A COMPARATIVE EVALUATION OF FLEXURAL STRENGTH OF HEAT POLYMERIZED POLYMETHYAL METHACRYLATE PROVISIONAL FIXED RESTORATIVE RESIN REINFORCED WITH DIFFERENT PERCENTAGES OF 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	Mpa	Mega Pascal
7	^{0} C	Degree Celsius
8	^{0}	Degree
9	mm	Millimeter
10	i.e.	that is
11	PMMA	Polymethyl methacrylate
12	µ	Micron
13	Al_2O_3	Aluminium oxide
14	ZrO_2	Zirconium Oxide
15	TiO_2	Titanium dioxide
16	$ZrSiO_4$	Zirconium Silicate
17	SiO_2	Silicon dioxide
18	TMSPM	Trimethoxysilylpropylmethacrylate
19	Rpm	Rotations per minute
20	mins	Minutes
21	gms	Grams
22	Nps	nanoparticles

INTRODUCTION

Fixed prosthodontic treatment involves, the restoration of compromised natural teeth with crowns or partially edentulous arches with a fixed dental prostheses or implant supported prostheses. During the tooth preparation procedure, considerable amount of the tooth structure is removed and it becomes comparatively smaller in size which compromises the esthetics, masticatory efficiency and occlusal harmony.[1] Also, when an implant is placed an unaesthetic display of metal is observed. This is when the role of provisional restoration is to be considered. The need for provisional restorations emerges due to the considerable time that is required for the fabrication of the definitive prostheses.[2]

Provisional fixed dental prostheses (FDPs) are fundamental part of fixed prosthodontics including tooth and implant supported restorations.[3]

Provisional restoration has its function only for a limited period of time, after which it is to be replaced by a definitive prosthesis. The purpose of providing a provisional restoration would include immediate replacement of missing teeth, to protect pulp and maintain periodontal health, to provide occlusal stability, to maintain tooth position and for masticatory efficiency.[1] Hence, in the rehabilitation of mutilated natural teeth or partially edentulous arches, provisionalization forms an integral part of the comprehensive treatment plan. Over the past several years, provisional restorations have also become a vital diagnostic tool to evaluate function, esthetics, occlusion, periodontal response, implant healing and patient acceptance.[2]

For provisional restorations to be clinically efficient in all the above aspects, they must satisfy biological and esthetic needs, as well as mechanical requirements such as resistance to functional loads and wear, especially when it is used for long term provisionalization mainly in implant therapy, long span restorations and in areas of heavy occlusal loads.[4] In the due course of the treatment, provisional restorations may have to be removed several times and re-cemented without distortion and fracture.[5]

Currently available provisional materials can be divided into four resin groups namely, poly (methyl methacrylates), poly (R′ methacrylates), bis-acryl composite resins and visible light cured urethane dimethacrylates.[6] Poly (methyl methacrylates) are relatively inexpensive, excellent polishability, good color stability and marginal adaptation.[7]

Two forms of polymethyl methacrylate are available namely heat polymerized acrylic resin and auto polymerized acrylic resin.[8] Provisional restorations fabricated

from heat-processed acrylic resin have been used successfully.[9-1]

In clinical situations, fixed partial dentures are subjected to various functional loads. However, in certain clinical cases where there is increased parafunction, abnormal jaw relationships, cases of raised vertical dimension, long span bridges, forces acting on provisional restoration are far more than normal. Also, where provisional materials are used for extended periods of time like full mouth rehabilitation its strength assumes paramount importance.[1]

In order to assess, if a provisional restorative material is strong enough to withstand such forces, flexural strength should be determined. The flexural strength is a combination of tensile and compressive strength tests and includes the elements of proportional limit and elastic modulus measurements.[12] The flexural strength tests are essentially a test of a bar supported at each end, subjected to three-point flexure. These tests evaluate stresses as compressive at the point of application of load and tensile and shear at the point of resistance to the load applied, making them similar to the stresses produced by multi-unit fixed partial dentures.[13]

Recently, much attention has been directed towards the incorporation of inorganic nanoparticles into denture base polymethyl methacrylate (PMMA) to improve its properties. Recently incorporation of Ceramic materials to PMMA have been found to be biocompatible and also improves the mechanical properties. Various ceramic powders such as sapphire (Al_2O_3), boron nitride (BN), silicon nitride (Si_3N_4), aluminum nitride (AlN) and zirconium oxide (ZrO_2) have the advantage of being white without compromising the aesthetic appearance of the denture base material

than the metal powders.[14,15] However studies are scarce regarding its application for provisional restoration resins.

Zirconium oxide (ZrO_2), commonly referred to as zirconia, possesses strong ionic inter-atomic bonding, giving rise to its desirable material characteristics.[10] Zirconium oxide fillers (ZrO_2) can be used because of their excellent biocompatibility and also for being white; so they are less likely to alter esthetics. The nano-filler particles of zirconium oxide yields a better dispersion, eliminate aggregation and improve its compatibility with organic polymer. [16] Zirconium oxide exists in three crystalline phases i.e Monoclinic, cubic & tetragonal .[17] Tetragonal Zirconium oxide nanoparticles powder has been selected to improve the properties of PMMA, as it is a bio-compatible material that possesses high fracture resistance, improves flexural strength and fracture toughness of resin denture base.[10]

ZrO_2 ceramic material is designated as 3Y-TZP i.e yttria stabilized tetragonal zirconium polycrystal. It has several mechanical properties such as good mechanical strength, fracture toughness, hardness, wear and chemical resistance, good thermal stability and micro crack propagation during transformation toughening.[18-19]

The fracture toughness of tetragonal 3Y-TZP is approximately 8–10.3 Mpa.m1/2 and flexural strength is approximately 900 Mpa which is more than cubic and monoclinic phase of zirconia. The percentages - 1%, 2.5% and 5% of zirconium oxide are selected as they are found effective in improving the flexural strength in auto polymerized provisional acrylic resins.[20]

Silanes have the ability to bond inorganic particles to organic matrix resulting

in improved mixing, better bonding and increased matrix strength.[19] Treating the surface (Silanization Process) of an inorganic particle with an organic substance is a useful way to reduce its surface energy, increase dispersion homogeneity and thus improve the properties of the polymer nanocomposites.[10]

As limited amount of data is available in literature regarding the effect of different percentages of zirconium oxide nanoparticles on flexural strength of heat polymerized PMMA, the purpose of this study is to evaluate and compare the flexural strength of heat polymerized PMMA provisional restorative resin reinforced with different percentages of silanized zirconium oxide nanoparticles.

AIM AND OBJECTIVES

Setting Aim is the first thing in turning the invisible into the visible.

-Tony Robbins

AIM:

"To evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate provisional restorative resin reinforced with different percentages of silanized zirconium oxide nanoparticles."

OBJECTIVES:

PRIMARY OBJECTIVE:

To evaluate and compare the effect of different percentages of silanized zirconium oxide nanoparticles on flexural strength of heat polymerized PMMA provisional fixed restorative resin.

OTHER OBJECTIVE 1:

To evaluate the flexural strength of heat polymerized PMMA provisional fixed restorative resins without reinforcement.

OTHER OBJECTIVE 2:

To evaluate the flexural strength of heat polymerized PMMA provisional fixed restorative resin reinforced with 1%, 2.5% and 5% (by weight) silanized zirconium oxide nanoparticles respectively.

OTHER OBJECTIVE 3:

To compare the flexural strength of heat polymerized PMMA provisional restorative resin without reinforcement and with reinforcement by 1%, 2.5% and 5% (by weight) silanized zirconium oxide nanoparticles respectively.

OTHER OBJECTIVE 4:

To find the optimum percentage of silanized zirconium oxide nanoparticles for maximum increase in flexural strength of heat polymerized PMMA provisional restorative resin.

REVIEW OF LITERATURE

Polymethyl Methacrylate (PMMA), introduced by **Dr. Walter Wright** in 1937, is still one of the most widely used material in the field of prosthetic dentistry. The use of PMMA in fixed prosthodontics appeared in the literature in 1940 for the fabrication of inlays, crowns and fixed partial dentures. [21]

To be successful, they must fulfill biological, mechanical and esthetic requirements. These restorations should provide pulpal protection, patient comfort, positional stability, occlusal function, hygiene access, esthetics, strength and retention.[1] In clinical situations, fixed partial dentures are subjected to various functional loads.

However, in certain clinical cases where there is increased parafunction, abnormal jaw relationships, cases of raised vertical dimension, long span bridges, forces acting on provisional restoration are far more than normal. Also, where provisional materials are used for extended periods of time like full mouth

rehabilitation its strength assumes paramount importance.[1] Many trials have been made to enhance mechanical properties of provisional restorative materials.

Recently incorporation of Ceramic materials to PMMA have been found to be biocompatible and also improve the mechanical properties.[14] Few of these materials have obtained promising results in improving the fracture resistance of heat polymerized acrylic resin denture base (PMMA). The reinforcement of polymers used in dentistry with these metal-composite systems has been of prime interest.

Krug RS (1985)[21] gave a description on the functions, desirable requirements and materials used for provisional restorations. Esthetic acceptance, color stability, minimal pulpal irritation, good strength, poor thermal conductivity and ease of fabrication are important features that a provisional material should possess. Techniques for fabricating provisional restorations with poly (methyl methacrylate), poly (ethyl methacrylate) and epoxy resins were described.

