

Original Article

# Effect of Zirconium Oxide Nano-Fillers Addition on Transverse Strength And Impact Strength of Heat-Polymerized Acrylic Resin, An in Vitro Study.

Mohamed I. Ebrahim<sup>1</sup>, Alaa N. Syam<sup>2</sup>, Shoukry Gamal<sup>3</sup>

<sup>1</sup> Department of Dental Biomaterials, Faculty of Dental Medicine Al Azhar University, Cairo(boys)

<sup>2</sup> Department of Dental Biomaterials, Faculty of Dental Medicine Al Azhar University, Assuit

<sup>3</sup> Department of Pedodontics, Faculty of Dental Medicine Al Azhar University, Assuit

E-mail: alaasyam@yahoo.com

Received 13-6-2019

Accepted For Publishing 9-7-2019

## Abstract

**Aim:** The purpose of this study was to evaluate the effect of Zirconium oxide ( $ZrO_2$ ) nanofillers powder with different concentration (1.5%, 3%, 5% and 7%) on the transverse strength and impact strength of heat-polymerized acrylic resin. **Materials and Methods:** Zirconium oxide powders with different concentrations (1.5%, 3%, 5% and 7%) were incorporated into heat-cure acrylic resin (PMMA) and processed with optimal condition (2.5:1 Powder/monomer ratio, conventional packing method and water bath curing for 2 hours at 95°C) to fabricate test specimens of PMMA of dimensions (65 x 10 x 2.5mm) for the transverse strength and (60 x 7 x 4mm) for impact strength. PMMA without additives was prepared as a test control. Two types of mechanical tests; transverse strength and impact strength were carried out on the samples. The recorded values transverse strength in (MPa) and impact strength in (J) were collected, tabulated and statistically analyzed. One way analysis of variance (ANOVA) and Tukey's tests were used for testing the significance between the means of tested groups which are statistically significant when the P value  $\leq 0.05$ .

**Results:** Addition of Zirconium oxide nanofillers to PMMA significantly increased the transverse strength and impact strength **Conclusion:** Zirconium oxide nanofillers added to PMMA has a potential as a reliable denture base material with increased transverse strength and impact strength. According to the results of the present study, the best mechanical properties were achieved by adding 7% wt  $ZrO_2$  concentration.

**Keywords:** Zirconium Oxide; polymethyl methacrylate; Impact Strength

## 1. Introduction

Acrylic resin polymethyl methacrylate (PMMA) has been the most popular material for the construction of dentures for many decades as it has many advantages such as good aesthetics, accurate fit, stability in the oral

environment, easy laboratory and clinical manipulation, and inexpensive equipment's [1]. Although it is the most widely used in dentistry for fabrication of denture bases, this material is still insufficient to fulfill the perfect mechanical requirements for dental applications. This issue was attributed mainly to its low fracture resistance and plaque accumulation [2] [3]. In a survey

to compare ten types of denture base resins it was found that nearly 70% of dentures had broken within the first 3 years of their delivery [2]. In a study evaluating the denture fracture, it was reported that 33% of the repairs were due to debonded/detached teeth, 29% of the repairs were because of midline fractures which were more commonly seen in the upper dentures and the rest were other types of fracture.

In another study the authors reported that the Mandibular partial denture was the most commonly needing repair [4]. So, the measuring of mechanical properties of the denture base materials is important to evaluate the effect of adding different strengthening materials [5].

Undoubtedly that, many trails were made to enhance mechanical properties of denture base materials either by adding chemical solutions such as a polyfunctional cross linking agent (polyethylene glycol dimethacrylate) [6] or by incorporating a rubber phase [7], metal fram [8], metal oxides [9], or fibers [10]. Despite these efforts to improve the fracture resistance of PMMA few have obtained promising results [11] [12]. The reinforcement of polymers used in dentistry with metal-composite systems has been a prime interest [12].

Zirconium oxide nano-particles powder has been selected to improve the properties of PMMA, as a bio-compatible material that possesses high fracture resistance, and to improve fracture toughness of ceramics by developing a new generation of ceramic-matrix composites [13] [14].

Since only limited amount of data regarding the effect of metal oxides on heat-cured PMMA are available in the literature, the purpose of this study was to investigate the influence of addition of metal oxides [zirconium oxide powder ( $ZrO_2$ )] on some mechanical properties of heat cured PMMA.

## 2. Subjects and Methods

An in vitro study was conducted to evaluate the effect of Zirconium oxide nanofillers powder ( $ZrO_2$ ) (5 - 15 nm) with different concentration (1.5%, 3%, 5% and 7%) on transverse strength and impact strength of heat-polymerized acrylic resin.

