

# Comparative Evaluation of Flexural Strength of Heat Polymerised Polymethyl Methacrylate Provisional Fixed Restorative Resin Reinforced with Different Percentages of Silanised Zirconium Oxide Nanoparticles: An In-vitro Study

PRIYA GUPTA<sup>1</sup>, SAAE DESHPANDE<sup>2</sup>, VAISHNAVI BANODE<sup>3</sup>

## ABSTRACT

**Introduction:** The purpose of providing a provisional restoration includes immediate replacement of missing teeth, for protection of pulp and maintenance of periodontal health, to maintain occlusal stability, to preserve the position of the teeth and for masticatory efficiency. Provisional restorations fabricated from heat processed acrylic resin have been used successfully. Incorporation of inorganic nanoparticles into Polymethyl Methacrylates (PMMA) resin has been shown to improve its physical properties.

**Aim:** To evaluate and compare the flexural strength of heat polymerised PMMA provisional restorative resin reinforced with different percentages of silanised zirconium oxide nanoparticles.

**Materials and Methods:** This is an in-vitro study performed in the Department of Prosthodontics, VSPM's Dental College and Research Centre, Nagpur, Maharashtra, India, from June 2018 to February 2020. A die was used to create sixty heat-cured PMMA resin specimens, which were then divided into four groups: group A includes controls with conventional heat

polymerised provisional restorative resin (PMMA) and groups B, C and D conventional heat polymerised provisional restorative resin reinforced with different percentages of silanised zirconium oxide nanoparticles (1 wt%, 2.5 wt%, 5 wt%, respectively), having fifteen specimens in each group. Three point bending tests were conducted on all samples using a universal testing machine. The flexural strength of each specimen was calculated. Mean value of flexural strength of each group was used for statistical analysis. One-way analysis of variance test was used to find out the mean value of flexural strength of each group.

**Results:** The mean flexural strength for control group was 53.76±2.97 MPa. For group B (1% Zirconium oxide) it was 58.14±4.86 MPa, for group C (2.5% Zirconium oxide) was 63.29±4.22 and for group D (5% Zirconium oxide) it was 59.02±3.99 MPa. Statistical analysis showed that maximum strength was obtained by reinforcement with 2.5% silanised zirconium oxide nanoparticles.

**Conclusion:** Polymethyl methacrylate reinforced with silanised 2.5% of zirconium oxide nanoparticles promises to be better material in terms of flexural strength.

**Keywords:** Acrylic resin, Fixed partial denture, Reinforcement of provisional restorations, Temporary restorations

## INTRODUCTION

The fundamental component of fixed prosthodontics, which includes tooth and implant supported restorations, is provisional Fixed Dental Prostheses (FDPs) [1]. The purpose of providing a provisional restoration includes immediate replacement of missing teeth, for protection of pulp and maintenance of periodontal health, to maintain occlusal stability, to preserve the position of the teeth and for masticatory efficiency [2]. Currently, available provisional materials can be divided into four groups namely- Polymethyl Methacrylate (PMMA), polyethyl methacrylates, bis-acryl composite resins and visible light cure resin [3]. PMMA are relatively inexpensive, with good colour stability, excellent polishability and good marginal adaptation [4]. Provisional restorations fabricated from heat processed PMMA have been used successfully [5,6].

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 [7]. The incorporation of

inorganic nanoparticles of titanium dioxide, zirconium oxide and silicon dioxide into PMMA has been shown to improve its physical properties [8-10]. The characteristics of polymers nanocomposites depend on the types of nanoparticles, their dimensions, as well as the concentration and interaction with polymer matrix [11]. Tetragonal zirconium oxide nanoparticle powder has been reported to improve the properties of PMMA, as it is a biocompatible material that possesses high fracture resistance, improves flexural strength and fracture toughness of resin denture base [12]. Zirconium Oxide (ZrO<sub>2</sub>) ceramic material is designated as 3Y-TZP i.e., 3 mol% Yttria stabilised Tetragonal Zirconium Polycrystal. It includes a variety of mechanical qualities, including good mechanical strength, fracture toughness, hardness, wear resistance, chemical resistance, good thermal stability and micro crack propagation during toughening [13,14]. The percentages 1%, 2.5% and 5% of zirconium oxide are selected as they are found effective in improving its flexural strength in auto-polymerised provisional acrylic resins [15].

