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Original research

Effect of zinc oxide nanoparticles on the flexural strength of polymethylmethacrylate denture base resin

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Purpose
This study evaluated the flexural strength of polymethyl methacrylate (PMMA) reinforced with various concentrations of zinc oxide (Zn O) nanoparticles

Materials and Methods
Nano ZnO was added in 0, 0.4, 0.6, 0.8, 1.2 and 1.4 percentage to PMMA denture base material. 60 specimens of heat cure polymerizing acrylic resin of dimensions 10mm x 4mm x 80mm were fabricated in accordance to ISO 20795-1-2013. The specimens were divided into 6 groups. Acrylic specimens were processed according to manufacturer's instruction. Three-point bending test was performed to evaluate the flexural strength. Surface analysis was performed with scanning electron microscopy (SEM) to observe the fracture surfaces of specimens. ANOVA and Tukey tests were used for the statistical analysis ($p < 0.05$).

Results
Statistical analysis revealed significant differences in strength between groups. The flexural strength improved with the addition ZnO nanoparticles. Highest mean value was observed in Group nZn -14 (91.31 MPa) and lowest in control Group nZn-0 (61.36 MPa). ANOVA and Tukey's honestly significance test found statistical significant differences among the groups ($p<0.001$).

Conclusion
The addition of ZnO nanoparticles in all concentrations increased the flexural strength of acrylic resin when compared to the control group.

Keywords: *Flexural strength; heat cure acrylic; nano particles; poly methyl metha acrylate; zinc oxide*

Introduction

Polymethyl methacrylate (PMMA) is the commonly used denture base material. It possesses a combination of favorable characteristics such as easy laboratory manipulation, light weight, inexpensive fabrication, stability in the oral environment, lack of toxicity and appropriate aesthetic and color matching ability (1,2). Limitations inherent in the resin are poor fatigue failure, high coefficient of thermal expansion, low thermal conductivity, dimensional inaccuracy, denture fracture and wear of the denture teeth (3,4). Clinicians encounter fracture of denture to low resistance to impact, flexural, or fatigue stresses (5). In order to prevent fracture of the dentures, the thickness of acrylic resin in susceptible regions was increased or reinforced (6). Copolymerization by rubber (7), reinforcement by incorporation of different forms like metallic wire (8), fibers (9-11) and the use of metallic oxides (12) were attempted to improve the properties of PMMA denture base resins.

Nanoparticles have been increasingly used in material science for its wear and tear resistance and anti-corrosion abilities. The alteration of filler

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size improves the properties of the material (13). The earlier studies conducted showed a marginal improvement of flexural strength but had several shortcomings in fatigue life, fatigue crack, propagation resistance and long term wear that restricted immediate clinical application (14-17). The search exists with more ideal reinforcement. The nano materials and technology provides wider opportunity in identifying the better reinforcement material.

Nano zinc oxide (ZnO) has excellent antibacterial, antifungal properties. ZnO in blending with denture base resins can improve the properties of denture base resins, significantly the biological properties of acrylic resins. This study was done with the objective to evaluate the flexural strength of PMMA with various concentrations of Zn O nanoparticles.

Material and Methods

A total of 60 heat cure acrylic denture base specimens (DPI Heat Cure, India) with dimension of 65mm x 40mm x 5mm were fabricated according to manufacturer's recommendations. The specimens were divided into six groups for flexural strength evaluation (n=10). Sample distribution and composition of material listed in Table 1.

The heat cure acrylic resin (DPI Heat Cure, India) with liquid monomer and polymer powder of 0.10mm particle was used for specimen fabrication. ZnO nanoparticles of 12 nm particles were procured from external source (Zigma Elhrich). Zinc oxide nanoparticles are incorporated into the heat cure polymer by twin screw extruder. Specimen groups were fabricated with 0.4 % (group nZn4), 0.6% (group nZn6), 0.8% (group nZn8), 1.2% (group nZn12) and 1.4% (group nZn14) nano ZnO by weight. The specimens were fabricated by mixing the nano ZnO with monomer in ratio of 25g/10ml.

