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## **To determine the effects of various concentrations of nano zirconia particles on the tensile strength of poly methyl methacrylate material**

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**Abstract**--Objective: The aim of this study was to determine the tensile strength of Poly methyl methacrylate denture base material before and after addition of various concentrations of Nano-Zirconia. Study design: Experimental study, Laboratory based. Materials and Methods: 60 Dumb bell shaped specimens were prepared from heat polymerized acrylic resins and were divided into six groups. (n=10).

Zirconia nanoparticles size and shape was observed under TEM. Zirconia nanoparticles surfaces were treated with silane coupling agent and was observed under Fourier Transform Infrared Spectroscopy FTIR. Nano Zirconia particles were not added in the control group while test groups were reinforced with 1%,3%,7%,10%,12% Nano Zirconia. Tensile strength test was applied with the help of Tensile testing machine. With the help of Scanning electron microscopy fractured specimens were observed. 1-way ANOVA was used for statistical analysis and paired sample t-test for multiple comparison was applied using IBM-SPSS 20 ( $p < .0.05$ ). Results: Groups in which zirconium oxide nanoparticles are added exhibited an increase in tensile strength i.e 1%NZ, 3%NZ, 7%NZ, 10%NZ and 12%NZ by 45.67MPa, 51.59MPa, 55.77MPa, 59.69MPa and 67.70MPa compared with that of control group 49,67MPa respectively. Conclusion: Zirconium oxide nanoparticles that are added to PMMA denture base material increases its tensile forte.

**Keywords**---Zirconium oxide, nanoparticles, denture base.

## **Introduction**

In Prosthodontics the most commonly used denture base material for the manufacture of partial and full dentures is Poly methyl methacrylate (PMMA). Due to its unique properties PMMA is a very suitable biomaterial in various types of dental treatments due to its like ease of handling, manipulation, cost imp activeness and better esthetics.<sup>(1)</sup> Forte, which encompasses compressive and tensile fortes, hardness and dimensional stability is one of the most essential features of denture base resin.<sup>(2)</sup> Many recent clinical studies based on denture fracture proved that mechanical properties of PMMA were not satisfactory. Thus due to this reason PMMA with some modifications have been used.<sup>(3)</sup> One method is to include nanoparticles that can alter the characteristics of polymers due to their composition, size and shape.<sup>(4)</sup> Nanoparticles like silicon dioxide and diamond nanoparticles incorporated in PMMA has positive impact on mechanical properties.<sup>(4)</sup>

Among nanoparticles, Zirconia is a ceramic substance that resembles mineralized tissues like enamel, dentin and bone in color and has mechanical properties comparable to certain metals.<sup>(5)</sup> The flexural forte of PMMA denture base impregnated with Nano zirconia and aluminum borate whisker increased when the concentration of Nano zirconia gets increases from 1% to 2% but flexural forte gets decreased when the amount of Nano zirconia increased from 2% to 4%.<sup>(6)</sup> Very few studies have been done determining the addition of lower and greater concentration of Nano zirconia to PMMA material. So the motive of this survey is the determination of the various concentrations of Nano zirconia impregnated PMMA denture on the tensile forte of the denture.