Silanes can bond inorganic substances like metal and metal oxides to organic resins, improving mixing, improving bonding and boosting matrix strength [14]. Limited amount of data is available in literature regarding

the effect of silanisation as well as varying different percentages of zirconium oxide nanoparticles on flexural strength of heat polymerised PMMA [12,14]. Therefore, the aim of this study was to investigate and compare the flexural strength of heat polymerised PMMA provisional restorative resin reinforced with different percentages of silanised zirconium oxide nanoparticles. The null hypothesis was that there is no significant difference in the flexural strength between the groups.

## MATERIALS AND METHODS

This was an in-vitro study was carried out in the Department of Prosthodontics, VSPM's Dental College and Research Centre, Nagpur. It was approved by institutional ethics committee no. ECR/885/Inst/MH/2017 and study period was during June 2018-Feb 2020.

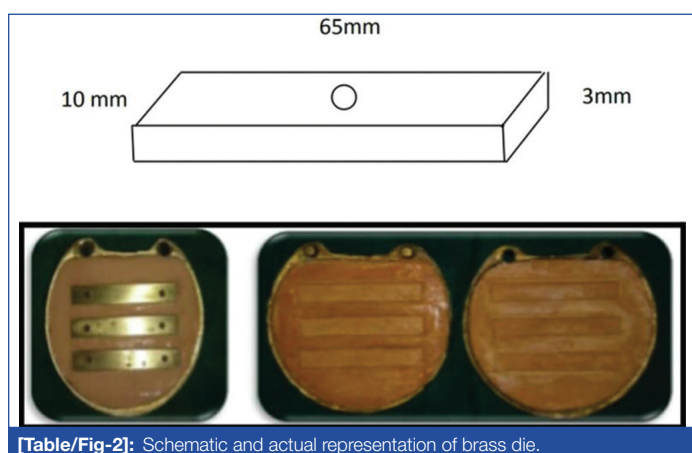
**Sample size calculation:** Sample size was calculated on OpenEPI calculator considering flexural strength as the main outcome measure. Mean±Standard Deviation (SD) in control group and experimental group=85.54±1.145 and 116.04±3.028, respectively [16], by keeping confidence 95% and power 80% and significance at p-value≤0.05. Formula for calculating sample size was  $n = \frac{(Z_{\alpha/2} + Z_{\beta})^2 \times (2(\sigma^2))}{(\mu_1 - \mu_2)^2}$ . There was one control and three experimental groups, n=15 (in each group). Hence, total samples were 60 specimens [Table/Fig-1].

Control group	Experimental groups		
Group A	Group B	Group C	Group D
Heat polymerised PMMA provisional restorative resin without nanoparticles	Heat polymerised PMMA provisional restorative resin + 1% wt silanised ZrO <sub>2</sub> Nanoparticles	Heat polymerised PMMA provisional restorative resin + 2.5% wt silanised ZrO <sub>2</sub> Nanoparticles	Heat polymerised PMMA provisional restorative resin + 5% wt silanised ZrO <sub>2</sub> nanoparticles
15 samples	15 samples	15 samples	15 samples
<b>Total- 60 samples</b>			

[Table/Fig-1]: Distribution of samples.

### Study Procedure

The materials used were heat polymerised acrylic resin, zirconium oxide nanoparticles, die stone, silane coupling agent, toluene and cold mould seal. Three brass metal dies of dimension 65 mm in length, 10 mm in width, and 3 mm in height [Table/Fig-2] were fabricated (ISO 1567 standard) [17]. Gypsum was moulded with uniform mould gaps, and sample replica blocks were fabricated.



[Table/Fig-2]: Schematic and actual representation of brass die.

For sample preparation of group B, group C and group D, the silanisation of metal oxide fillers is done as follows: in toluene solution, tetragonal zirconium oxide nanoparticles were mixed and then sonicated for 20 minutes after which the silane coupling agent i.e., Trimethoxysilylpropylmethacrylate (TMSPM) were added. The mixtures were stirred separately with magnetic stirrers for 30 minutes after which the toluene was completely evaporated using a vacuum rotary evaporator. The silanised metal oxide nanoparticles were well dispersed in monomer by weight of the polymer of heat polymerised

PMMA provisional restorative material with the help of an ultrasonicator until a homogeneous mixture is obtained [13]. The samples were created by combining heated PMMA powder with the appropriate modified and unmodified monomer, and processing was carried out in accordance with the manufacturer's instructions [Table/Fig-3].



[Table/Fig-3]: Test specimens of all the groups.

