrylate denture base material reinforced with silanized tetragonal zirconium oxide nanoparticles (3% by wt)

Group N: Heat polymerized polymethyl methacrylate denture base material reinforced with non-silanized tetragonal zirconium oxide nanoparticles (3% by wt)

Group C: Heat polymerized polymethyl methacrylate denture base material without reinforcement

The statistical analysis was done using the Statistical Package for the Social

Science (SPSS version 22, Armonk, NY: IBM Corp). The recorded values were statistically evaluated using the one-way analysis of variance test (ANOVA) for comparison amongst the three groups followed by Tukey post hoc test for multiple comparisons. The "p" values were considered significant at or below 0.05.

Table 1 depicts the distribution of mean values of the impact strength between the three groups. The values were highest for the Group S (17.91±3.96) followed by Group N (15.81±3.76). The impact strength was least when measured in the control group (12.74±2.81). This difference between each of the group was statistically highly significant (p=0.001) when analyzed by ANOVA as shown in table 2.

Table no. 3 depicts the multiple comparisons when subject to post hoc analysis. The impact strength was higher for Group S when compared to Group N. This difference was statistically significant (p=0.05).

The impact strength was higher for Group S when compared to Group C. This difference was statistically highly significant (p=0.001).

Similar trend was observed when the impact strength was compared between Group N and Group C. This difference was also statistically significant (p=0.03).

DISCUSSION

Poly (methyl methacrylate) [PMMA] material was used as denture base material for the first time in 1937 by Walter Wright, since that time. In 1940 PMMA became the most excellent denture base material [1].

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.[4] Since its introduction, polymethyl methacrylate is still the most predominantly used denture base material because of its excellent esthetics, being economical, easy for processing and repair. [7,8] 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.[9,57]

The denture base resin is subjected to various stresses during function, which include compressive, tensile, shear, and impact stresses.[11] A study by Johnston et al[58] observed that 68% of acrylic resin dentures fractured within the first three years after fabrication.

Smith (1961)[6] 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 takes place because of the fatigue and chemical degradation of denture base material.[9] Any factor that exacerbates deformation of the base or alters its stress distribution will predispose the denture to fracture.[11]

Nanomaterials are categorized according to dimension – those with all 3 dimensions less than100 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).[59] 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, silver, zirconia (ZrO_2), gold, platinum, silicon dioxide (SiO_2) is among the fillers that have been incorporated to enhance the mechanical properties of denture base resins. [60]

Asopa et al. [52] 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.

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.

Chandler H et al (1971)[61] suggested that one of the disadvantages 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. Sehjpal and Sood (1989)[11] 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[1] concluded that increase in the impact strength occurred with addition of 2-5wt% ZrO_2 nanoparticles due to good distribution of the particles, whereas increasing the percentage of modified nano-ZrO2 to 7wt%, due to agglomeration lowered the impact strength and transverse strength due of nanoZrO2. Hence, in this study, Zirconium oxide nanoparticles were treated with trimethoxysilylpropylmethacrylate (TMSPM) to improve adhesion of nanoparticles to resin matrix.[57]

In this study zirconium oxide nanoparticles were used in a concentration of 3% by wt. with silane coupling agent to obtain a chemical bond between zirconium oxide and acrylic resin powder. The impact strength was tested with Charpy's Impact testing machine to determine whether the addition of zirconium oxide had an effect on the impact strength on the PMMA denture base material.

Many materials have been used to reinforce the denture such as; metal in the form of wires, plate's, or powder but it has poor aesthetic and adhesion between metal and acrylic resin matrix. While the rubber toughening agents have been used as strengthen material but it's expensive[62]. Zirconium oxide is a crystalline metal oxide that has found its way into the ceramics industry. It is characterized by its high thermal resistivity, mechanical resistance, and abrasive properties. [63]

The impact strength is a measure of absorb energy by the material before fracture. For denture, the impact failure can be represented by suddenly fall off dentures and collide with ground. Heat-polymerized PMMA is a brittle material in the temperature of the oral cavity, whereas zirconium oxide nano particles have a high impact strength which gives resistance to the denture.[64]