Vahidi (1987)[1] advocated that the utilization of properly fabricated provisional restoration will permit a higher rate of success of the definitive treatment. This phase of restorative treatment should not be merely considered a temporary treatment but as a template for the ensuing prosthesis. The information that is obtained in this phase of treatment will reduce the problems that may be encountered in the definitive treatment. This study discussed the functions and requirements of provisional restorations. The methods of fabrication and the materials employed have been enumerated.

Gegauff AG (1987)[22] conducted a study to compare the fracture resistance of

six resins used as provisional restorative materials. These were broadly categorized as- one epimine, two poly (methyl methacrylates), one composite, two poly (ethyl methacrylates).The effect of pressure curing on their strength was also studied. They concluded that the epimine and the two poly (methyl methacrylates) had the greatest toughness whereas poly (ethyl methacrylates) had the lowest. Pressure curing had no significant effect on their fracture toughness.

Sejpal SP and Sood VK (1989)[23] studied the effect of metal fillers on thermal conductivity, tensile strength, compressive strength and radio-opacity of PMMA. Silver, copper and aluminum particles of 10 microns were used as fillers. They were added to PMMA in percentages of 5, 10, 15, 20 and 25. The study concluded that with the increase in metal fillers concentration there was a progressive increase in compressive strength of polymethyl methacrylate and decrease in tensile strength. The maximum increase in thermal conductivity was seen with aluminum, silver and copper fillers in 25% by volume concentration.

Gary S. Solnit (1991)[24] studied the effect of reinforcement of methyl methacrylate with silane treated and untreated glass fibres. In this study, transverse strength of one methyl methacrylate was tested after it was filled with either untreated or silane-treated glass fibers. Samples with untreated fibers found to be weaker than samples without fibers. Samples with silane-treated fibers were found to be stronger but the differences in strength were not statistically significant. The variances among samples with treated fibers were high. They suggested that glass fibers can be pretreated with a silane coupling agent to obtain a chemical bond between the fibers and the acrylic resin.

Vallittu PK and Lasilla VP (1992)[25] studied the effect of different types of commonly used metal wires and glass fibres as well as carbon and aramid fibres on the fracture resistance of polymethyl methacrylate. Metal strengtheners used were remanium's spring hard clasp wire, semi-circular wire, braided wire plate, stainless steel mesh and they were further divided into: glossy and sandblasted. Fibres were divided into 2 groups untreated and silanized. They concluded that all metal strengtheners increased the fracture resistance except stainless steel mesh. They found that the unsilanized glass fibres slightly weakened the test specimens. However, the weakening was not statistically significant when compared to the silanized glass fibres, which had a significant strengthening effect.

Vallittu PK (1993)[26] studied the effect of two different silane compounds on the adhesion between different fibres and acrylic resin. The fibres used were glass, carbon and aramid fibres and each type of fibres were either untreated or treated with silane agents. The fibres were studied by a scanning electron microscope (SEM) to establish the adhesion between the fibres and acrylic resin and the fracture resistance of the specimens were tested. The results showed that the adhesion between the fibres and acrylic resin improved with silanization of glass and aramid fibres. SEM photographs were taken which has confirmed these finding.

Gough M (1994)[27] reviewed the importance of provisional restorations in fixed prosthodontics. Successful crown and fixed partial dentures relies upon the precise use of provisional restorations. This article also reviewed the recent materials like autopolymerizing and light cure resins available for fabricating provisional restorations.

Christensens GJ (1996)[28] gave a description on the different materials available for provisionalization and mentioned the advantages and disadvantages of each. He stated that provisional restorations for crowns and fixed partial dentures are often poorly made, contributing to inadequately fitting restorations, sensitive teeth and occlusal instability. Provisional restorations are easy and inexpensive to be made by qualified auxiliary staff and they provide assurance that the final restorations will fit well with minimal clinical adjustments.

Lodding DW (1997)[29] stated that the role of provisional restorations used for indirect restorative and prosthodontic procedures has changed dramatically in the past several years. These restorations are no longer regarded as temporary restorations but rather as provisional restorations with distinct functions and purposes. Provisional restorations have become a vital diagnostic and assessment tool to evaluate function color, shape, contour, occlusion, periodontal response, implant healing and overall esthetics. An accurate fit is essential to maintain pulpal health of the tooth. With increased demands being placed on provisional restorations, new materials and techniques are being developed and some existing protocols are being refined to accomplish the desired goals. Provisional restorations are often placed in the oral environment for several months, requiring a well-fabricated and stable restoration. This article reviewed the current provisional materials, techniques and concepts in fabricating and maintaining long-term esthetic provisional restorations.

Zuccari AG (1997)[30] carried out a study to evaluate the effect of improving the mechanical properties of acrylic resins for provisional restorations. Aluminum oxide, magnesium oxide and zirconium oxide powders and pulverized glass particles

were separately admixed with pre-polymerized acrylic resin beads before mixing with monomer liquid. Particle loading ratios were 1, 2 and 3 vol. % with respect to pre-polymerized beads. Poly (methyl methacrylate), poly (ethyl methacrylate) and poly (isobutyl methacrylate) were used as the resins. A metal primer agent was applied in order to form a strong interphase between admixed particles and polymer matrix phase. Samples were subjected to three-point transverse bending tests at a crosshead speed of 10 mm/min. It was concluded that two vol. % admixtures in a PMMA resin matrix showed significant improvement in the mechanical properties and among the oxide particles, zirconia exhibited the highest modulus of elasticity, transverse strength, toughness and hardness.

Samadzadeh A (1997)[31] was conducted a study to evaluate the effects of a plasma-treated woven polyethylene fiber (Ribbond) on the fracture strength of poly(methyl methacrylate) (Coldpac) and a light cure provisional restorative material (Provipont DC). The specimens were divided into 4 groups of 10 each which were reinforced Provipont DC, unreinforced Provipont, reinforced PMMA and unreinforced PMMA. Plasma-treated polyethylene fiber reinforced PMMA restorations showed no significant increase in fracture strength when compared to the unreinforced restorations whereas reinforced light cure provisional restorative material revealed significantly higher fracture strength than the unreinforced light cure and PMMA provisional restorations.

P. K. Vallittu (1997)[32] conducted a study to determine how curing gamma methacryloxy-propyl-trimethoxy-silane (ý-MPS) to the surface of E-glass fibre affects the transverse strength of autopolymerizing polymethyl methacrylate-glass fibre

composite (PMMA-GF). Unidirectional glass fibres treated with y-MPS solution and cured for various lengths of time at different temperature were used as a strengthener of PMMA test specimens. They found that mean concentration of glass fibres in the test specimens was 17 wt%. The unreinforced test specimens and the test specimens reinforced with unsilanized fibres were compared, but no statistical difference in their transverse strengths was found.

They also found that when the transverse strength compared at different curing temperatures of the ý-MPS, the highest transverse strength (152 MPa) for the PMMA-GF composite was obtained by curing the MPS for 120 min at + 100°C and the lowest strength (91 MPa) was obtained by polymerizing the MPS at + 150°C . However, SEM photomicrographs taken from the interface of the glass fibre and PMMA showed that the fibre adhered equally well to the PMMA treated at + 100°C or at + 150°C.

They suggested that reduction in transverse strength of composite test specimens is caused by other factors such as by improper impregnation of PMMA into the fibre bundle, rather than by inadequate adhesion.

P.K Vallittu and DT Docent (1999)[33] conducted a study on flexural properties of heat cure acrylic resin polymers reinforced with pre-impregnated unidirectional and woven glass fibres. They used continuous unidirectional and woven pre-impregnated glass fibre reinforcements (Stick and Stick Net) to reinforce heat cured denture base and auto-polymerizing denture base polymers and a temporary fixed partial denture polymer was also reinforced with the Stick reinforcement material. A 3-point bending test was used to measure transverse

strength and flexural modulus and ultimate strain at fracture was calculated. The cross section of the specimens was examined with SEM to evaluate degree of impregnation of fibres in polymer matrix. They found that the Stick reinforcement increased considerably transverse strength and flexural modulus of polymers. The Stick Net reinforcement increased strain at fracture of the polymers. Fibres of Stick and Stick Net were well impregnated with the resin of polymer matrix.

Kanie T, Fujii K, Arikawa H and Inoue K (2000)[34] studied the reinforcing effect of woven glass fibres on deflection, flexural strength, flexural modulus and impact strength of acrylic denture base polymer. The flexural strength and deflection was significantly higher in specimens reinforced with silanized glass fibres of 1 mm thickness than those of unreinforced specimens. Also, the impact strength was significantly higher in specimens reinforced with silanized glass fibre of 2 mm thickness than that of unreinforced specimens.

John J, Gangadhar SA (2001)[35] were conducted a study to determine whether the flexural strength of a commercially available, heat-polymerized provisional restorative material could be improved through reinforcement with 3 types of fibers. The resin was reinforced with glass, aramid, or nylon fibers. Flexural strength was evaluated with a 3-point bending test. Highest flexural strength was for the material reinforced with glass fibers, followed by aramid and nylon reinforcements. Within the limitations of this study, it was concluded that the flexural strength of heat-polymerized PMMA provisional restorative material was improved after reinforcement with glass or aramid fibers.