Each sample for measuring flexural strength was to be stored in distilled water at room temperature for one week before testing. Using a Universal testing device, a three-point bending test was performed on the samples at a 5.0 mm/minute crosshead speed [18]. At the point of fracture, the amount of force and deflection were recorded.

Flexural strength will be recorded using the following formula [19]:

$$FS = 3PI/2bd^2$$

Where, FS=Flexural strength (N/mm<sup>2</sup>), P=Load of fracture (N), b=Width of the sample (mm), d=Thickness of the sample (mm) and l=Distance between the supporting wedges (mm).

## STATISTICAL ANALYSIS

The statistical calculations were performed using the software Statistical Package for the Social Sciences (SPSS) for Windows (SPSS Inc. 1999, New York) software version 19.0. Descriptive statistics including mean and standard deviation were calculated. Statistical analysis using one-way Analysis of Variance (ANOVA) test and Tukey's post-hoc test were performed so as to facilitate interpretation of data. The p-value <0.05 was considered statistically significant.

## RESULTS

The mean flexural strength of group C (63.29 MPa) was the highest when compared to other groups [Table/Fig-4]. Statistical difference was found between the groups when ANOVA was applied (p-value<0.001). There was statistically significant difference (p-value=0.003) between flexural strength between group A and group B (p-value=0.00041). Statistically significant difference (p-value=0.02) existed between group A and group D (p-value=0.004). It is to be noted that statistically significant difference (p-value<0.021) was found between group B and group C (p-value=0.005) along with group C and group D (p-value=0.028). Statistically highly significant difference was found between group A and group C (p-value=0.0005). While, no statistically significant difference (p-value=0.04) was observed between group B and group D (p-value=0.935) [Table/Fig-5].

Groups	Mean	Standard deviation	Standard error	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/Fig-4]: Descriptive statistics of different percentages of silanised zirconium oxide nanoparticles on flexural strength of heat polymerised Polymethyl Methacrylate (PMMA) provisional fixed restorative resin in MPa.

## DISCUSSION

The null hypothesis was rejected as flexural strength of PMMA provisional fixed restorative resins reinforced with different percentages of silanised zirconium oxide nanoparticles was significantly different from the others. Asopa V et al., used

Tukey's post-hoc test to find individual pair-wise comparison.			
Group	Comparison group	Mean difference	p-value
Group A	Group B	4.37	<b>0.024</b>
	Group C	9.52	<b>0.001</b>
	Group D	5.25	<b>0.004</b>
Group B	Group C	5.15	<b>0.005</b>
	Group D	0.87	0.935
Group C	Group D	4.27	<b>0.028</b>

**[Table/Fig-5]:** Individual pair-wise comparison of silanised zirconium oxide nanoparticles on flexural strength of heat polymerised Polymethyl Methacrylate (PMMA) provisional fixed restorative resin using Tukey's post-hoc test. p-value in bold font indicates statistically significant values

zirconium oxide as a filler in the high impact acrylic resin resulted in increase in transverse strength as compared to the control group [20]. They stated that zirconium oxide, possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics. Addition of zirconia nanofillers to acrylic resin was found to improve mechanical properties [21]. In addition to that  $ZrO_2$  is known to have excellent biocompatibility and white colour which was less likely to alter esthetics. A study by Zuccari AG et al., concluded that the provisional restorative resin enhanced with zirconium oxide particles demonstrated the significant improvements in elasticity modulus, transverse strength, toughness and hardness [22].

A study by Ihab NS et al., concluded that increase in the transverse strength occurred with addition of 2-5 wt%  $ZrO_2$  nanoparticles due to good distribution of the very fine size of nanoparticles [12]. However, due to nano- $ZrO_2$  agglomeration, increasing the percentage of modified nano- $ZrO_2$  to 7 wt% decreased the impact strength and transverse strength. Hence,  $ZrO_2$  in the percentage of 1 wt%, 2.5 wt%, 5 wt% percentages were selected in the present study. The hydrophilic ionic nature of the inorganic filler particles typically causes them to display high surface energy. But due to difference in surface energy, the hydrophobic polymer does not wet or interact with the filler particles [23]. Therefore, it is important to modify the filler surface for better dispersion and improve surface wetting, thereby improving the physical properties of the composites [24]. Hence, in this study, zirconium oxide nanoparticles were treated with TMSPM to improve adhesion of nanoparticles to the resin matrix [25]. The role of silanisation has been postulated as an agent which increases resin matrix strength, decreases resin component water intake, and enhances the bonding of colour or fillers to resin and reduction in polymerisation shrinkage [14].