One type of heat-cure acrylic resin (PMMA) was used as the control (Acrostone (A), Anglo-Egyptian Company, Hegaz, Cairo, Egypt, Batch No.505/04), Zirconium oxide nanofillers powder ( $ZrO_2$ ) (Sigma-Aldrich Germany, Trade 544,760) with different concentrations (1.5%, 3%,

5% and 7%) was added into heat-cure acrylic resin (PMMA) and processed with optimal condition (2.5:1 Powder/monomer ratio, conventional packing method and water bath curing for 2 hours at 95°C) 100 bar shapes specimens were prepared to be used in this study. 50 specimens were used for each test [transverse strength (group A) and impact strength (group B)].

### Grouping of the specimens:

Each group was further divided into five subgroups (1, 2, 3, 4 and 5) of 10 specimens each as shown in Table 1.

### Transverse strength testing:

The transverse strength test (TS) was conducted according to International Standards Organization specification 1567 for denture base polymers [15]. Rectangular-shaped specimens (65 x 10 x 2.5) were prepared. Testing was conducted under 3-point loading, with a crosshead speed of 5mm/min. using a Lloyd universal testing machine (model LRX plus II, Fareham, England).

### Impact strength testing

Rectangular-shaped specimens (60 x 7 x 4 mm) were prepared for impact strength testing (IS). Strength test method and specimens dimensions were similar to those used by Uzun et al [16]. Using a notch cutter (Hounsfield notching machine, Tensometer Ltd., Croydon, U.K.), a 3.5 mm notch was prepared in each specimen. A Charpy-type impact tester (Hounsfield plastic impact machine, Tensometer Ltd.) was used to apply force to the specimens from the un-notched side. During testing those specimens that did not fracture at the first trial were excluded from the study.

The recorded values of transverse strength and impact strength were collected, tabulated and statistically analyzed. One way analysis of variance (ANOVA) and Tukey's tests were used for testing the significance between the means of tested groups which are statistically significant when the P value  $\leq 0.05$ .

## 3. Results

### Transverse strength

Both Table 2 and Figure 1 show a comparison between mean transverse strength in (MPa) of the tested groups of PMMA. ANOVA test showed statistically significant difference between all groups. PMMA specimen with 7% zirconium oxide nanofillers ( $ZrO_2$ ) (group A5) showed significantly highest mean transverse strength followed by

Groups	Subgroups	Description	No. of Specimens
<b>Group A</b>	Group A1	Heat-cure acrylic resin (PMMA) without additives as control.	10 specimens
	Group A2	PMMA with 1.5% zirconium oxide nanofillers powder (ZrO <sub>2</sub> ).	10 specimens
	Group A3	PMMA with 3% ZrO <sub>2</sub> .	10 specimens
	Group A4	PMMA with 5% ZrO <sub>2</sub> .	10 specimens
	Group A5	PMMA with 7% ZrO <sub>2</sub> .	10 specimens
<b>Group B</b>	Group B1	Heat-cure acrylic resin (PMMA) without additives as control.	10 specimens
	Group B 2	PMMA with 1.5% zirconium oxide nanofillers powder (ZrO <sub>2</sub> ).	10 specimens
	Group B 3	PMMA with 3% ZrO <sub>2</sub> .	10 specimens
	Group B 4	PMMA with 5% ZrO <sub>2</sub> .	10 specimens
	Group B 5	PMMA with 7% ZrO <sub>2</sub> .	10 specimens
<b>Total</b>			<b>100 specimens</b>

**Table 1:** Classification and grouping of the specimens.

<b>Group A1</b> Control group		<b>Group A2</b> (1.5% ZrO <sub>2</sub> )		<b>Group A3</b> (3% ZrO <sub>2</sub> )		<b>Group A4</b> (5% ZrO <sub>2</sub> )		<b>Group A5</b> (7% ZrO <sub>2</sub> )		<b>P-value</b>
Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
79.1 <sup>e</sup>	1.148	86.58 <sup>d</sup>	3.42	95.03 <sup>c</sup>	3.67	101.8 <sup>b</sup>	3.076	128.6 <sup>a</sup>	1.96	0.000*