Henry M. Young, Charles T.Smith, Dean Morton (2001)[36] conducted a

study to evaluate the quality of a polymethyl methacrylate resins and bis-acryl composite resin based provisional materials. They used one bis-acryl composite Integrity and two Polymethyl methacrylate resins- C&B resin and Snap. In their study they evaluated occlusion, contour, marginal adaptation and 222 provisional crowns fabricated by the above materials. They found that of all the materials, Integrity was superior in the categories of contour and marginal adaptation but no significant difference were established for occlusion and finish. They also found that Integrity was statistically superior to Snap in categories of occlusion, contour and marginal adaptation.

Burns DR, Beck DA, Nelson SK (2003)[7] reviewed on provisional fixed restorative materials and concluded that provisional therapy involves numerous materials and techniques that require knowledge and technical experience. No ideal material, suitable for all clinical conditions is presently available. The material selection should be based on the strength and weakness of a given material relative to the clinical mandates for specific treatments.

Tamer Hamza, Stephen F. Rosensteil, Mohamed M. Elhosary,Rabab M. Ibraheem (2004) [37] conducted a study to determine the effect of fibre reinforcement on fracture toughness and flexural strength of provisional restorative resins. They incorporated six types of fibres (Construct, Fibrestick, Ribbond normal, Ribbond THM, Ribbondtriaxial or Firenet) in three different provisional materials (Jet, Trim and Temphase) and tested for fracture toughness and flexural strength in a universal testing machine. They found that the addition of fibres to provisional resin increased both fracture toughness and flexural strength. They concluded that the use of fibres is

an effective method to increase fracture toughness and flexural strength of provisional restorative resins. The surface treatment of fibres also greatly influences the fracture toughness and flexural strength.

S.M Chung, A. U.Yap, S. P.Chandra, C. T. Lim (2004)[38] conducted a study to compare two test methods used to evaluate the flexural strengths of resin based dental composites. The materials used in this study were from the same manufacturer 3M ESPE and included microfill (A110), Minifilled (Z100 and Filtek Z250), polyacid modified (F2000) and flowable (FiltekFlowable) composites. The mean flexural strengths ranged from 66.1 to 147.21 Mpa for Three-point bending test and 67.27 to 182.82 Mpa for Ball-on-three-ball Biaxial test method. Compared with the three point bending test , the ball –on –three ball biaxial test method has an advantage of using appropriate size scale specimens. However because of complex contact stresses, the reproducibility of biaxial test method is not good (coefficient of variation >10%). So they concluded that biaxial test is not a more reliable test method when compared to that of ISO three-point- bending test in the mechanical evaluation of brittle dental materials.

Matinlinna J, Lassila L, Özcan M, Yli-Urpo A (2004)[39] reviewed silanes and their clinical application in dentistry. They stated that silanes are hybrid organic-inorganic compounds which function as mediators and through dual reactivity promote adhesion between dissimilar, inorganic and organic matrices. They are called primers, coupling agents, or sizes, depending on their function and substrates. Methacryloxypropyltrimethoxysilane (or 3-trimethoxysilylpropyl methacrylate [MPS]) is most commonly applied silane in dental laboratories and chairside. MPS is

used to optimize and promote the adhesion, through chemical and physical coupling, between metal-composite, ceramic-composite and composite-composite.

S. Dagar,A. Pakhan,A.Tunkiwala (2005)[9] evaluated the flexural strength of auto polymerizing poly-methyl methacrylate (PMMA) and heat polymerizing poly-methyl methacrylate with a newly introduced composite resin Protemp-II . They stored the specimen at room temperature for 24 hours and then to simulate the oral environment the specimens were stored at room temperature for 24 hours and incubated in normal saline at 37 Degree Celsius for 5 days. They tested the specimen with three point bent test and concluded that the highest value for fracture resistance was exhibited by heat polymerized PMMA followed by Protempt-II and self-polymerized PMMA.

Vojdani M, Khaledi AAR (2006)[40] conducted a study in which they observed that the transverse strength of heat polymerized denture base resin was considerably enhanced by including either metal wires or glass fibres. Moreover the flexural strength of specimens reinforced with continuous unidirectional glass fibres was significantly higher than that of metal wire or woven fibre reinforcements

Ayad NM, Badawi MF, Fatah AA (2008)[41] evaluated the effect of reinforcing high-impact acrylic resin (Metrocryl HI) with zirconia powder in two different concentrations (5% and 15%) on the impact strength, transverse strength, water sorption, surface hardness and solubility. They concluded that the addition of zirconia results in a highly significant increase in transverse strength of high-impact acrylic resin when compared to control samples by a factor of 29% and 76%, in a concentration of 5% and 15% respectively. This increase was directly proportional

to the concentration of zirconia. There was no significant difference in impact strength, surface hardness and water solubility when compared to zirconia free high impact resin.

Ellakwa AE, Morsy MA, El-Sheikh AM (2008)[42] conducted a study to evaluate the effect on the flexural strength and thermal diffusivity of heat-polymerized acrylic resin on adding 0%, 5%, 10%, 15%, and 20% by weight aluminium oxide powder (Al_2O_3). The study concluded that incorporating Al_2O_3 powder from 5% to 20% by weight into conventional heat-polymerized denture base resin resulted in an increase in both flexural strength and thermal diffusivity over control samples. The flexural strength increased significantly after incorporation of 10% aluminium oxide powder. The highest mean flexural strength was seen with addition of 15% by weight of aluminium oxide powder. Thus, increasing the flexural strength and heat transfer characteristics of the acrylic resin base material could result in more patient satisfaction.

Dagar SR, Pakhan AJ, Thombare RU, Motwani BK (2008)[43] evaluated the flexural and impact strength of commercially available heat polymerizing PMMA denture base resin reinforced with glass and nylon fibres. In this study it was concluded that fiber-reinforced specimens were more resistant to impact and flexural fatigue than conventional PMMA specimens. When compared with nylon fiber reinforcement it was found that glass fiber reinforcement considerably improves both impact and flexural strengths of denture base resin. Silane-impregnated glass fiber reinforcement suits best to increase the flexural and impact strengths of heat-polymerized PMMA denture base resin.

Ihab NS, Moudhaffar M (2011)[44] studied the effect of addition of modified nano-zirconium oxide (ZrO_2) on strength and radio-opacity of heat cured acrylic denture base material. The nanoparticles were coated with a layer of trimethoxysilypropylmethacrylate (TMSPM). They were sonicated in monomer (MMA) in different percentages 2%, 3%, 5% and 7% by weight. It was found that maximum increase in transverse strength, impact strength, and radio-opacity was observed in denture base reinforced with 5wt% of nano-ZrO_2.

Alhareb AO, Ahmad ZA (2011)[45] evaluated the effect of 5wt% of Al_2O_3/ZrO_2 (80:20 ratio) reinforcement on the fracture toughness, flexural, and tensile properties of PMMA denture base. They concluded that the PMMA reinforced with Al_2O_3/ZrO_2 improved the tensile modulus, fracture toughness and flexural properties of the denture base material

Vojdani M, Bagheri R, Khaledi AAR(2012)[15] evaluated the effects of 0.5, 1, 2.5 and 5 wt% of aluminum oxide addition on the flexural strength, surface hardness, and roughness of heat-polymerized acrylic resin They concluded that reinforcement of the conventional heat-cured acrylic resin with 2.5 wt% of aluminium oxide powder significantly increased its flexural strength and hardness with no adverse effects on the surface roughness.

Ihab NS, Hassanen KA, Ali N.A (2012)[10] evaluated impact strength, tensile strength and color stability of heat polymerized denture base reinforced with silanated and non-silanated zirconium oxide (ZrO_2) nanofillers. Silanization was done by coating with a layer of trimethoxysilypropylmethacrylate (TMSPM) and nanoparticles were sonicated in monomer in two percentages of 3% and 5% by weight of polymer.

The maximum increase in impact strength was observed in PMMA reinforced with 5%wt of silanated ZrO_2 nano-fillers. Silanized ZrO_2 nano-fillers were effective in improving impact strength while it was not effective in improving the tensile strength. Also, significant color differences were seen between control group and specimens incorporated with zirconium oxide nano-fillers in different immersion solutions.

Saritha MK, Shadakshari S, Nandeeshwar DB (2012)[14] conducted an *in vitro* study to investigate the flexural strength of conventional heat polymerized denture base resin reinforced with 5%, 10% and 15% by wt. of aluminium oxide powder. They concluded that incorporation of 10% and 15% by wt. of aluminium oxide powder to heat cure denture base resin significantly increased the flexural strength of denture base resin. The highest flexural strength was found with incorporation of 15% by wt. aluminum oxide powder to heat cure denture base resin.

Natarajan P and Thulasingam C (2013)[46] studied the effect of glass and polyethylene fiber reinforcement on flexural strength of provisional restorative resins and concluded that in both heat and self-cure poly methyl methacrylate resin, the polyethylene fiber reinforcement provides the greatest strength than glass fiber reinforcement.

Sodagar A, Bahador A, Khalil S, Shahroudi AS, Kassaee MZ (2013)[47] evaluated the effect of TiO_2 and SiO_2 nano-particles on flexural strength of polymethyl methacrylate acrylic resins containing nanoTiO_2, SiO_2 and TiO_2 with SiO_2 in two concentrations of 1wt % and 0.5wt % were made. It was concluded that incorporation of TiO_2 and SiO_2 nano-particles into acrylic resins adversely affected the flexural strength of the final products and this effect was directly correlated with

the concentration of nano-particles. The reason for this decrease was attributed to incorporation of nano-particles into polymethyl methacrylate acrylic resin which caused these particles to agglomerate and aggregate. The agglomerated compounds can act as stress concentrating centres in the matrix and adversely affect mechanical properties of the polymerized material. Hence it was suggested to use appropriate substances as silane coupling agent between nano-TiO_2 and SiO_2, thus alleviating its deleterious effect on mechanical properties.