Table 1: *Sample distribution*

Group	Sample	ZnO nanoparticles Conc.
nZn0	10	-
nZn4	10	0.4
nZn6	10	0.6
nZn8	10	0.8
nZn12	10	1.2
nZn14	10	1.4

Initially a master die was prepared according to ISO 20795.1.2013 with dimension of 65mm x 40mm x 5mm. The master die was duplicated with addition silicone impression material and resin specimen were fabricated with the specified dimensions by compression molding technique, processed by long heat cure polymerization cycle, trimmed with acrylic stone and finished with 600 grit sandpaper [Fig 1]. Each prepared specimen was cut lengthways with milling machine into three equal strips, 64 mm long, (10,0 ± 0,2) mm wide, and (3,3 ± 0,2) mm in height. The samples were subjected to a three-point bending test in universal testing machine (Autograph universal testing machine, Shimadzu Corp, Japan). The flexural strength was tested by applying a load until fracture at the midpoint of specimen by means of a hardened steel cylinder with a cross head of 1mm/min. The flexural strength in MPa was calculated using the equation, $M = 3Wl/2bd^2$ Where M = flexural strength (MPa), W = fracture load (N), l = test span (center to center) distance between support points (mm), B = width of specimen (mm) and d = thickness of the specimen (mm). The mean flexural strength of each group was calculated, tabulated and statistically analyzed with ANOVA and Tukey HSD test.

Results

The mean flexural strength of specimen ispresented in Table 2. Group nZn0 control group showed lesser strength of 61.3 MPa and Group nZn14 – 91.31 MPa was the highest when compared to other groups. The flexural strength increased with the concentration of ZnO. Group nZn-4 to Group nZn-14 exhibited increase in strength of 71.73 MPa,77.05 MPa, 84.98 MPa, 86.92 MPa and 91.31 MPa. The data analysis was executed using statistical software SPSS Version 20.0 (SPSS Inc., Chicago, IL). ANOVA displayed statistically significant differences among the 6 groups ($p<0.5$). The Post hoc test multiple comparisons Tukey's HSD revealed significant differences. The scanning electron microscope (SEM) revealed the distribution of the nanoparticles in PMMA and the fracture of the material occurred in the midst of the nano particles (Figure 1-6).

Table 2: *Descriptive Analysis on flexural strength*

Groups	N	Mean	SD	Standard Error	95% Confidence Interval for Mean		Minimum	Maximum	P value
					Lower Bound	Upper Bound			
nZn0	10	61.36	4.91	1.55	57.85	64.88	52.48	67.38	0.000*
nZn4	10	71.73	3.49	1.10	69.23	74.22	66.49	77.52	0.000*
nZn6	10	77.05	2.41	0.76	75.33	78.77	73.36	81.22	0.000*
nZn8	10	84.98	2.49	0.79	83.57	86.77	80.79	88.02	0.000*
nZn12	10	86.92	1.89	0.59	85.57	88.28	83.16	89.32	0.000*
nZn14	10	91.31	1.15	0.36	90.48	92.13	89.59	92.68	0.000*
Total	60	78.89	10.62	1.37	76.15	81.64	52.48	92.68	