## Materials and Methods

This was an Experimental Laboratory base study. The duration of data collection was three months from November 2021 to January 2022. Study setting was at University of Peshawar and Department of Dental Materials, Peshawar Dental College. The study was approved by the Institutional Review Board (Prime/IRB/2021-351). On the basis of (ISO 527-2) specifications, sample size was determined. 15g of Zirconia nanoparticles surfaces were treated with 0.2g of silane coupling agent (Microsil SILANO) and was observed under Fourier Transform Infrared spectroscopy (FTIR, Nicolet 6700, Thermo Fischer Scientific, Waltham, MA, USA). Dumbbell shaped specimens were prepared in the stainless steel mold (75×10×2). Melted wax was then placed in mold cavities which was then placed in denture flask having dental stone poured in it. Wax was then eliminated by pouring boiling water onto molds. In this way a total of 60 mold cavities (n=10) were prepared. Acrylic powder and liquid were mixed in ratio of 3:1. For experimental groups B,C,D,E and F both zirconia powder and PMMA powder was weighed with digital weighing machine. At doughy stage it was packed in flask and curing was performed. Specimens were then trimmed finished and polished. Specimens were stored at 37°C for 48 hours and then were evaluated with a Universal testing machine (Instron 8871; Instron Co, Norwood, MA, USA) for tensile strength at a cross head speed of 0.5mm/min, with a 5kN load cell and a chart speed of 20 mm/min. With the help of Scanning electron microscopy SEM fractured specimens were observed.

Table I Grouping of specimens and their specifications

Cluster	Descriptions	No.
Control group	“Unreinforced heat-polymerized acrylic resin”	10
1% Nano Zirconia	“1 weightage nano-ZrO <sub>2</sub> reinforced heat-polymerized acrylic resin”	10
3% Nano Zirconia	“3 weightage nano-ZrO <sub>2</sub> reinforced heat-polymerized acrylic resin”	10
7% Nano Zirconia	“7 weightage nano-ZrO <sub>2</sub> reinforced heat-polymerized acrylic resin”	10
10% Nano Zirconia	“10 weightage nano-ZrO <sub>2</sub> reinforced heat-polymerized acrylic resin”	10
12% Nano Zirconia	“12 weightage nano-ZrO <sub>2</sub> reinforced heat-polymerized acrylic resin”	10

Specimens were statistically analyzed by 1-way ANOVA using IBM-SPSS 20. Statistical test i.e paired sample t-test was applied. If the p value was less than or equal to 0.05 then the difference was observed to be significant.

## Results

Table II

Sample	Control (C)	1%NZ(V)	3%NZ(W)	7%NZ (X)	10%NZ (Y)	12%NZ (Z)
Maximum	53.60	48.01	53.30	59.90	63.79	74.18
Minimum	45.79	43.30	49.76	51.65	55.60	61.22
Mean $\pm$ SD	49.67 $\pm$ 2.03 <sup>A</sup>	45.67 $\pm$ 1.98 <sup>B</sup>	51.59 $\pm$ 2.39 <sup>C</sup>	55.77 $\pm$ 2.91 <sup>D</sup>	59.69 $\pm$ 1.87 <sup>E</sup>	67.70 $\pm$ 1.00 <sup>F</sup>
95% CI	47.64–51.70	43.99–47.35	49.20–53.98	52.86–58.68	57.82–61.56	66.70–68.70
<i>p</i> -value	–	versus C=0.002	versus C=0.001	versus C=0.001	versus C=0.000	versus C=0.012
	–		versus V=0.000	versus V=0.000	versus V=0.000	versus V=0.000
	–			versus W=0.000	versus W=0.000	versus W=0.000
	–				versus X=0.000	versus X=0.000
	–					versus Y=0.000

The experimental groups displayed noteworthy rise in tensile strength i.e 45.67 MPa, 51.59 MPa, 55.77 MPa, 59.69 MPa and 67.70 MPa for 1%NZ, 3%NZ, 7%NZ, 10%NZ and 12%NZ when associated with control cluster (49.67 MPa) respectively. Figure 1 shows TEM picture of Nano zirconia. The particles had a spherical shape with average size of 393nm.