Sharma SP, **Jain AR, Balasubramanian R, Alavandar S, Manoharan PS. (2013)**[48] compared the flexural strengths of provisional restorative material fabricated using light polymerized composite resin, urethane dimethacrylate (UDMA), auto polymerized resin and poly Methyl Methacrylate (PMMA).They observed that the mean flexural strength of polymethyl methacrylate materials was significantly higher than the urethane dimethacrylate material. They concluded that PMMA could be a better provisional restorative material for an extended period, when the patient exhibits parafunction habits or when long span prosthesis is planned.

Fonseca RB, Favar IN, Kasuya AV, Abr M, da Luz NF, Naves LZ (2014)[49] **e**valuated the flexural strength of acrylic resin bars depending on the addition of glass fibers with or without previous 3-methacryloxypropyl-trimethoxysilane (silane) application. Flexural strength and scanning microscopy (SEM) evaluation were performed. SEM showed better fiber-resin interaction for silane groups and fractures around fibers on non silane groups. Previous silane application enables the addition of greater quantity of glass fibers and better interaction with the acrylic resin resulting in higher flexural strength. Without silane, fibers seem to act as initial crack points due

to poor interaction.

Xiu-Yin ZHANG, Xin-Jing ZHANG, Zhuo-Li HUANG, Bang-Shang ZHU and Rong-Rong CHEN (2014)[50] investigated the hybrid effects of ZrO_2 nanoparticles (nano-ZrO_2) and aluminum borate whiskers (ABWs) on flexural strength and surface hardness of denture base resin, polymethyl methacrylate (PMMA). ZrO_2 nanoparticles had an average granularity of 90 nm and an average surface area of 7±2 m2/g. Aluminum borate whiskers were of 5–30 μm length and a diameter of less than 1.5 nm, with a surface area of 2.0–2.5 m2/g. Both nano-ZrO_2 and ABWs were modified by silane coupling agent (3-Methacryloxy propyl trimethoxysilane) before being mixed with PMMA. The nanocomposites were divided into four groups according to different amounts of nano-ZrO_2 at 1, 2, 3, and 4 wt%. Each group was subdivided according to ZrO_2/ABW mass ratios of 2:1, 1:1, 1:2, and 1:3. Unsilanized ZrO_2-ABW/PMMA nanocomposites were prepared in the same way by admixing unsilanized nano-ZrO_2 and ABWs, and were regarded as the control groups. Pure PMMA was used as control group. They concluded that maximum flexural strength was achieved with 2% of nano-ZrO_2 at ZrO_2/ABW ratio of 1:2, causing flexural strength to increase by 52% when compared with pure PMMA.

Jasim BS and Ismail IJ (2014)[51] evaluated the effect of addition of surface treated aluminium oxide nanofillers on some properties of heat cured (PMMA) where silanized (Al2O3) nanoparticles 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 transverse strength was observed with the addition of (Al2O3) nanoparticles to (PMMA) at the percentage of 1wt%, the value

was 117.72 Mpa and significant increase at 2wt%; while a significant reduction occurred in transverse strength at the percentage of 3% the value was 90.110 Mpa. They concluded that addition of $Al2O_3$ nanoparticles to acrylic resin improves the thermal properties and transverse strength of acrylic resin at the same time this addition decreases water sorption and solubility.

Kareem S, Moudhaffer M (2015)[52] studied the effect of silanized zirconium silicate nanopowder reinforcement on some mechanical and physical properties of heat cured polymethyl methacrylate denture base material. Silanization was done by coating zirconium silicate nanoparticles with a layer of trimethoxysilylpropylmethacrylate (TMSPM). Addition of silanized zirconium silicate nano fillers was done in two groups 1% and 1.5% by weight of polymer. They concluded that the maximum increase in impact strength, transverse strength, and surface hardness was observed in denture base nanocomposites containing 1.5% $ZrSiO_4$ nanoparticles. In addition, highly significant decrease in water sorption and solubility and non-significant increase in surface roughness was also noticed.

Alhavaz A, RezaeiDastjerdi M, Ghasemi A, Ghasemi A, AlizadehSahraei A (2017)[8] Conducted a study on autopolymerized poly methyl methacrylate resin. They categorized the specimen in to four group as follows: pure PMMA, PMMA with 1%, PMMA with 2.5% and PMMA with 5% weight of untreated zirconia nanofillers. They had drawn an inference that the reinforcement of autopolymerized acrylic resin with 2.5 % weight of untreated zirconia nanofillers significantly increased its flexural strength and surface hardness over control samples.

Astudillo-RubioD, Delgado-Gaete A, Bellot-Arcís C, Montiel-Company J,

Pascual-Moscardó A, Almerich-Silla J. (2018)[53] conducted a systematic review on mechanical properties of provisional dental material. It may be concluded that dimethacrylate-based provisional restorations possess better mechanical behavior than monomethacrylate in terms of flexural strength and hardness, but there is no significant differences in fracture toughness. PMMA shows greater flexural strength than PEMA, among the monomethacrylates.

Ergun G, Sahin Z, Ataol AS (2018)[54] studied the mechanical and physical properties of heat cure PMMA denture base material by adding different percentages of tetragonal zirconium oxide nanoparticles after thermocycling. The specimen were divided into four groups that were- 5% nano-ZrO_2, 10% nano-ZrO_2, 20% nano-ZrO_2 and PMMA without nano-ZrO_2.The study concluded that there is increase in hardness, surface roughness and water sorption value of PMMA with zirconium oxide nanoparticles. 5% of reinforced nano-ZrO_2 showed the highest value of hardness.

MATERIALS AND METHOD

Provisional fixed dental prostheses (FDPs) are fundamental part of fixed prosthodontics including tooth and implant supported restorations.[3]

Provisional restorations fabricated from heat-processed acrylic resin have been used successfully.[9-10] In clinical situations, fixed partial dentures are subjected to various functional loads. However, in certain clinical cases where there is increased parafunction, abnormal jaw relationships, cases of raised vertical dimension, long span bridges, forces acting on provisional restoration are far more than normal.[1] In order to assess if a provisional restorative material is strong enough to withstand such forces, flexural strength should be determined.[12] Several studies have reported the effect of different concentrations of zirconium oxide (ZrO2) powder on the flexural strength, fracture toughness, and hardness of heat-polymerized acrylic resin.[8] Modifications can be done in the composition of conventional acrylic resin provisional restorative material to achieve this purpose.

This *in-vitro* study was done to evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate provisional restorative resin reinforced with different percentages of silanized zirconium oxide nanoparticles.

Material and methods have been divided under the following heads:

I) Materials

II) Armamentarium and equipments

III) Method

I) Materials (PLATE I)

SR. NO.	MATERIALS	MANUFACTURER	BATCH NO.
1	Heat polymerized acrylic resin (Fig.1)	DPI Heat Cure, (Dental products of India Ltd)	655
2	Die stone (Fig.2)	Ultrarock; Kalabhai Karson Pvt Ltd, India	161003
3	Zirconium oxide nanoparticles (Fig. 3)	Nanoshell	NS6130-01-180
4	Silane coupling agent (3-Trimethoxypropylsilylmethacrylate TMPSM) (Fig 4)	Sigma Aldrich	440159
5	Toluene (Fig 5)	Emplura R	108323
6	Cold mould seal (separating medium) (Fig. 6)	DPI(Dental products of India Ltd)	8117

II) Armamentarium and equipments (PLATE II & III)

1. High accuracy balance (Fig.7)
2. Ultrasonicator (Fig.8)
3. Magnetic stirrer (Fig. 9)
4. Vacuum rotary evaporator (Fig10.)
5. Acrylizer with thermostat (Fig.11)
6. Universal testing machine (Fig.12)
7. Rubber bowl and plaster spatula (Fig.13)
8. Varsity flasks and clamps (Fig13)
9. Sand paper (No. 120) (Fig.14)
10. Camel hair brush (Fig. 14)
11. Glass Beaker (Fig.14)
12. Sterile Syringe (Fig.14)
13. Mixing spatula (Fig.14)
14. Petroleum jelly (Fig.14)
15. Porcelain jar and Dapen dish (Fig. 14)
16. Vernier caliper (Fig.15)
17. Brass metal dies (Fig.16)
18. Para-film (Fig.17)
19. Hydraulic bench press (Fig.18)
20. Distilled water (Fig.19)

III) Methodology (PLATE IV)

The basic methodology consisted of –

a. Die preparation

b. Silanization of zirconium oxide nanoparticles

c. Preparation of gypsum moulds for fabrication of specimens

d. Preparation of heat polymerized polymethyl methacrylate provisional restorative resin specimens

e. Preparation of heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 1 % wt silanized zirconium oxide nanoparticles

f. Preparation of heat polymerized polymethyl methacrylate provisional restorative resin

g. specimens reinforced with 2.5 % silanized zirconium oxide nanoparticles

h. Preparation of heat polymerized polymethyl methacrylate provisional restorative resin

i. specimens reinforced with 5% silanized zirconium oxide nanoparticles

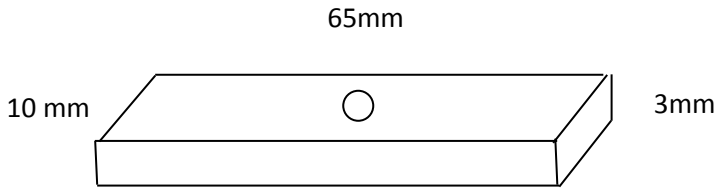
j. Testing of specimens for flexural strength.