The results of the present study indicate that there was highly significant increase in impact strength in Heat polymerized polymethyl methacrylate denture base material reinforced with silanized tetragonal zirconium oxide nanoparticles (3% by wt) (Group S). The increase in impact strength that occur with the addition of 3wt% ZrO2 nanoparticles was due to good distribution of the very fine nanoparticles that enable them to enter between linear macromolecular chains of the polymer and fill spaces between chains, segmental motions of the macromolecules are restricted lead to increase strength and rigidity of the resin, so cause improvement in fracture resistance and lead to improved impact strength. [65]

Zirconium oxide (ZrO2) was used because it is excellent biocompatible material. Silanization of the nano-filler particles yields a better dispersion, eliminate aggregation and improve its compatibility with organic polymer. The addition of silanized ZrO2 nano particles increased the value of the impact strength and transverse strength compared to control group, 3wt% group has the highest impact strength. The increase in impact strength of Group S when compared to Group C was because of the strength between nanofiller and matrix which in turn prevent propagation of crack.

The result of this study stated that heat polymerized polymethyl methacrylate denture base material without reinforcement (Group C) showed the least impact strength of 12.74 ± 2.81 when compared with heat polymerized polymethyl methacrylate denture base material reinforced with silanized tetragonal zirconium oxide nanoparticles (3% by wt) (Group S). Similar results were observed in the study conducted by **Asopa et al** [52] which stated 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, possess strong atomic bond, giving rise to its desirable material characteristics. Addition of zirconia nano-fillers to acrylic resin was found to improve mechanical properties.

The highly significant increase in the impact strength when polymethyl methacrylate was reinforced with zirconium silicate nano particles 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. Forces applied are transferred to the nanoparticles which improves the impact strength. Addition of nanoparticles fills the free spaces and attract resin molecules.

The mean impact strength obtained in Group S (17.91 Kj/m^2), Group N (15.81 Kj/m^2), Group C (12.74Kj/m^2) respectively. The results obtained are similar to the study done by **Soodad A. Al-Hiloh**[58] who concluded that highly significant increase in the impact strength, transverse strength and surface hardness occurred with the incorporation of 3%wt silanized zirconium oxide nano particles.

The results of the present study indicate that there was highly significant increase in impact strength when 3% silanized zirconium oxide nano particles were added to polymethyl methacrylate denture base material. The increase in impact strength was due to the interfacial shear strength between nano filler and matrix is high because of formation of cross-links or supra molecular bonding which cover or shield the nano fillers which in turn prevent crack propagation. The crack propagation can be changed by good bonding between nano filler and resin matrix[58].

CLINICAL IMPLICATION

When the entire spectrum of this study was analyzed, it becomes evident that the heat polymerized acrylic dentures reinforced with silanized zirconium oxide nanoparticles increases the impact strength of the denture base material and thus, reduces the probability of occurrence of fracture. In addition to this, it imparts radio-opacity to the material so that any fractured remnants can be detected radiographically. This will provide it clinical advantage over conventional PMMA resin.

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. Further research is also needed to quantify the filler distribution in the polymer matrix.

LIMITATIONS OF THE STUDY

In this study samples were prepared in accordance with ADA specification number 12. The study was designed and carried out with utmost accuracy. The present study has certain limitations which are enlisted below.

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 oxide nano fillers on the surface of PMMA was not performed.

SUMMARY

The heat cure denture base resins are widely used for their excellent properties such as ease of handling , aesthetics and also polishing. Conversely, to maintain the durability of the denture . The fracture of acrylic resin denture is a usual incident. Through the ages, loss of teeth due to trauma, disease or any other cause has distressed mankind. The means of replacing missing teeth structures by artificial materials continues to account for a large part of the application of material science.[3]

This study was conducted to evaluate and compare the impact strength of heat polymerized polymethyl methacrylate denture base material reinforced with non-silanized tetragonal zirconium oxide nanoparticles and 3% silanized tetragonal zirconium oxide nanoparticles.

The samples tested were fabricated according to ISO standard 179 with 32

samples in each group.

Impact strength was tested using Charpy's impact testing machine at a speed of 6 m/s. The findings were statistically analyzed and impact test was calculated in Kj/m^2.