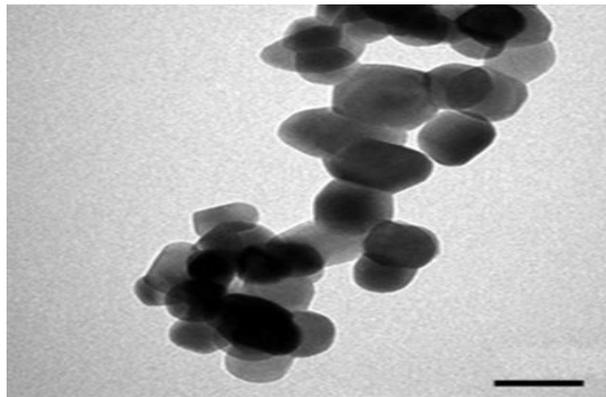


Figure 1: TEM image of Nano zirconia particle

Figure 2 depicts the images of Scanning electron microscopy of specimens that are fractured at 2000 power at a varied nano Zirconia concentrations.

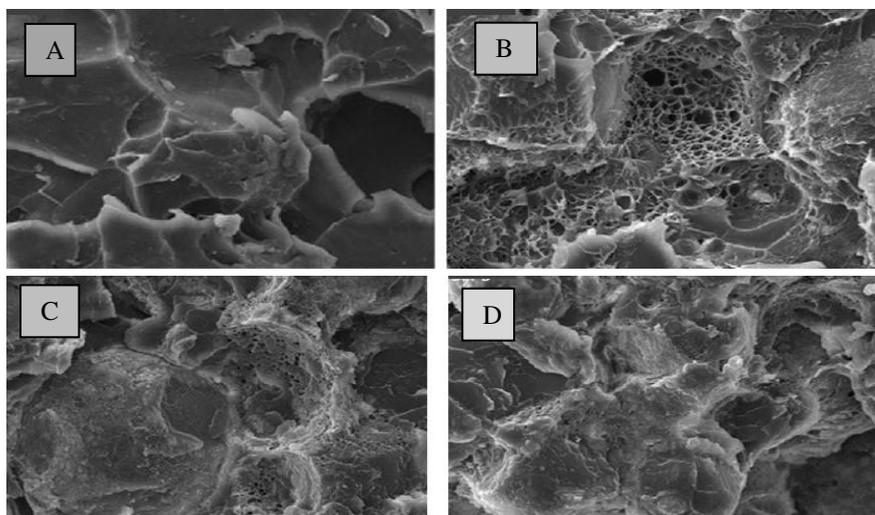


Figure 2: SEM images of fractured specimens

Figure 2A The control specimen shows sheet like assemblies and broad dispersed pores of  $50\mu\text{m}$ . Figure 2B 3%NZ group shows a dense structure with tiny pores occupied by some particles of zirconia. The tiny holes gets vanished in Figure 2D (12%Nano Zirconia). In 7%NZ, a visible stretched pattern was found.

The FTIR spectra are displayed in Figure 3. Specimens have 1%,3%,7%,10% and 12% nano zirconia filler as reinforcement. The principal PMMA resin characteristic bands and asymmetric stretching band of methyl ( $\text{CH}_3$ ) cluster ( $2918\text{cm}^{-1}$ ). The  $\text{CH}_3$  group's deformation modes ( $1447$  and  $1387\text{cm}^{-1}$ ), and  $\text{CH}_3$  twisting ( $1144\text{cm}^{-1}$ ). The spectra also showed in- and out-of-plane bending of  $\text{C}=\text{O}$  at  $750\text{cm}^{-1}$ . The spectra shows typical ester carbonyl ( $\text{C}=\text{O}$ ) stretching vibrations of PMMA. The typical PMMA bands' band positions were unaffected by the adding of nano- $\text{ZrO}_2$  particles to the PMMA matrix; instead, only the intensity of some bands changed. The PMMA/3% nano- $\text{ZrO}_2$  specimen showed the bands with the lowest peak intensities. The inclusion of nano- $\text{ZrO}_2$  nanoparticles in the PMMA matrix and their interface with PMMA molecules were both verified by the FTIR spectra.