A total of 60 specimens were prepared with each group having 15 specimens. The specimens were divided under the following groups:-

CONTROL GROUP	**EXPERIMENTAL GROUPS**		
GROUP-A	**GROUP- B**	**GROUP- C**	**GROUP- D**
Heat polymerized PMMA provisional restorative resin without nanoparticles	Heat polymerized PMMA provisional restorative resin + 1wt% silanized ZrO_2 nanoparticles	Heat polymerized PMMA provisional restorative resin + 2.5wt% silanized ZrO_2 nanoparticles	Heat polymerized PMMA provisional restorative resin + 5wt% silanized ZrO_2 nanoparticles
15 samples	15 samples	15 samples	15 samples
TOTAL- 60 SAMPLES			

a) Die preparation:

Metal dies were fabricated to prepare moulds for the fabrication of heat polymerized polymethyl methacrylate provisional restorative resin material specimens. Three brass metal dies of dimension 65 mm in length, 10 mm in width, and 3 mm in height (65×10×3) were fabricated. (ISO 1567 standard).[42]



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

holes to facilitate easy removal of dies from the stone mold.

b) Silanization of zirconium oxide nanoparticles

Pure toluene solvent in the amount of 200ml and 30gms zirconium oxide nanoparticles (ZrO_2) were placed in the glass beaker of capacity of 250ml and sonicated by ultrasonic probe for 20 mins. Then magnetic stirrer was placed in the beaker. Then 1.5gm (5%wt to nano-filler) of silane (Trimethoxypropylsilyl methacrylate TMPSM) was added dropwise by using sterile syringe under rapid stirrer. The beaker was covered with parafilm and the slurry was left for two days. The slurry was placed in rotary evaporator under vacuum of 60ºC, rotation of 150 rpm for 30 mins to remove toluene solvent. Finally, the silanized nanoparticles were made moisture free by placing in vacuum oven (20 hours at 60ºC) and then stored at room temperature before use.[55]

c) Preparation of gypsum mould for fabrication of specimens

Preformed brass metal dies were used to prepare gypsum moulds. Before investing them, the threaded holes on the dies were blocked with carding wax. A thin layer of petroleum jelly was applied on three metal dies which were then invested in the lower half of the varsity flask. Die stone was used for base flasking and care was taken to embed half the thickness of the metal die in it.[56] Once the investment material had set, a thin layer of petroleum jelly was applied to the metal dies and to the investment material and then the counter flasking was done. The flasks were closed to ensure metal to metal contact between the base of the flask and its counterpart. After the investment material had set (1 hour)[13] the flasks were opened and the carding wax within the holes was removed. The dies were engaged with a screw and gently teased

out. The moulds were then immersed in hot water to remove any traces of petroleum jelly, wax and also to facilitate application of separating medium. These moulds thus obtained were used for the fabrication of heat polymerized acrylic resin provisional restorative resin specimens (PMMA).

d) Preparation of heat polymerized polymethyl methacrylate provisional restorative resin specimens without reinforcement (n=15)

15 specimens were prepared using conventional heat polymerized provisional restorative resin (PMMA).

Monomer and polymer were mixed in a ratio of 1:2.5 by weight as per manufacturer's recommendations.[57]The materials were weighed using electronic balance of high accuracy. 7.5gms of polymer powder and 3ml of monomer was used for preparing 3 specimens. Packing was done at dough stage, following which trial closure was performed. Final closure was done under a hydraulic bench press at a pressure of 3000psi for 3 mins (according to the manufacturer). The flask was clamped and maintained under pressure for 1 hour.[13] It was then immersed in water in an acrylizer at room temperature. The temperature was raised slowly upto 74^0C and was held for 2 hours. The temperature was then raised to 100^0C and was maintained for 1 hour.[58]After completion of this short curing cycle, the flask was removed from the water bath and allowed to bench cool at room temperature prior to deflasking.[58]

The polymerized specimens were carefully removed and specimens with defects were discarded. Finishing of the specimens was done using sand paper (No.

120). The finished specimens were stored in distilled water for 1 week at room temperature.[15]

e) Preparation of heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 1wt % silanized zirconium oxide nanoparticles (n=15)

15 specimens were prepared using conventional heat polymerized provisional restorative resin (PMMA) reinforced with 1 % silanized zirconium oxide nanoparticles.

For complete homogenous dispersion, 1 % silanized zirconium oxide nanoparticles were added to the monomer.[8] As per group A, same proportion was followed for fabrication of specimens. 7.425gms of polymer powder, 3ml of monomer and 0.075gms of silanized zirconium oxide nanoparticles were taken for fabrication of 3 specimens. An electronic balance of high accuracy was used to weigh the materials.

Silanized zirconium oxide nanoparticles were well dispersed in monomer by using an ultrasonicator. The ultrasonication was done at 120W,60KHz for 3 minutes. This allowed the homogenous dispersion of silanized zirconium oxide nanoparticles in the monomer. [8]

Immediately to this suspension, polymer powder was added gradually to reduce the possibility of particle aggregation and phase separation. Mixing was done according to the manufacturer's instructions. Packing, curing, deflasking and finishing was done in the same manner as that for fabrication of heat polymerized polymethyl

methacrylate provisional restorative resin specimens (Group A). Specimens with defects were discarded. The finished specimens were stored in distilled water for 1 week at room temperature.[52]

f) Preparation of heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 2.5% silanized zirconium oxide nanoparticles (n=15)

15 specimens were prepared using conventional heat polymerized provisional restorative resin (PMMA) reinforced with 2.5% silanized zirconium oxide nanoparticles.

For complete homogenous dispersion, 2.5% silanized zirconium oxide nanoparticles were added to the monomer. [8] As per group 1, same proportion was followed for fabrication of specimens. 7.313gms of polymer powder, 3ml of monomer and 0.187gms of silanized zirconium silicate nanoparticles were taken for fabrication of 3 specimens. As per group A, same proportion and procedure was followed for fabrication of specimens reinforced with 2.5% silanized zirconium oxide nanoparticles.

The finished specimens were stored in distilled water for 1 week at room temperature. [52]

g) Preparation of heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 5 % silanized zirconium oxide nanoparticles (n=15)

15 specimens were prepared using conventional heat polymerized provisional restorative resin material (PMMA) reinforced with 5 % silanized zirconium oxide nanoparticles.

For complete homogeneous dispersion, 5% silanized zirconium oxide nanoparticles were added to the monomer. [8] As per group A, same proportion was followed for fabrication of specimens. 7.125gms of polymer powder, 3ml of monomer and 0.375gms of silanized zirconium oxide nanoparticles were taken for fabrication of 3 specimens. As per group A, same proportion and procedure was followed for fabrication of specimens reinforced with 5% silanized zirconium oxide nanoparticles.

The finished specimens were stored in distilled water for 1 week at room temperature. [52]

h) Testing of specimens (PLATE IV)

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

Flexural strength was tested with a universal testing machine system, at a 5.0mm/minute crosshead speed.[14]The specimens were supported on the jig separated at a distance of 50 mm. Load was applied at the centre of the specimen. Stress- strain curves were recorded on a chart throughout the flexural tests. The maximum load

during fracture was determined from the chart and recorded as fracture load in N (Newton) and the flexural strength was calculated in MPa.

Flexural strength (FS) was calculated using the formula.

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

Where, FS = flexural strength (N/mm^2),

P = load at fracture (N),

I = distance between the supporting wedges (mm),

b = width of the specimen (mm) &

d = thickness of the specimen (mm).[15]