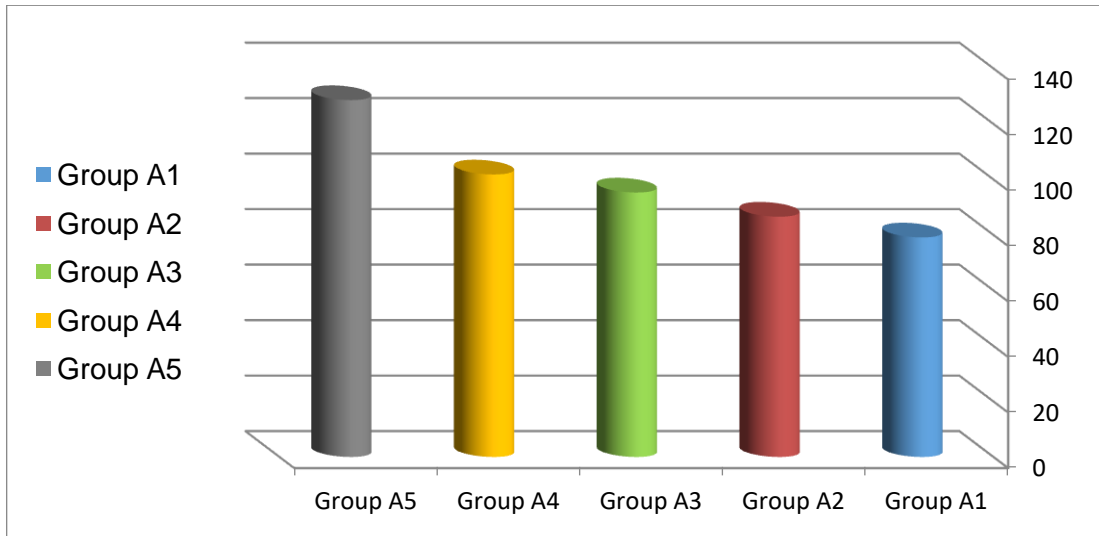
\*Significant at  $P \leq 0.05$ , Means with different letters are significantly different according to Tukey's test.

**Table 2:** Comparison between mean transverse strength (MPa) of the tested groups of PMMA.

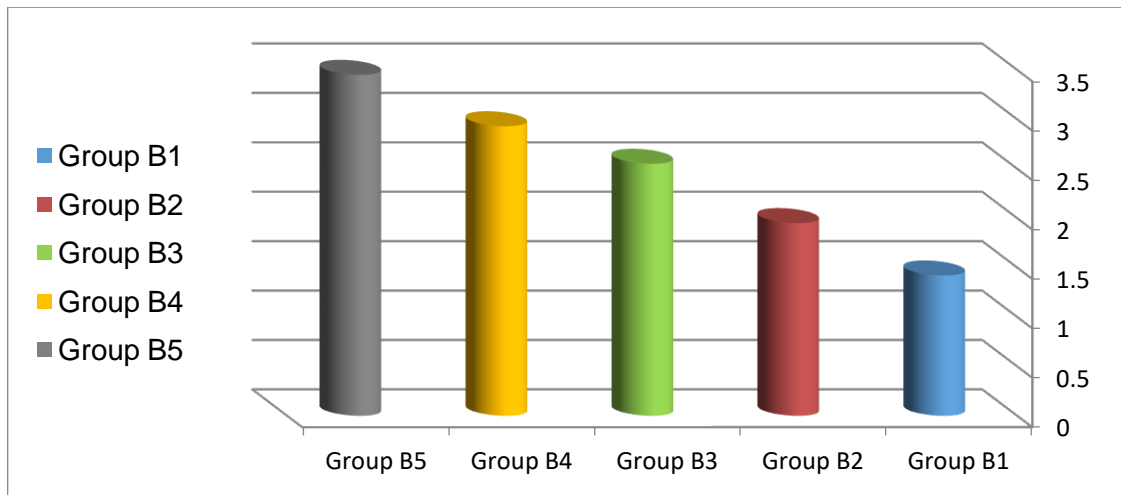
<b>Group B1</b> Control group		<b>Group B2</b> (1.5% ZrO <sub>2</sub> )		<b>Group B3</b> (3% ZrO <sub>2</sub> )		<b>Group B4</b> (5% ZrO <sub>2</sub> )		<b>Group B5</b> (7% ZrO <sub>2</sub> )		<b>P-value</b>
Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
1.42 <sup>e</sup>	0.96	1.95 <sup>d</sup>	0.63	2.55 <sup>c</sup>	1.07	2.93 <sup>b</sup>	0.56	3.45 <sup>a</sup>	1.05	0.000*

\*Significant at  $P \leq 0.05$ , Means with different letters are significantly different according to Tukey's test.