Results of the study shows that the mean Impact strength of Group S was maximum which was **17.91 Kj/m^2**. For **Group N,** the mean impact strength was **15.81 Kj/m^2**. And, for **Group C** the mean impact strength was **12.74 Kj/m^2**.

Statistical analysis shows highly significant difference on comparison of mean impact strength of Group S and Group N (p=0.05). Where Group S has higher Impact strength than Group N. Also, there exist high statistical difference (p=0.001) between Group S and Group C where Group S is having highest mean Impact strength as compared to Group C. Similar trend was observed when the impact strength was compared between Group N and Group C. This difference was also statistically significant (p=0.03). Where, Group N having higher mean impact strength than Group C.

Thus, the study results shows that reinforcement of polymethyl methacrylate denture base material with 3% silanized zirconium oxide nanoparticles show a significant increase in impact strength when compared with non silanized zirconium oxide nanoparticles and polymethyl methacrylate denture base material without reinforcement.

CONCLUSION

Within the limitations of this study, following conclusions were drawn:

1. Specimens with reinforcement has increased impact strength.

2. There is a significant increase in the impact strength of heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium oxide nanoparticles, when compared with Non-silanized zirconium oxide nanoparticles.

3. There is a highly significant increase in the impact strength of heat polymerized polymethyl methacrylate denture base specimens reinforced with 3% silanized zirconium oxide nanoparticles, when compared with Heat polymerized polymethyl methacrylate denture base material without reinforcement.

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TABLES

Tab.1. Distribution of impact strength across the three groups.

Groups	N	Mean	SD
Group S	32	17.91	3.96
Group N	32	15.81	3.76
Group C	32	12.74	2.81

Tab.2. Comparison of impact strength between the three groups

	Group S	Group N	Group C
Mean Impact strength	17.91±3.96	15.81±3.76	12.74±2.81
p-value	0.001*		

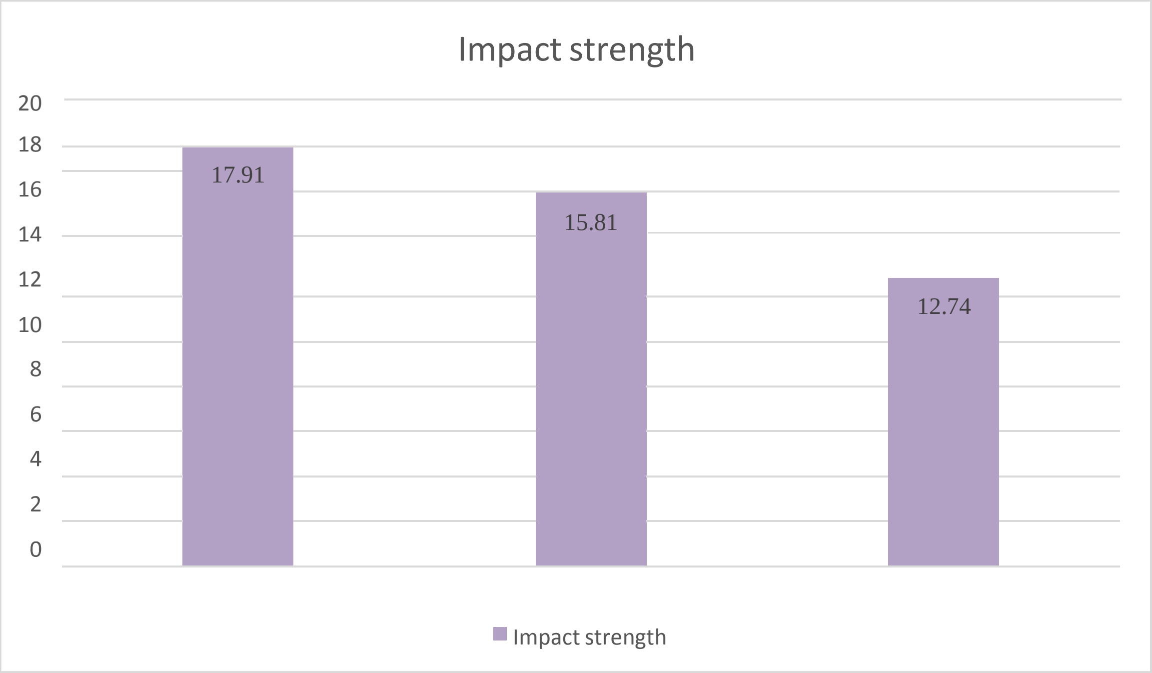
*p-value calculated using ANOVA, *p≤0.001 statistically highly significant