PLATE I

MATERIALS

Fig 1: Heat polymerized acrylic resin

Fig 2: Die Stone

Fig 3: Zirconium oxide nanoparticles

Fig 4: Silane coupling agent

Fig 5: Toluene

Fig 6: Cold mould seal

PLATE II

ARMAMENTARIUM AND EQUIPMENTS

Fig 7: High accuracy balance

Fig 8: Ultrasonicator

Fig 9: Magnetic stirrer

Fig 10: Vacuum rotary evaporator

Fig 11: Acrylizer with thermostat

Fig 12: Universal testing machine

PLATE III

ARMAMENTARIUM AND EQUIPMENTS

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

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

Fig 15: Vernier Caliper

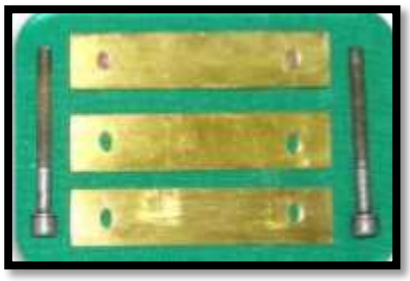


Fig 16: Brass metal dies

Fig 17: Parafilm

Fig 18: Hydraulic bench press

Fig 19: Distilled water

PLATE IV

METHODOLOGY

Fig 20: Preparation of gypsum mould to obtain specimens

Fig 21: Silanization process of zirconium silicate nanoparticles

Fig 22: Testing of specimens

PLATE V

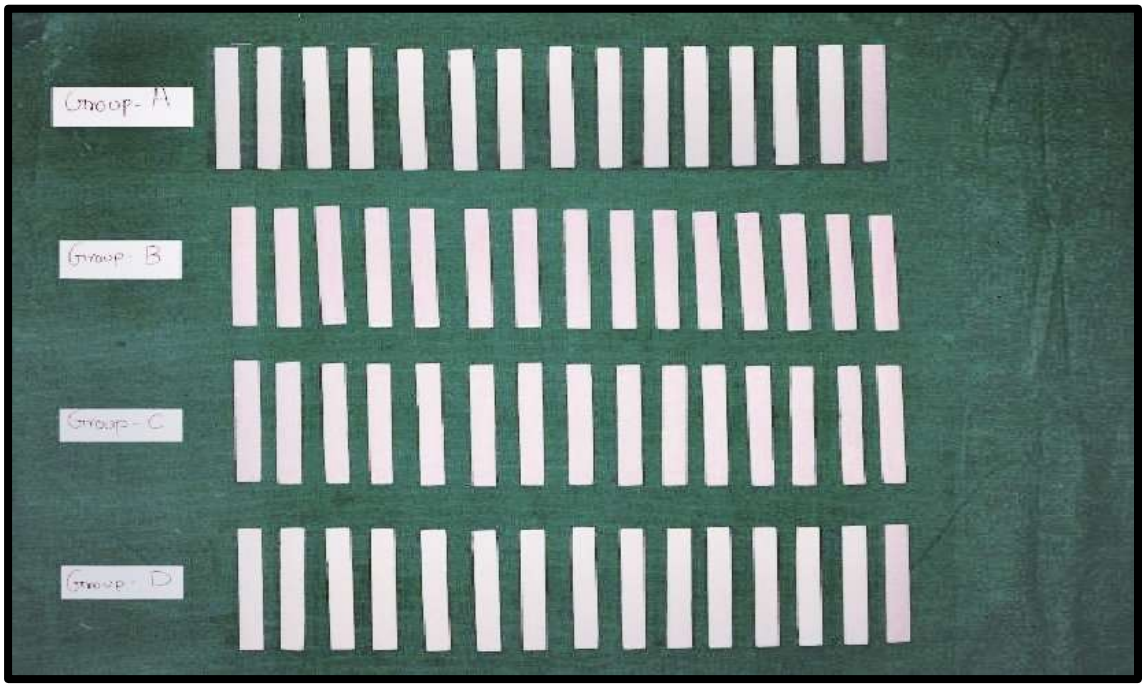


Fig 23: Samples of Group A, Group B, Group C, Group D before flexural strength testing

Fig 24: Samples of Group A, Group B, Group C, Group D after flexural strength testing

RESULTS

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

In this study the flexural strength of heat polymerized polymethyl methacrylate provisional restorative resin without reinforcement and reinforced with different percentages of silanized zirconium oxide nanoparticles was evaluated and compared.

A total 60 specimens were prepared and were divided into four groups. Each group included 15 specimens.

Distribution of samples into groups

Sr no.	Group	Code	n = no. of samples
1	The control group; heat polymerized polymethyl methacrylate provisional restorative resin without reinforcement	Group A	15
2	Heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 1wt% silanized zirconium oxide nanoparticles	Group B	15
3	Heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 2.5wt% silanized zirconium oxide nanoparticles	Group C	15
4	Heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 5wt% silanized zirconium oxide nanoparticles	Group D	15
	Total number of samples		60

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

STATISTICAL ANALYSIS

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

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

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

Where:

$\overline{X}$	=	Mean
X	=	Values of the variables
Σ	=	Sum of the value
n	=	Number of observations
Min	=	Minimum Value
Max	=	Maximum Value

- **Null Hypothesis:** There is no significant difference in the flexural strength between the groups i.e. Group A = Group B = Group C = Group D

- **Alternate Hypothesis:** There is a significant difference in the score recorded between the groups i.e. Group A ≠ Group B ≠ Group C ≠ Group D

- **Level of Significance:** α=0.05

One-way ANOVA

- The One-Way ANOVA test produces a one-way analysis of variance for a quantitative dependent variable by a single factor (independent) variable. Analysis of variance is used to test the hypothesis when several means are equal. This technique is an extension of the two-sample t test.

- In addition to determining that differences exist among the means, we may require to know which means differs and post hoc tests was used. In the present study one- way ANOVA was applied to find out the mean difference.

Tukey's multiple post hoc Test

- Once it is determined that differences exist among the means, post hoc range tests and a pair-wise multiple comparisons aid in determining which means differ. Range tests identify homogeneous subsets of means that are not different from each other. Pairwise multiple comparisons test the difference between each pair of means, and yield a matrix where asterisks indicate significantly different group means at an alpha level of 0.05.

- Tukey's HSD (Honestly significant Difference) is one of the widely used post hoc tests. In the present study Tukey's HSD post hoc Test was applied to make pairwise multiple comparisons to find out the difference between each pair of mean values of the six groups included in the study.

Table 1 provides the descriptive statistics for flexural strength of test specimens in six study groups. The mean of **Group A** was **53.76** MPa and the strength ranged between 50.05 to 59.13 MPa. For **Group B**, the mean flexural strength was **58.14** MPa and ranged between 51.75 to 69.75 MPa. In **Group C**, the mean flexural strength was **63.29** MPa and ranged between 55.40 to 69.33 MPa. For **Group D**, the mean flexural strength was **59.02** MPa and ranged between 54.29 to 67.50 MPa. A graphical visualization of mean strength along with error bar is given in Graph 1.

Table 2 reveals that using ANOVA F test, the mean flexural strength across groups differed significantly across four groups, as indicated by ***p*-value =0.001 (p <0.05)**.

In order to determine, which groups contributed to overall significance, a pair-wise comparison of mean strength was performed using Tukey's HSD *test*.

Table 3 shows individual pair-wise comparison of flexural strength between groups using Tukey's post-hoc test. It shows statistically significant difference **(p <0.05)** exist on comparison of flexural strength between **Group A** (control) and **Group B** (1wt% silanized zirconium oxide nanoparticles) **(p <0.001)**. Statistically significant difference **(p <0.05)** exist on comparison of flexural

strength between **Group A** (control) and **Group D** (5wt% silanized zirconium oxide nanoparticles) **(p = 0.004)**. It is to be noted that statistically significant difference **(p <0.05)** was found between **Group B** (1wt% silanized zirconium oxide nanoparticles) and **Group C** (2.5wt% silanized zirconium oxide nanoparticles) **(p = 0.005)** along with **Group C** (2.5wt% silanized zirconium oxide nanoparticle) and **Group D** (5wt% silanized zirconium oxide nanoparticles) **(p = 0.028)**. Statistically highly significant difference exist on comparison of flexural strength between **Group A** (control) and **Group C** (2.5wt% silanized zirconium oxide nanoparticles) **(p <0.001).** While, no statistical significant difference (p >0.05) was observed between **Group B** (1wt% silanized zirconium oxide nanoparticles) and **Group D** (5wt% silanized zirconium oxide nanoparticles) **(p = 0.935)**.

DISCUSSION

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

The success of dental research in last few decades has upheld our professional commitment to keep the dentition healthy and intact throughout life. Fixed prosthodontic treatment involves the restoration of compromised natural teeth with crowns or partially edentulous arches with a fixed dental prostheses or implant supported prostheses. During the tooth preparation procedure, much of the tooth structure is removed and it becomes comparatively smaller in size compromising the esthetics, masticatory efficiency and occlusal harmony.[1] Provisional fixed dental prostheses (FDPs) are fundamental part of fixed prosthodontics including tooth and implant supported restorations.[3]

Provisional restoration has its function for a limited period of time, after which it is to be replaced by a definitive prosthesis.[1] The time period depends upon

type of prosthetic treatment and varies considerably from case to case from few days to few months.

As stated by **Federick in 1975** [2],with regard to its significance in both periodontal prostheses and rehabilitation, the purposes for the provisional (treatment) restoration is to protect pulp and sedate prepared abutment, evaluate parallelism of abutments and immediately replace missing teeth, prevent migration of abutments, improve esthetics, provide a matrix for the retention of periodontal surgical dressings, stabilize mobile teeth during periodontal therapy, aid in developing and evaluating an occlusal scheme before the final prosthesis is made, allows evaluation of vertical dimension, phonetics, and masticatory function and aid in determining the prognosis of questionable abutments in the comprehensive restorative treatment plan.

Currently available provisional materials can be divided into four resin groups, namely, poly (methyl methacrylates), poly (R′ methacrylates), bis-acryl composite resins and visible light cured urethane dimethacrylates.[6] Polymethyl methacrylates are relatively inexpensive, with good color stability, excellent polishability and good marginal adaptation.[7]

Two forms of polymethyl methacrylate are available namely heat polymerized acrylic resin and auto polymerized acrylic resin.[8] Provisional restorations fabricated from heat-processed acrylic resin have been used successfully.[9-10]

Clinically provisional restorations are subjected to complex masticatory forces with a considerable amount of flexural stresses. In clinical situations, fixed partial dentures are subjected to various functional loads. However, in certain clinical

cases where there is increased parafunction, abnormal jaw relationships, cases of raised vertical dimension, long span bridges, forces acting on provisional restoration are far more than normal. Also, where provisional materials are used for extended periods of time like full mouth rehabilitation its strength assumes paramount importance.[1] The continuous cyclic stresses generated by masticatory forces creates the fatigue stress in the restoration which leads to fracture of the restoration.