**Table 3:** Comparison between mean impact strength (J) of the tested groups of PMMA.



**Figure (1):** Bar chart of mean transverse strength (MPa) of the tested groups of PMMA.



**Figure (2):** Bar chart of mean impact strength (MPa) of the tested groups of PMMA

PMMA specimen with 5% ( $ZrO_2$ , group A4) followed by PMMA specimen with 3% ( $ZrO_2$ , group A3) then PMMA specimen with 1.5% ( $ZrO_2$ , group A2). There were significant differences ( $P < 0.05$ ) between studied groups. PMMA specimen without any additives (control group) showed significantly lowest mean transverse strength.

#### **Impact strength**

The impact strength data showed there was significant improvement in the tested groups which were reinforced with zirconium oxide nanofillers ( $ZrO_2$ ) (Table 3 and Figure 2).

There was significant increase in the impact strength for groups reinforced with (1.5%, 3%, 5% and 7%)  $ZrO_2$  when compared with control group.

#### **4. Discussion**

We principally aimed to assess possible improvements in the mechanical properties of PMMA, in particular, the transverse strength, and impact strength, through incorporating of  $ZrO_2$  Nano particles. There are three ways to improve the mechanical properties of PMMA: replacing PMMA with an alternative material; chemically modifying it; and reinforcing the PMMA with other materials [17] [18].

Addition of Zirconia Nano fillers to acrylic resin was found to improve mechanical properties. In addition to that  $ZrO_2$  was used because it has excellent biocompatibility and white color which less likely to alter esthetic. The Nano-filler particles were used in this study as it yields a better

dispersion eliminate aggregation and improve its compatibility with organic polymer [19] [20]. Proper percentage range of zirconium oxide Nano-fillers (Percentages of 1.5% - 7% by weight) was selected because percentages above 7% leads to massive changes occurred in the color of acrylic [21].

The transverse strength test, one of the mechanical strength tests, is especially useful in comparing denture base materials in which a stress of this type is applied to the denture during mastication [22]. The transverse (flexural) strength is a combination of compressive, tensile and shear strengths, all of which directly reflect the stiffness and resistance of a material to fracture [23].

The fracture of acrylic resin dentures as a result of being dropped is a common occurrence and research continues to dates to produce a denture base material with improved impact resistance. Impact strength is an important parameter as it can reflect the contact force required to cause fracture in a denture under situations such as accidental dropping [24].

The Results of the present study demonstrated a significant increase in transverse strength and impact strengths as the percentage of ZrO<sub>2</sub> fillers increased. This improvement in mechanical properties could be attributed to the high interfacial shear strength between the nanofillers and resin matrix as a result of formation of cross-links or supra molecular bonding which cover or shield the nanofillers which in turn prevent propagation of crack, also complete wetting of the nanofillers by resin lead to increase in flexural strength, fracture toughness, and hardness as volume of filler increased [25].

The results of this study are in agreement with the findings reported by others [26-28] who concluded that reinforcement of ceramics, dental restorative resins as well as acrylic resin with zirconia could exhibit improvement in their mechanical properties.

## 5. Conclusion:

Within the limitation of this study, we can conclude that addition of zirconium oxide nanofillers to PMMA increased transverse strength and impact strength of heat polymerized acrylic resin. According to the results of the present study, the best result was got when using the concentration of 7% wt.

Further studies are needed to investigate its effect on other mechanical and physical properties with different concentrations.