*Oneway p < 0.001 (99.9 % sig), SD: Standard Deviation

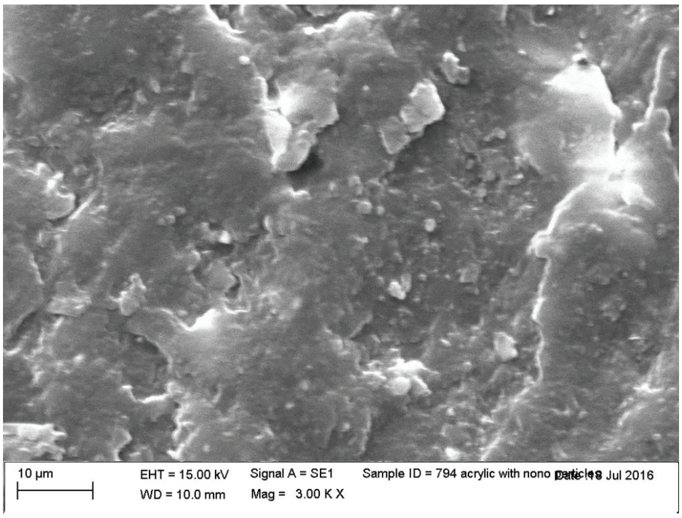

Figure 1. *SEM of heat cure acrylic with no reinforcement.*

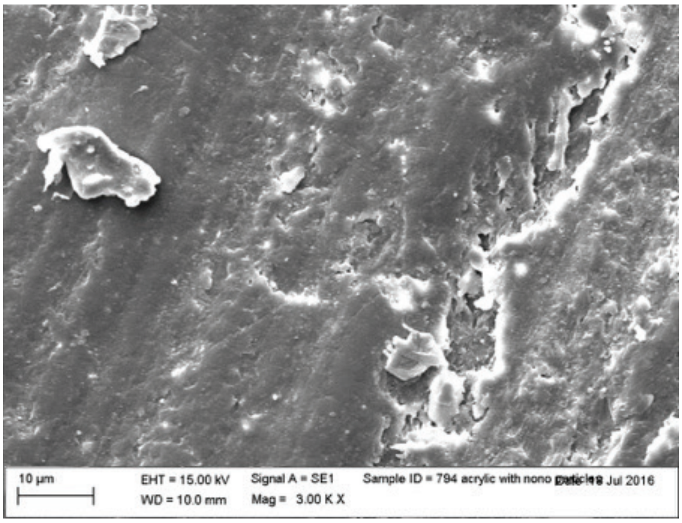

Figure 2. *SEM of n Zn 4 nanoparticles reinforcement specimen.*

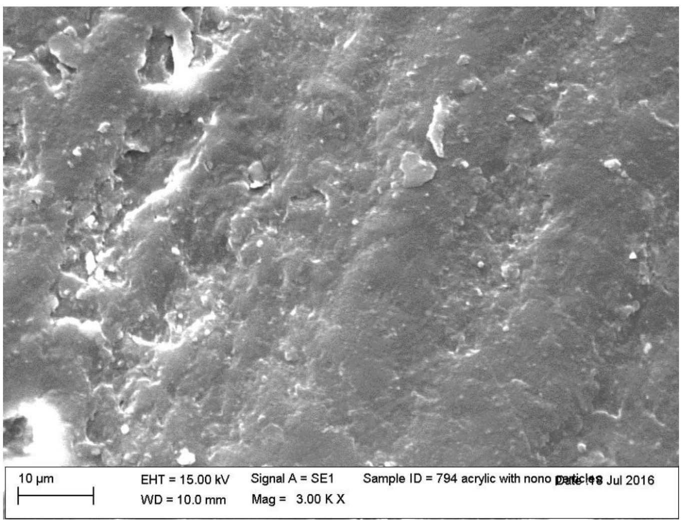

Figure 3. *SEM of n Zn 6 nanoparticles reinforcement specimen.*

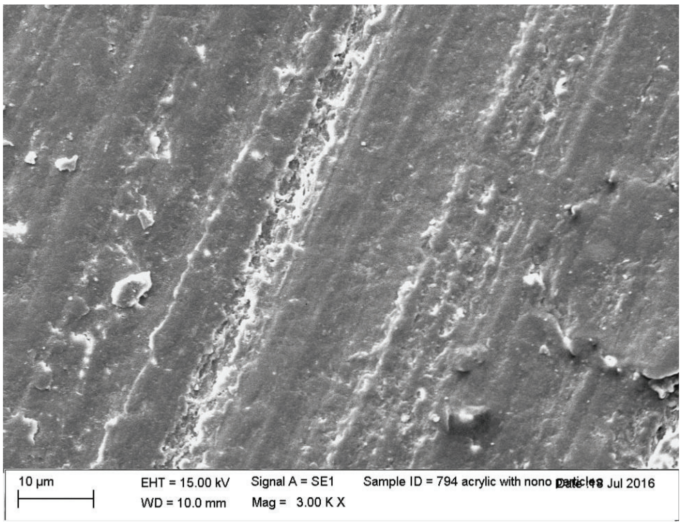

Figure 4. *SEM of n Zn8 nanoparticles reinforcement specimen.*

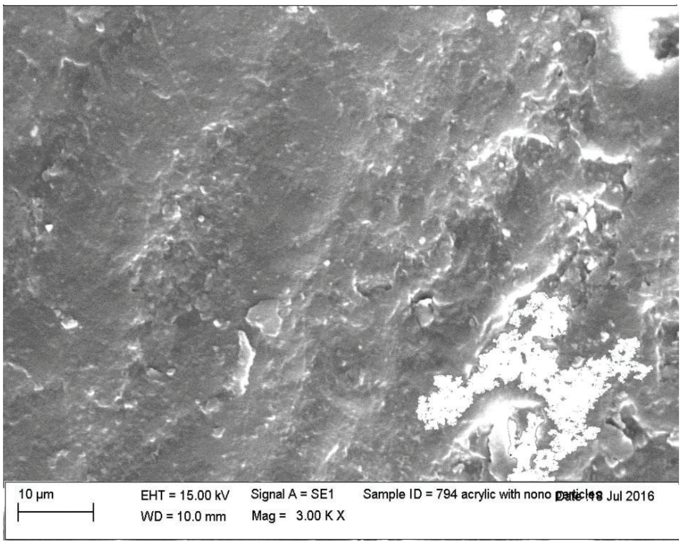

Figure 5. *SEM of nZn12 nanoparticles reinforcement specimen.*

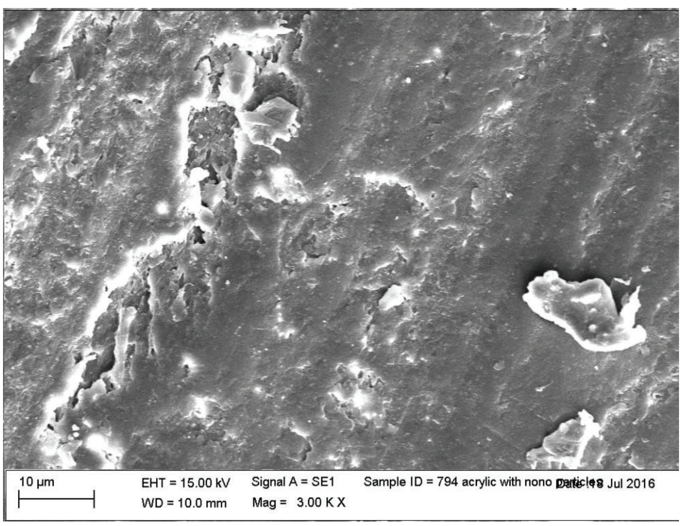

Figure 6. *SEM of nZn14 nanoparticles reinforcement specimen.*

Discussion

The flexural strength of PMMA denture base resins improved with the concentrations of nano Zn0. Rahim et al (18) established that the addition of metal nanoparticles increases the surface hydrophobicity and reduce the agglomeration of biomolecules. The studies on aluminum dioxide (19), cobalt-chromium (20), silver (21), zinc oxide (22), zirconia (23), titanium dioxide (24) nano particles have improved the flexural strength and documented the theory of surface hydrophobicity and decreased molecular agglomeration (25).

Nano particles are considered over macroscopic materials for their higher surface to volume ratios and an increased percentage of atoms at the grain boundary. The nano particles reduces the filler size increases the compaction of materials improves the mechanical properties of materials (15-17). Various nanoscale fillers, including silica, calcium carbonate, and metal oxides when added to dental -polymer matrix improved the properties. Nano-sized ZnO fillers was considered because of the unique physical properties, low cost and extensive applications in diverse areas (26-28). Xie et al. (27) observed the antibacterial properties of ZnO nanoparticles. Studies have indicated that ZnO nanoparticles at a concentration of between 3 and 10mM caused 100% inhibition of bacterial growth. Additionally, ZnO has superior biocompatibility properties and less likely to alter esthetics of denture base. The percentages of ZnO nano particles analyzed had effective antibacterial effect, obtained from the studies of Xie et al (27), Raj et al. (29).