According to present study the average values of flexural strength of heat polymerised acrylic resins is  $53.76 \pm 2.97$  MPa. The mean flexural strength obtained for group A (control) was  $53.76 \pm 2.97$  MPa, group B was  $58.14 \pm 4.86$  MPa, group C was  $63.29 \pm 4.22$  MPa and group D was  $59.02 \pm 3.99$  MPa. The maximum increase in the flexural strength was obtained when PMMA was reinforced with silanised 2.5% of zirconium oxide nanoparticles.

These results are in accordance with the study done by Alhavaz A et al., on untreated zirconia nanoparticles who concluded that highly significant increase in the flexural strength occurred with the incorporation of 2.5%wt zirconium oxide nanofiller than unreinforced controls [2]. Explanation for enhanced flexural strength is by the incidence of interstitial  $ZrO_2$  filling in acrylic resin matrix, which interferes with fracture propagation [26]. The decline in the flexural strength values above 2.5 wt% concentration are in accordance with study by Raouf L et al., who concluded that flexural strength decreases significantly above 3 wt% 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 [27].

### Limitation(s)

Provisional restorations are exposed varying forces in different directions in the oral cavity. The same situation could not be simulated in the present in-vitro study. Scanning Electron Microscopy (SEM) examination of the samples to evaluate the adhesion of zirconium oxide nanoparticles to the surface of PMMA was not performed.

### CONCLUSION(S)

Within the constraints of this research, it may be stated that reinforcement with silanised zirconium oxide nanoparticles increased the flexural strength of heat polymerised PMMA provisional restorative materials. The maximum increase was found with 2.5 wt% concentration zirconium oxide nanoparticles. The flexural strength declined with increasing the zirconium oxide nanoparticles concentration to 5 wt%. It is highly recommended to reinforce the provisional fixed restoration with 2.5 wt% silanised zirconium oxide especially when long term provisional are given to the patient.

### REFERENCES

- Madhav V, Digholkar S, Palaskar J. Evaluation of the flexural strength and microhardness of provisional crown and bridge materials fabricated by different methods. *J Indian Prosthodont Soc.* 2016;16(4):328.
- Alhavaz A, Rezaei Dastjerdi M, Ghasemi A, Alizadeh Sahraei A. Effect of untreated zirconium oxide nanofiller on the flexural strength and surface hardness of autopolymerised interim fixed restoration resins. *J Esthet Restor Dent.* 2017;29(4):264-69.
- Vahidi F. The provisional restoration. *Dent Clin North Am.* 1987;31(3):363-81.
- Schillenburger HT, Hobo S, Whitsett LD, Bracklett SE. *Fundamentals of fixed prosthodontics.* 1997. 3rd ed. Chicago: Quintessence: 225-56.
- Burns D, Beck D, Nelson S. A review of selected dental literature on contemporary provisional fixed prosthodontic treatment: report of the committee on research in fixed prosthodontics of the academy of fixed prosthodontics. *J Prosthet Dent.* 2003;90(5):474-97.
- Davidoff S. Heat-processed acrylic resin provisional restorations: An in-office procedure. *J Prosthet Dent.* 1982;48(6):673-75.
- Binkley C, Irvin P. Reinforced heat-processed acrylic resin provisional restorations. *J Prosthet Dent.* 1987;57(6):689-93.
- Bangera MK, Kotian R, Ravishankar N. Effect of titanium dioxide nanoparticle reinforcement on flexural strength of denture base resin: A systematic review and meta-analysis. *Jpn Dent Sci Rev.* 2020;56(1):68-76.
- Al-Thobity AM, Gad MM. Effect of silicon dioxide nanoparticles on the flexural strength of heat-polymerised acrylic denture base material: a systematic review and meta-analysis. *Saudi dent J.* 2021;33(8):775-83.
- Aldegheshishem A, AlDeeb M, Al-Ahdal K, Helmi M, Alsagob EI. Influence of reinforcing agents on the mechanical properties of denture base resin: A systematic review. *Polymers.* 2021;13(18):3083.
- Dagar S, Pakhan A, Tunkiwal A. An in-vitro evaluation of flexural strength of direct and indirect provisionalization materials. *J Indian Prosthodont Soc.* 2005;5(3):132.
- Ihab NS, Hassanien KA, Ali NA. Assessment of zirconium oxide nano-fillers incorporation and silanisation on impact, tensile strength and colour alteration of heat polymerised acrylic resin. *J Bagh Coll Dentistry.* 2012;24(2):36-42.
- Saad-Eldeen MA, El-Fallah AA, El-Hawary Y. Effect of zirconium oxide reinforcement on epithelial oral mucosa, immunoglobulin and surface roughness of complete acrylic heat-cured denture. *Egyptian Dental Journal.* 2004;53(2):941-50.
- Goyal S. Silanes: Chemistry and applications. *J Indian Prosthodont Soc.* 2006;6(1):14.
- Wang RL, Moore BK, Goodacre CJ, Swartz ML, Andres CJ. A comparison of resins for fabricating provisional fixed restorations. *Int J Prosthodont.* 1989;2(2):173-84.
- Ebrahim M, Ashour Ahmed M, Felemban N. Effect of nanoparticles reinforced adhesive layers on microleakage of tooth restorations. *World Journal of Nano Science and Engineering.* 2016;06(02):64-69.
- Ellakwa AE, Morsy MA, El-Sheikh AM. Effect of aluminum oxide addition on the flexural strength and thermal diffusivity of heat-polymerised acrylic resin. *J Prosthodont.* 2008;17:439-44.
- Saritha MK, Shadakshari S, Nandeeshwar DB, Tewary S. An in vitro study to investigate the flexural strength of conventional heat polymerised denture base resin with addition of different percentage of aluminium oxide powder. *Asian J Med Clin Sci.* 2012;1:80-85.
- Vojdani M, Bagheri R, Khaledi AAR. Effects of aluminum oxide addition on the flexural strength, surface hardness, and roughness of heat-polymerised acrylic resin. *Journal of Dental Sciences.* 2012;7:238-44.
- Asopa V, Suresh S, Khandelwal M, Sharma V, Asopa SS, Kaira LS. A comparative evaluation of properties of zirconia reinforced high impact acrylic resin with that of high impact acrylic resin. *Saudi J Dental Res.* 2015;6(2):146-51.