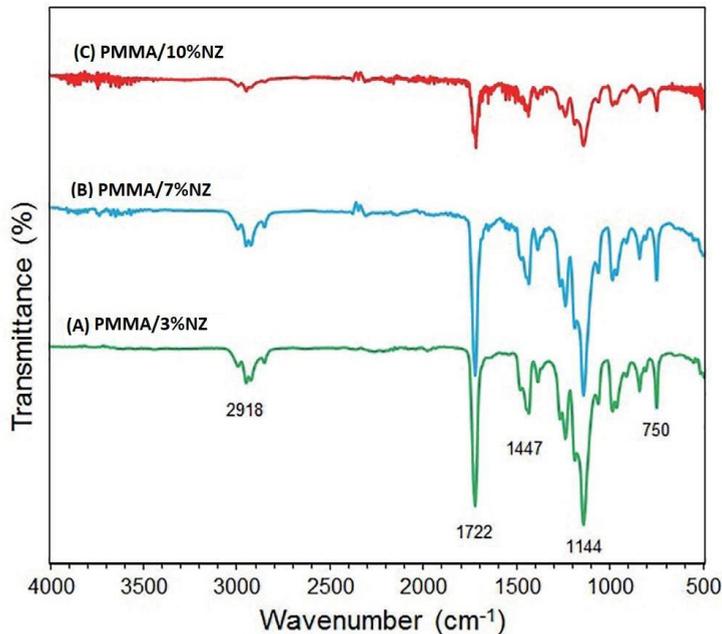


Figure 3: FTIR spectra of heat polymerized samples

## Discussion

The aim of the present investigation was to regulate how adding nano zirconia to PMMA material affected the tensile capabilities. Groups reinforced with nano zirconia concentrations had an increase in tensile forte. It is relatable to previous research that discovered a direct relationship amongst the addition of nano zirconia and increase in the properties.<sup>(7)</sup> Reinforcing filler that will be used must blend consistently with the matrix.<sup>(8)</sup> The coupling agent's adherence to PMMA matrix and nano Zirconia matrix boosts the mechanical characteristics of nanocomposites.<sup>(9)</sup>

In contrast to the outcomes of the current experiment, Chledek et al.(2012) disclosed that the addition of silver nanoparticles to the nanocomposites reduces the properties of these materials as the attention of these particles declined.<sup>(10)</sup> Thus Chatterjee established in 2010 that the tensile strength decreased as concentration of titanium oxide nanoparticles rose.<sup>(11)</sup>

In a related work by Argon et al, 2019, the adding of nano particles to traditional poly methyl methacrylate material generated greater roughness on the surface.<sup>(12)</sup> When aluminum oxide particles were added to PMMA denture bases, Vojdani et al, 2012 evaluated that 2.5% of  $\text{Al}_2\text{O}_3$  enhanced the flexural strength of PMMA compared to control group.<sup>(13)</sup> Study conducted by Zhang et al, 2014 demonstrated that addition of aluminium borate crystals to PMMA denture bases enhances the flexural strength of PMMA, while adding zirconia between 3% to 5% also enhances the flexural properties of PMMA.<sup>(14)</sup>

The linkage of strength associated to that of bond in the literature can not be related to this study due to differences in various properties, different techniques

of polymerization and different material used.<sup>(15)</sup> In our study the SEM micrographs with different nano zirconia concentrations showed acceptable surface characteristics. Microscopic images revealed that when the Nano Zirconia concentration increases, nanoparticles prevents cracks from spreading in the matrix, resulting in stronger material.<sup>(11)</sup>

Unlike our study, nanoparticles incorporated in PMMA denture base resin showed decline in flexural properties of PMMA.<sup>(16)</sup> Furthermore, in future there is still some work to be done in order to test the samples in a replicated oral environment following prosthesis manufacture.

## Conclusion

Despite some limitations related to this research, the accumulation of nano zirconia particles at concentrations of 1%,3%,7%,10% and 12% significantly rises the tensile strength of poly methyl methacrylate (PMMA) material. Increase in tensile forte of PMMA material was directly proportional to the concentration of zirconium oxide nanoparticles.

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