The polymerization reaction is significant in determining the mechanical properties of denture base resin. The availability and generation of free radicals, the control of temperature during polymerization are some of the significant factors that influence the properties of the material. The compression molding technique and long curing polymerization cycle enabled to obtain to optimize the procedure and aided in obtaining the superior flexural strength.

The nanoparticles where incorporated by twin stage extruder. It aided is better dispersion in polymer matrix and homogenous distribution of the particles. The addition of nano particles to resin matrix is significant in improving the properties. The technique adapted aided in better distribution and it is visualized in SEM (28).

The distribution and dissolving of nano ZnO in PMMA aided in obtaining improved strength properties. Earlies studies on different nanoparticles emphasized on the need importance of homogenous distribution of particles for improved strength. Additionally, the particles should have displayed improved wettability with PMMA monomer. The SEM images displayed uniform distribution and no voids were observed. The images confirmed the blending of materials that improved the strength of the materials.

The study followed stringent testing protocol. Fewer limitations were unavoidable in the testing set up. In future, different concentrations of particles, size of particles, custom made nano particles, other forms of nano rods, tubes, polymerization techniques, types of PMMA resins can be evaluated. More studies are required to evaluate thermal properties , impact strength, mechanical, physical, antifungal and antibacterial spectrum for better interpretation.

Conclusion

Within the limitation of this study it can be concluded that the flexural strength of PMMA denture base increased with the addition ZnO nano particle to the PMMA denture base.

Türkçe Öz: *Çinko Oksit nanopartiküllerinin Polimetilmetakrilat protez kaidelerinin bükülme direncine etkisi. Amaç: Bu çalışmada, farklı konsantrasyonlarda çinko oksit (Zn O) nanopartkülleriyle güçlendirilmiş polimetilmetakrilatın (PMMA) bükülme direnci değerlendirilmiştir. Gereç ve yöntem: Nano Zn O yüzde 0, 0.4, 0.6, 0.8, 1.2 ve 1.4 oranlarda PMMA protez kadie materyaline ilave edilmiştir. ISO 20795-1-2013 standardına uygun olarak 10mm x 4mm x 80mm boyutlarında 60 adet ısı ile polimerize olan akrilik örnek hazırlanmıştır. Örnekler, 6 gruba bölünmüştür. Akrilik örnekler üreticinin önerileri doğrultusunda hazırlanmıştır. Bükülme direncinin ölçümü için 3 nokta eğme testi uygulanmıştır. Örneklerin yüzeylerindeki kırılmaları tespit etmek için yüzey analizi scanning electron microscobu(SEM) ile gerçekleştirilmiştir. İstatistiksel analiz için ANOVA and Tukey testleri kullanılmıştır (p<0.05). Bulgular: İstatistiksel analiz gruplar arasında anlamlı değişiklikler göstermiştir. Zn O nanopartküllerinin ilavesi bükülme dayanuımını arttırmıştır. En yüksek ortalama değer nZn -14 (91.31 MPa) ve en düşük değer control grubu nZn-0 (61.36 MPa) de bulunmuştur. ANOVA and Tukey testleri gruplar arası farklılıklar tespit etmiştir (p<0.001). Sonuç: Zn O nanopartküllerinin ilavesi her konsantrasyonda control grubuna göre akrilik rezinin bükülme direncini arttırmıştır. Klinik sonuç: PMMA'ın çinko oksit nanopartkülleriyle güçlendirilmesi protezlerin bükülme direncini arttırabilir. Anahtar kelimeler: Bükülme dayanımı; ısı ile polimerize olan akrilik; nanopartkül; polimetilmetakrilat; çinko oksit*

Ethics Committee Approval: Not required.

Informed Consent: Not required.

Peer-review: Externally peer-reviewed.

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Conflict of Interest: The author had no conflict of interest to declare.

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