Flexural strength serves as an indicator to assess if a provisional restorative material is strong enough to withstand such forces. It is a combination of tensile and compressive strength tests and includes the elements of proportional limit and elastic modulus measurements.[12]

There are three ways to improve the properties of PMMA viz., **development of an alternative materia**l to PMMA, the **chemical modification of PMMA** such as by the addition of a rubber graft copolymer and the **reinforcement of PMMA with other materials** such as glass fibres, carbon fibres and ultra-high modulus polyethylene.[59]

P.K Vallittu and DT Docent in 1999 [33] conducted a study on flexural properties of heat cure acrylic resin polymers reinforced with pre-impregnated unidirectional and woven glass fibres. They used continuous unidirectional and woven pre-impregnated glass fibre reinforcements (Stick and Stick Net) to reinforce heat cured denture base and auto-polymerizing denture base polymers and a temporary fixed partial denture polymer was also reinforced with the Stick reinforcement material. A 3-point bending test was used to measure transverse strength and flexural modulus and ultimate strain at fracture was calculated. The cross

section of the specimens was examined with SEM to evaluate degree of impregnation of fibres in polymer matrix. They found that the Stick reinforcement increased considerably transverse strength and flexural modulus of polymers. The Stick Net reinforcement increased strain at fracture of the polymers. Fibres of Stick and Stick Net were impregnated with the resin of polymer matrix.

In 1959, **Feynman** introduced the concept of nanotechnology. Since then, nanotechnology has been widely used in many applications, including medical sciences where it plays an important role in diagnosis, treatment and regenerative medicine. A nanomaterial is an object, in which at least one of its dimensions is at the nanometer scale (approximately 1 to 100 nm). Nanomaterials are categorized according to dimension – those with all 3 dimensions less than 100 nm [nanoparticles (Nps) and quantum dots]; those that have 2 dimensions less than 100 nm (nanotubes, nanofibers, and nanowires); and those that have one dimension less than 100 nm (thin films, layers, and coatings).[60] Recently, researchers have used nanofillers for reinforcement of denture base resins. Size, shape, surface area, concentration and dispersion of nanofillers into resin matrix all have an effect the mechanical properties of the filler/resin composite. Nanoparticles of Alumina, titanium (TiO_2), silver, zirconia (ZrO_2), gold, platinum, silicon dioxide (SiO_2) are among the fillers that have been incorporated to enhance the mechanical properties of denture base resins.[61]

Asopa et al.[62] 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**.**

A study by **Zuccari et al**[30] concluded that zirconium oxide particles reinforced provisional restorative resin exhibited the greatest improvements in modulus of elasticity, transverse strength, toughness and hardness. Therefore incorporation of ZrO_2 nanoparticles were selected for present study.

A study by **Ihab et al**[44] concluded that increase in the transverse strength occurred with addition of 2-5wt% ZrO_2 nanoparticles due to good distribution of the very fine size of nanoparticles. But increasing the percentage of modified nano-ZrO_2 to 7wt% lowered the impact strength and transverse strength due to agglomeration nano-ZrO_2. Hence ZrO_2 in the percentage of 1wt%, 2.5wt%, 5wt% percentages were selected in present study.

The inorganic filler particles usually display high surface energy because of hydrophilic ionic nature. But due to difference in surface energy, the hydrophobic polymer does not wet or interact with the filler particles.[63] Therefore it is important to modify the filler surface for better dispersion and improve surface wetting, thereby improving the physical properties of the composites.[64] Hence in this study, Zirconium oxide nanoparticles were treated with trimethoxysilylpropylmethacrylate (TMSPM) to improve adhesion of nanoparticles to the resin matrix.[52] The role of silanization has been postulated as an agent which improves bonding of pigment or fillers to resin, increased matrix strength, decreased water intake of resin component and reduction in polymerization shrinkage.[19]

According to present study the average values of flexural strength of heat polymerized acrylic resins is 53.76 ±2.97 MPa. The mean flexural strength obtained for Group A (control) was 53.76 ±2.97 MPa, Group B (1% Zirconium oxide) was 58.14 ±4.86 MPa), Group C (2.5% Zirconium oxide) was 63.29±4.22 MPa and Group D (5% Zirconium oxide) was 59.02±3.99 MPa. The maximum increase in the flexural strength was obtained when PMMA was reinforced with silanized 2.5% of zirconium oxide nanoparticles.

These results are similar to the study done by **Alhavaz A**[8] on untreated zirconia nanoparticles who concluded that highly significant increase in the flexural strength occurred with the incorporation of 2.5%wt zirconium oxide nano-filler than unreinforced controls. Explanation for enhanced flexural strength is by the phenomenon of interstitial filling of acrylic resin matrix with ZrO_2 which interrupts with the crack propagation [65.]

The decline in the flexural strength values above 2.5wt% concentration are in accordance with study by **Raouf L** who concluded that flexural strength decreases significantly above 3wt% of ZrO_2 nanoparticles concentration. Possible explanations for reduction in strength with increasing in percentage could be stress concentration as a result of too many filler particles and due to nanoparticles agglomeration.[66]

LIMITATIONS OF THE STUDY

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

1. In the oral cavity, reinforced provisional restorative resin is exposed to forces of varying magnitudes acting in different directions. The same situation could not be simulated in present in vitro study.

2. Scanning electron microscopy (SEM) examination of the samples to evaluate the adhesion of zirconium oxide nanoparticles to the surface of PMMA was not performed.

CLINICAL IMPLICATION

Within the limitations of this study, it becomes evident that reinforcement of the heat polymerized polymethyl methcrylate provisional restorative resin with silanized zirconium oxide nanoparticles increased the flexural strength of the provisional restorative material and thus, reduces the probability of occurrence of fracture. It is highly recommended to reinforce the provisional fixed restoration with 2.5wt% silanized zirconium oxide especially when long term provisional are given to the patient.

SCOPE FOR FURTHER STUDIES

1. Fatigue testing of these materials under dynamic loading using the provisional material 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 provisional restorative resin before clinical application.

3. Other physical and mechanical properties like thermal diffusivity, abrasion resistance, color stability and disinfectant property can be studied.

4. The heat polymerized provisional restorative resin can be reinforced with even further different sized nanoparticles and various physical and mechanical properties can be evaluated.

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

SUMMARY

The heat cure provisional restorative resins have been used successfully.[9] However, in certain clinical cases where there is increased parafunction, abnormal jaw relationships, cases of raised vertical dimension, long span bridges, forces acting on provisional restoration are far more than normal. Additionally provisional materials are used for extended periods of time like full mouth rehabilitation its strength assumes paramount importance.[1]

This study was conducted to evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate provisional restorative resin without reinforcement and reinforced with different percentages of silanized zirconium oxide nanoparticles. Standard heat cured acrylic resin specimens were fabricated according to ADA specification no. 12 and total 60 specimens were fabricated with 15 specimens in each group. Group A- The control group; heat polymerized polymethyl methacrylate provisional restorative resin specimens without reinforcement. Group

B,C,D- Heat polymerized polymethyl methacrylate provisional restorative resin specimens reinforced with 1wt%, 2.5wt%, 5wt% silanized zirconium oxide nanoparticles respectively.

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

Results showed that the mean flexural strength for Group A (control) was 53.76±2.97 MPa, Group B (1wt% silanized zirconium oxide Nps) was 58.14±4.86 MPa, Group C (2.5wt% silanized zirconium oxide) was 63.29±4.22 MPa and Group D (5wt% silanized zirconium oxide Nps) was 59.02±3.99 MPa.

The statistical analysis showed statistically significant difference **(p <0.05)** exist on comparison of flexural strength between **Group A** (control) and **Group B** (1wt% silanized zirconium oxide nanoparticles) **(p <0.001)**. Statistically significant difference **(p <0.05)** existed on comparison of flexural strength between **Group A** (control) and **Group D** (5wt% silanized zirconium oxide nanoparticles) **(p = 0.004)**. It is to be noted that statistically significant difference **(p <0.05)** was present between **Group B** (1wt% silanized zirconium oxide nanoparticles) and **Group C** (2.5wt% silanized zirconium oxide nanoparticles) **(p = 0.005)** along with **Group C** (2.5wt% silanized zirconium oxide nanoparticles) and **Group D** (5wt% silanized zirconium oxide nanoparticles) **(p = 0.028)**.Statistically highly significant difference exist on comparison of flexural strength between **Group A** (control) and **Group C** (2.5wt% silanized zirconium oxide nanoparticles) **(p <0.001).** While, no statistical significant difference (p >0.05) was observed between **Group B** (1wt% silanized zirconium

oxide nanoparticles) and **Group D** (5wt% silanized zirconium oxide nanoparticles) **(p = 0.935)**.

Thus amongst the various concentrations of silanized zirconium oxide tested in this study, reinforcement with 2.5wt% of silanized zirconium oxide Nps (Group C) showed statistically highly significant increase in the flexural strength as compared to unreinforced specimens (Group A).

CONCLUSION

Science is organized knowledge. Wisdom is organized life

-Immanuel Kant

Within the limitations of this study following conclusions were drawn:

- Reinforcement with silanized zirconium oxide nanoparticles increased the flexural strength of heat polymerized polymethyl methacrylate provisional restorative materials.

- The maximum increase was found with 2.5wt% concentration zirconium oxide nanoparticles (63.29±4.22 MPa) as compared to control (53.76±2.97 MPa) and 1wt% concentration (58.14±4.86 MPa).

- The flexural strength declined with increasing the zirconium oxide nanoparticles concentration to 5wt% (59.02±3.99 MPa).