## 6. References

1. Nejatian T, Johnson A, Van Noort R: Reinforcement of Denture Base Resin. *Adv Sci Tech.* 2006; 49: 124-29.
2. Darbar U, Huggett R, Harrison A: Denture Fracture-A Survey. *Brit Dent J* 1994;176: 342-45.
3. John J, Gangadhar S, Shah I: Flexural Strength of Heat-Polymerized Polymethyl Methacrylate Denture Resin Reinforced with Glass, Aramid, or Nylon Fibers. *J Prosth Dent* 2001; 86: 424-27.
4. Sheikh A: SBA-Z: Causes of Denture Fracture: A Survey. *Sau Dent J.* 2006;18: 149-54.
5. Vallittu P, Alakuijala P, Lassila V, et al: In Vitro Fatigue Fracture of an Acrylic Resin-Based Partial Denture: An Exploratory Study. *J Prosthetic Dent.* 1994;72: 289-95.
6. Kanie T, Fujii K, Arikawa H, et al: Flexural Properties and Impact Strength of Denture Base Polymer Reinforced with Woven Glass Fibers. *Dent Mater J.* 2000;16:150-158.
7. Knott N: The Durability of Acrylic Complete Denture Bases in Practice. *Quint Inter.* 1989; 20: 341-43.
8. Balch, J, Smith P, Marin M, et al: Reinforcement of a Mandibular Complete Denture with Internal Metal Framework. *J Prosth Dent.* 2013; 109: 202-05.
9. Venkat R, Gopichander N, Vasantakumar M: Comprehensive Analysis of Repair/Reinforcement Materials for Polymethyl Methacrylate Denture Bases: Mechanical and Dimensional Stability Characteristics. *J Ind Prosth Soci.* 2013;13: 439-49.
10. Xu J, Li Y, Yu T, et al: Reinforcement of Denture Base Resin with Short Vegetable Fiber. *Dent Mater J.* 2013;29: 1273-79.
11. Franklin P, Wood D, Bubb, N: Reinforcement of Poly (methyl methacrylate) Denture Base with Glass Flake. *Dent Mater J.* 2005; 21: 365-70
12. Asar N, Albayrak H, Korkmaz, T, et al: Influence of Various Metal Oxides on Mechanical and Physical Properties of Heat-Cured Polymethyl Methacrylate Denture. *J Adv Prosth.* 2013;5: 241-47.
13. Mohamed A, Fallal, A, Hawary Y: Effect of Zirconium Oxide Reinforcement on Epithelial Oral Mucosa, Immunoglobulin and Surface Roughness of Complete Acrylic Heat-Cured Denture. *Egy Dent J.* 2007; 53: 941-46.
14. Skukla S, Seal, S: Phase Stabilization in Nano crystalline Zirconia. *Reviews on Advanced Materials Science,* 2003;5: 117-20.
15. International Organization for Standardization. Specification 1567: denture base polymers. 2<sup>nd</sup> ed. Switzerland: ISO; 1988.
16. Uzun G, Hersek N, Tincer T: The effect of five woven fiber reinforcements on the impact and transverse strength of a denture base resin. *J Prosthet Dent.* 1999;81:616-20.
17. Kim, S.H. and Watts, D.C. (2004) The Effect of Reinforcement with Woven E-Glass Fibers on the Impact Strength of Complete Dentures Fabricated

- with High-Impact Acrylic Resin. *The Journal of Prosthetic Dentistry*, 91, 274-280.
18. Jagger D, Harrison A, Jandt K: The Reinforcement of Dentures. *J Ora Reha*. 1999;26:185-94.
  19. Suna L, Gibson R, Gordaninejad F, Suhr J: Energy Absorption Capability of Nanocomposites: A Review. *Composites Science and Technology*. 2009;69:2392-09.
  20. Ihab, N, Hasanayn K, Ali N: Assessment of Zirconium Oxide Nano-Fillers Incorporation and Silanation on Impact, Tensile Strength and Color Alteration of Heat Polymerized Acrylic Resin. *J Bag Coll Dent*. 2012;24:36-42.
  21. Shi J, Bao Y, Huang Z, Weng Z: Preparation of Poly (Methyl Methacrylate)/Nanometer Calcium Carbonate Composite by in Situ Emulsion Polymerization. *J Zhej Uni Sci A*. 2004;5:709-13.
  22. Craig R: *Restorative Dental Materials*. 11th ed. St Louis, MO: Mosby; 2002: pp. 87-88.
  23. Jagger D, Jagger R, Allen S, Harrison A: An investigation into the transverse and impact strength of "high strength" denture base acrylic resins. *J Ora Reha*. 2002; 29: 263-67.
  24. Jagger D, Harison A: An investigation into the transverse and impact strength of high strength denture base acrylic resins. *J Ora Reha*. 2002;29:263-67.
  25. Ayad N, Badawi M, Fatah A: Effect of Reinforcement of High-Impact Acrylic Resin with Zirconia on Some Physical and Mechanical Properties. *Revista de Clínica e Pesquisa Odontológica*. 2008;4,:145-51.
  26. Giodano R: A comparison of all-ceramic restorative systems: Part 2. *Gen Dent*. 2000;48:43-8.
  27. Furman B, Rawls H, Wellinghoff S, Dixon H, Lankford J, Nicolella D: Metal-oxide nanoparticles for the reinforcement of dental restorative resins. *Crit Rev Biomed Eng*. 2000;28:439-43.
  28. Tinschert J, Natt G, Mautsch W, Augthun M, Spiekerman H. Fracture resistance of lithium disilicate-, alumina-, and zirconiabased three unit fixed partial dentures: a laboratory study. *Int J Prosthodont*. 2001;14:231-38.