Tab.3. Multiple comparisons of impact strength amongst the three groups

Groups	Mean difference	p-value
Group S-Group N	2.1	0.05*
Group S-Group C	5.17	0.001**
Group N-Group C	3.07	0.03*

*p≤0.05 statistically significant; **p≤0.001 statistically highly significant calculated using post-hocTukey test

Fig. Graph showing distribution of impact strength between the three groups:-



ANNEXURE

GRAPH SHOWING DISTRIBUTION OF IMPACT STRENGTH :-

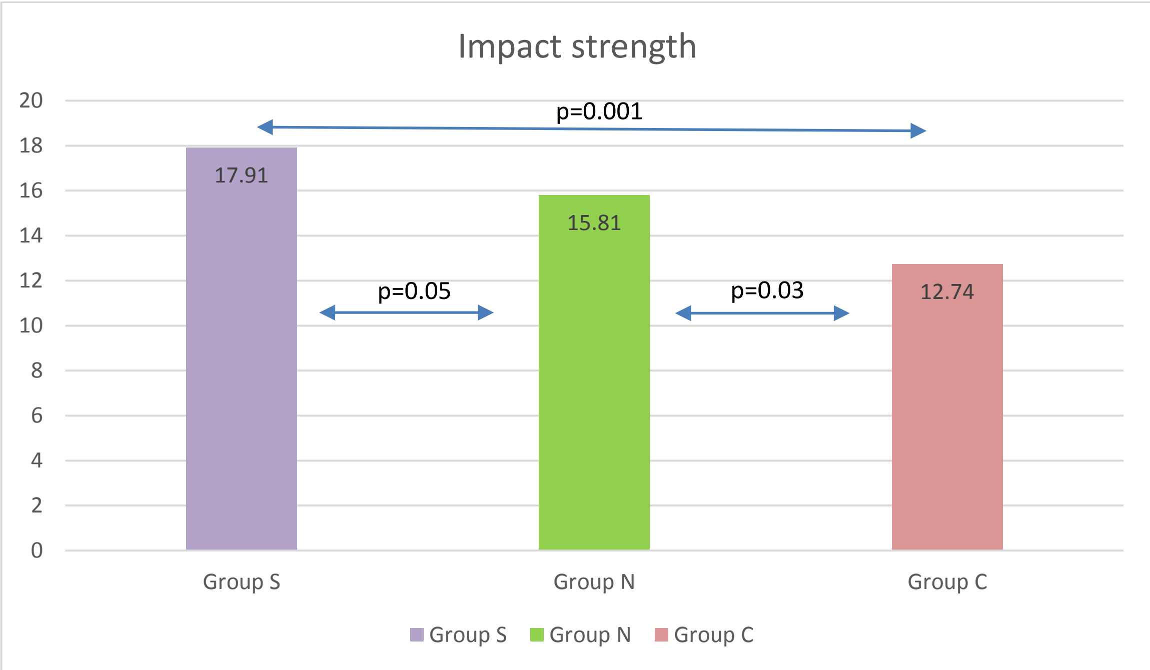


Fig. Distribution of impact strength between the three groups

SUBJECT OF RESEARCH : comparative evaluation of impact strength of heat polymerized polymethyl methacrylate denture base material reinforced with non-silanized and silanized tetragonal zirconium oxide nanoparticles : An in vitro study.

MACHINE SPECIFICATION: Charpy Impact tester (computerized,software based)
Company : International Equioment, India. Serial no.430
(As per ASTM-D 256-2002 standard)

DIMENSION : Thickness : 4mm, Width: 10mm, Length : 80 mm.