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Table 1: Descriptive statistic of different percentages of silanized zirconium oxide nanoparticles on flexural strength of heat polymerized polymethyl methacrylate provisional fixed restorative resin

GROUPS	MEAN	Standard Deviation (S.D)	Standard error (S.E)	Minimum	Maximum
GROUP A (CONTROL)	53.76	2.97	0.76	50.05	59.13
GROUP B (1 % Zirconium oxide)	58.14	4.86	1.25	51.75	69.75
GROUP C (2.5% Zirconium oxide)	63.29	4.22	1.09	55.40	69.33
GROUP D (5% Zirconium oxide)	59.02	3.99	1.03	54.29	67.50

Table 2: Comparison of different percentages of silanized zirconium oxide nanoparticles on flexural strength of heat polymerized polymethyl methacrylate provisional fixed restorative resin using ANOVA F test

GROUPS	MEAN	Standard Deviation (S.D)	ANOVA F TEST	P value, Significance
GROUP A (CONTROL)	53.76	2.97	F = 13.816	p < 0.001**, Highly significant difference
GROUP B (1 % Zirconium oxide)	58.14	4.86		
GROUP C (2.5% Zirconium oxide)	63.29	4.22		
GROUP D (5% Zirconium oxide)	59.02	3.99		

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

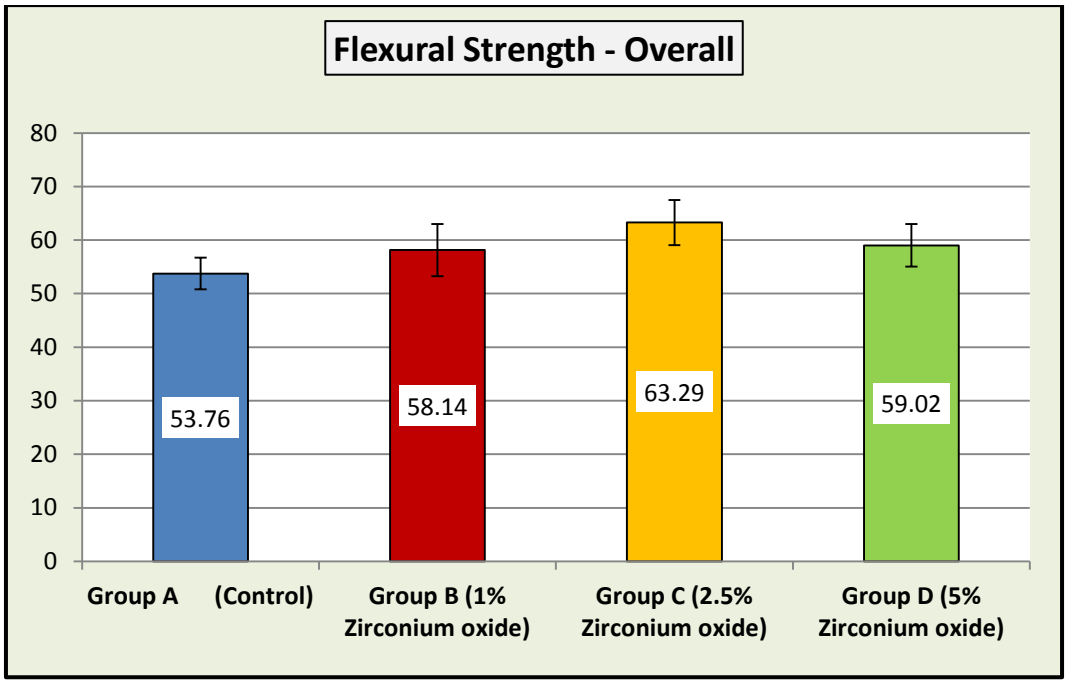
Table 3: Individual pair –wise comparison of different percentages of silanized zirconium oxide nanoparticles on flexural strength of heat polymerized polymethyl methacrylate provisional fixed restorative resin using Tukey's post – hoc test

Tukey's post hoc test to find individual pair wise comparison			
GROUP	**COMPARISON GROUP**	**MEAN DIFFERENCE**	**p value, Significance**
GROUP A (CONTROL)	**GROUP B (1 % Zirconium oxide)**	**4.37**	**p = 0.024***
	GROUP C (2.5% Zirconium oxide)	**9.52**	**p <0.001****
	GROUP D (5% Zirconium oxide)	**5.25**	**p = 0.004***
GROUP B (1 % Zirconium oxide)	**GROUP C (2.5% Zirconium oxide)**	**5.15**	**p = 0.005***
	GROUP D (5% Zirconium oxide)	**0.87**	**p = 0.935**
GROUP C (2.5% Zirconium oxide)	**GROUP D (5% Zirconium oxide)**	**4.27**	**p = 0.028***

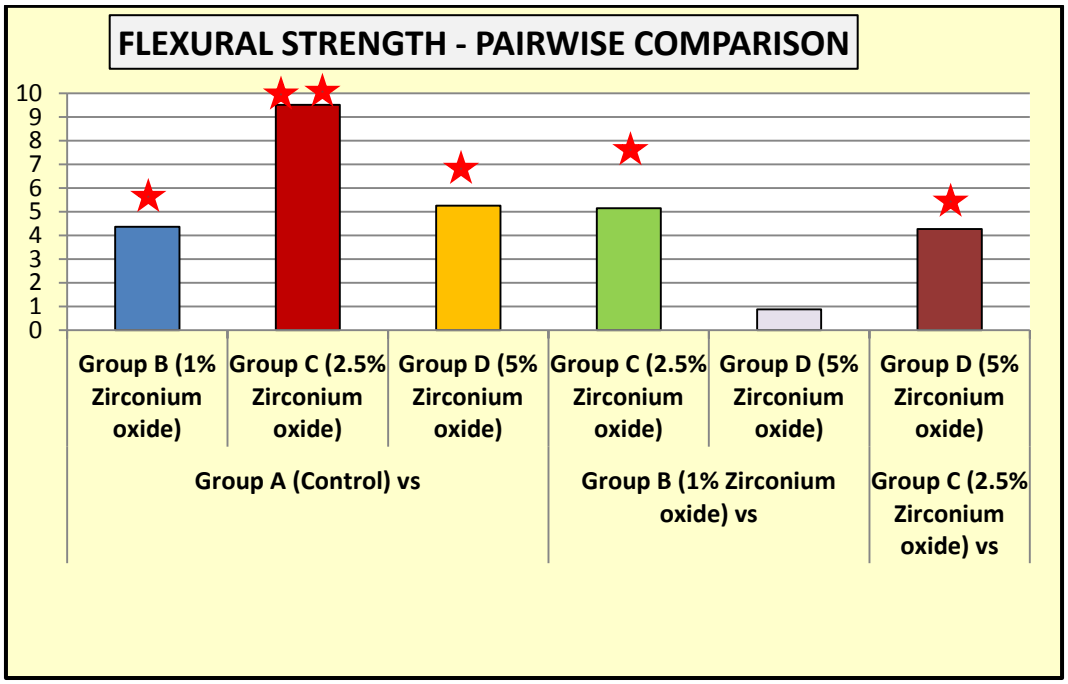
p >0.05 – not significant * p <0.05 – significant **p <0.001 – highly significant

GRAPHS

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



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



ANNEXURE

Group A : Control (Heat polymerized polymethyl methacrylate without any reinforcement)			**Group B : Heat polymerized polymethyl methacrylate reinforced with 1% Zirconium Oxide Nanoparticles**	
Sr. No	**Flexural Load (N)**	**Flexural Strength (MPa)**	**Flexural Load (N)**	**Flexural Strength (MPa)**
1	65.195	50.15	75.946	58.42
2	72.319	55.63	69.498	53.46
3	67.977	52.29	69.719	53.63
4	71.344	54.88	76.648	58.96
5	71.669	59.13	69.602	53.54
6	69.069	53.13	76.154	58.58
7	74.529	57.33	71.279	54.83
8	73.554	54.58	83.135	63.95
9	65.065	50.05	90.675	69.75
10	76.193	58.61	69.979	53.83
11	68.939	53.03	78.975	60.75
12	70.577	54.29	75.556	58.12
13	81.042	52.34	80.652	62.04
14	65.299	50.23	78.754	60.58
15	66.131	50.87	67.275	51.75
Mean		53.7693		58.146

Group C : Heat polymerized polymethyl methacrylate reinforced with 2.5wt% Zirconium Oxide Nanoparticles			**Group D: Heat polymerized polymethyl methacrylate reinforced with 5wt% Zirconium Oxide Nanoparticles**	
Sr. No	**Flexural Load (N)**	**Flexural Strength (MPa)**	**Flexural Load (N)**	**Flexural Strength (MPa)**
1	89.154	68.58	70.902	54.54
2	78.494	60.38	72.202	55.54
3	84.37	64.9	87.75	67.5
4	72.02	55.4	73.554	56.58
5	90.129	69.33	76.869	59.13
6	81.302	62.54	76.895	59.15
7	82.602	63.54	70.577	54.29
8	87.75	67.5	86.71	66.7
9	73.554	56.58	79.885	61.45
10	80.769	62.13	75.608	58.16
11	88.933	68.41	77.831	59.87
12	86.125	66.25	71.279	54.83
13	79.599	61.23	79.625	61.25
14	80.262	61.74	74.906	57.62
15	79.235	60.95	76.336	58.72
Mean		63.2973		59.022