- [21] Alshahrani FA. Effect of treated zirconium oxide (ZrO<sub>2</sub>) nanoparticles on the colour and surface properties of interim fixed prostheses. *J Int Soc Prevent Community Dent.* 2022;12:404-10.
- [22] Zuccari AG, Oshida Y, Moore BK. Reinforcement of acrylic resins for provisional fixed restorations. Part I: Mechanical properties. *Biomed Mater Eng.* 1997;7(5):327-43.
- [23] Asar NV, Albayrak H, Korkmaz T, Turkyilmaz I. Influence of various metal oxides on mechanical and physical properties of heat-cured polymethyl methacrylate denture base resins. *J Adv Prosthodont.* 2013;5:241-47.
- [24] Plueddemann EP. Silane coupling agents. Plenum: New York; 2 Ed,1991: 153.
- [25] Kareem S, Moudhaffer M. The effect of zirconium silicate nano powder reinforcement on some mechanical and physical properties of heat cured poly (methyl methacrylate) denture base materials. *J Bagh Coll Dentistry.* 2015;27(4):37-54.
- [26] Ahmed MA, Ebrahim MI. Effect of zirconium oxide nano-fillers addition on the flexural strength, fracture toughness, and hardness of heat-polymerised acrylic resin. *World J Nano Sci Eng.* 2014;4(2):50-57.
- [27] Raouf L, Faraj S, Azhdar B. Evaluation of flexural strength of heat cure PMMA denture base material reinforced with various concentrations of zirconium oxide. *Sulaimani Dent J.* 2019;6(2):22-30.

**PARTICULARS OF CONTRIBUTORS:**

1. Postgraduate, Department of Prosthodontics, VSPM Dental College and Research Centre, Nagpur, Maharashtra, India.
2. Postgraduate, Department of Prosthodontics, VSPM Dental College and Research Centre, Nagpur, Maharashtra, India.
3. Postgraduate, Department of Prosthodontics, VSPM Dental College and Research Centre, Nagpur, Maharashtra, India.

**NAME, ADDRESS, E-MAIL ID OF THE CORRESPONDING AUTHOR:**

Dr. Vaishnavi Banode,  
Postgraduate, Room No. 205, Department of Prosthodontics, VSPM Dental College and Research Centre, Hingna, Nagpur, Maharashtra, India.  
E-mail: vaishnaviangel06@gmail.com

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