Group 1: C group								
Sr. no	Sample ID	Impact energy(J)	Charpy Impact strength (KJ/m$^{2)}$		Sr. no	Sample ID	Impact energy(J)	Charpy Impact strength (KJ/m$^{2)}$
1	No.1	0.33	11.00		17	No.17	0.46	15.33
2	No.2	0.42	14.00		18	No.18	0.40	13.33
3	No.3	0.36	12.00		19	No.19	0.38	12.67
4	N0.4	0.44	14.67		20	No.20	0.32	10.17
5	N0.5	0.46	15.33		21	N0.21	0.34	11.33
6	No.6	0.40	13.33		22	No.22	0.28	9.33
7	No.7	0.36	12.00		23	No.23	0.35	11.67
8	No.8	0.42	14.00		24	No.24	0.37	12.33
9	No.9	0.39	13.00		25	No.25	0.43	14.33
10	No.10	0.36	12.00		26	No.26	0.45	15.00
11	No.11	0.41	13.67		27	No.27	0.32	10.67
12	No.12	0.44	14.67		28	No.28	0.38	12.67
13	No.13	0.34	11.33		29	No.29	0.40	13.33
14	No.14	0.30	10.00		30	No.30	0.36	12.00
15	No.15	0.42	14.00		31	No.31	0.41	13.67
16	No.16	0.36	12.00		32	No.32	0.39	13.00
Average				**12.74**				

Group II: N group								
Sr. no	Sample ID	Impact energy(J)	Charpy Impact strength (KJ/m^{2})		Sr. no	Sample ID	Impact energy(J)	Charpy Impact strength (KJ/m^{2})
1	No.1	0.53	17.67		17	No.17	0.46	15.33
2	No.2	0.42	14.oo		18	No.18	0.40	13.33
3	No.3	0.46	15.33		19	No.19	0.38	12.67
4	N0.4	0.44	14.67		20	No.20	0.52	17.33
5	N0.5	0.46	15.33		21	N0.21	0.48	16.00
6	No.6	0.49	16.33		22	No.22	0.50	16.66
7	No.7	0.56	18.66		23	No.23	0.57	19.00
8	No.8	0.42	14.00		24	No.24	0.49	16.33
9	No.9	0.54	18.00		25	No.25	0.51	17.00
10	No.10	0.52	17.33		26	No.26	0.43	14.33
11	No.11	0.41	13.67		27	No.27	0.46	15.33
12	No.12	0.44	14.67		28	No.28	0.53	17.67
13	No.13	0.54	18.00		29	No.29	0.49	16.33
14	No.14	0.40	13.33		30	No.30	0.41	13.67
15	No.15	0.42	14.00		31	No.31	0.54	18.00
16	No.16	0.49	16.33		32	No.32	0.47	15.67
Average				**15.81**				

Group III: S group

Sr. no	Sample ID	Impact energy(J)	Charpy Impact strength (KJ/m^{2})		Sr. no	Sample ID	Impact energy(J)	Charpy Impact strength (KJ/m^{2})
1	No.1	0.60	20.00		17	No.17	0.49	16.33
2	No.2	0.62	20.67		18	No.18	0.46	15.33
3	No.3	0.55	18.33		19	No.19	0.53	17.67
4	N0.4	0.59	19.47		20	No.20	0.55	18.33
5	N0.5	0.56	18.66		21	N0.21	0.43	14.33
6	No.6	0.61	20.33		22	No.22	0.50	16.67
7	No.7	0.49	16.33		23	No.23	0.57	19.00
8	No.8	0.56	18.67		24	No.24	0.63	21.00
9	No.9	0.64	21.33		25	No.25	0.59	19.67
10	No.10	0.47	15.67		26	No.26	0.54	18.00
11	No.11	0.61	20.33		27	No.27	0.59	19.67
12	No.12	0.48	16.00		28	No.28	0.52	17.00
13	No.13	0.41	15.00		29	No.29	0.47	15.67
14	No.14	0.47	15.67		30	No.30	0.58	19.33
15	No.15	0.52	17.33		31	No.31	0.52	17.33
16	No.16	0.53	17.67		32	No.32	0.48	16.00
Average				**17.91**				