International Journal of Medical Toxicology & Legal Medicine Volume 27, No. 5, 2024
e-ISSN: 0974-4614
p-ISSN: 0972-0448
https://doi.org/10.47059/ijmtlm/V27I5/105

Effect of Addition of Different Nano-Particle Materials on Some Mechanical and Physical Properties of Polymethyl Methacrylate Resin - In Vitro Study

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Received: 15.10.2024 Revised: 16.11.2024 Accepted: 27.12.2024

ABSTRACT

Objective: This study aims to evaluate a heat-cured polymethyl methacrylate resin material modified with different concentrations of silver (Ag) and zirconium oxide nanoparticles (Nps) regarding impact strength, flexure strength, and water sorption.

Material and Methods: For this study, 75 specimens were processed according to the specifications of each test (Impact strength, flexural strength, and water sorption). Specimens were divided into five groups according to the type and concentrations of Nps materials added to PMMA powder: Group A: control group only conventional heat cured PMMA resin, group B: PMMA reinforced with 0.5% Ag oxide Nps, group C: PMMA reinforced with 1% Ag oxide Nps, group D: PMMA reinforced with 0.5% zirconium oxide Nps, and group E: PMMA reinforced with 1% zirconium oxide Nps.

Results: Regarding the impact strength and water sorption, there was an insignificant increase for all modified groups compared with the control group, While the flexural strength value decreased significantly after the addition of nanoparticle materials ($P<0.05$).

Conclusions: Based on the findings of this study, we can conclude that the addition of different concentrations of silver and zirconium Oxide nanoparticles to the PMMA denture base material led to a slight increase in the impact Strength and water sorption with decreasing flexure strength.

Keywords: Nano-Particle Materials, Mechanical properties, Physical Properties, Polymethyl Methacrylate Resin.

INTRODUCTION

Polymethyl methacrylate (PMMA), has been widely used as a base material for prostheses because it is affordable, easy to fabricate and manipulate, easier to repair, low density, and has a satisfactory physical and mechanical property. It is used in tooth-or implant-supported prostheses and orthodontic appliances [1]. Improving the mechanical and physical properties of PMMA may be useful in points where masticatory forces are relatively high, such as distal extensions opposing natural teeth, single complete dentures, overdentures, and implant-supported complete dentures [2]. These improvements may help elderly patients, who may be impaired or have difficulty adapting to a new prosthesis, increase the lifespan of their prosthesis and eliminate situations such as prosthesis fracture [3]. Several researchers have demonstrated that PMMA can show

good fatigue behavior, impact strength, and anti-fungal and antibacterial effects by adding an appropriate additive to it which also can reduce the risk of fracture and improve the properties of the polymer[4-6]. PMMA was reinforced by using high-strength resins, metal wire, glass fibers, barium titanate, silicon dioxide, titanium dioxide, zinc oxide, or hydroxyapatite. However, there are drawbacks to utilizing these alternatives. Metal wires, for example, have poor resin adhesion. metal plates are costly and prone to corrosion. Furthermore, fibers irritate the tissue [7]. Polymers are historically reinforced with micrometer-sized fillers to increase strength and stiffness, improve solvent or fire resistance, or simply reduce cost. However, these micro fillers have several drawbacks, such as brittleness and opacity [8]. The science and applications of nanotechnology are constantly evolving as new products enter the market. This comes with a great deal of responsibility to ensure the safety, efficiency, and applicability of such new technologies [9]. Adding nano silver particles to the PMMA base has a favorable effect on improving the thermal conductivity and compressive strength of PMMA. Also,the use of nanosilver particles has been preferred to silver powder because the nanoparticles cause better processing and smoother surfaces than the pure powder.[10].Researchers have found that incorporating zirconia (ZrO2) fillers into PMMA greatly improved its flexural strength[11]. However, another opinion reported that the acrylic composite deteriorated due to particle agglomeration and clustering, which led to a little reduction in flexural strength[12]. Also Comparing the zirconium dioxide acrylic composite to the unfilled resin (control specimen), **Ayad et al.** found a little improvement in surface hardness and impact strength, while another study found a loss in both[13,14]. So, the mechanical properties of nanoparticle materials fall short in comparison to pure materials. Lately, worldwide research has shown several advancements in the nanocomposite field after extensive research on the mechanical and physical properties of these nanocomposites [15].In dentistry, chemical industry research is now focusing on the incorporation of nanoparticles (Nps) such as silver (Ag) oxide, titanium dioxide (TiO_2), silicon dioxide (SiO_2), aluminum oxide ($\mathbf{Al_2O_3}$), and zirconium dioxide (ZrO_2) into acrylic resins to create new materials with the same mechanical properties as the nano-oxide particles.[7]. Due to their small size, strong interactions with organic polymers, non-toxicity, non-biochemical reactions, and antifungal and antibacterial properties, nano-oxide particles are widely used. This work aimed to evaluate a heat-cured polymethyl methacrylate resin material modified with different concentrations of Ag and Zr oxide nanoparticles regarding impact strength, flexure strength, and water sorption.

MATERIALS AND METHODS

This study was carried out on 75 specimens processed according to the specification of each test (Impact strength, flexural strength, and water sorption).
In this study, Zirconium oxide Nps (Nano Gate, Egypt) and silver oxide Nps (Nano Gate, Egypt) are incorporated to heat-cured denture base material (Acrostone dental manufacture, Egypt), with different ratios.

Sample groups

Specimens were divided into five groups according to the type and concentrations of Nps materials added to PMMA powder:
Group A: control group only conventional heat-cured PMMA resin,
Group B: PMMA reinforced with 0.5% Ag oxide Nps,
Group C: PMMA reinforced with 1% Ag oxide Nps,
Group D: PMMA reinforced with 0.5% zirconium oxide Nps, and
Group E: PMMA reinforced with 1% zirconium oxide Nps.
The test specimens were made by incorporating the conventional heat-cured PMMA into the selected ratios of Nps materials. The proper monomer-to-polymer ratio was 1: 2.5 by weight and 1% of benzoyl peroxide (initiator) was added to the powder. The specimens were cured in the same curing cycle. And smoothed polished and stored in distilled water at 37 ± 1°C for 48 hours before testing.

Mold preparation

Three shapes of metal pattern mold fabricated by a CNC milling machine were used, (Vertical machining centers Millstar LMV710). Fig-1. According to. (British Standard Institute Specification No.771; 1984). a rectangular bar with a dimension of 75 mm (length) x 10 mm (width) x 10 mm (thickness) with a standard notch of 2mm (depth) at mid-span to be used for impact properties test.

Figure 1: CNC milling machine LMV710

While the other mold was designed as a flat strip by A.D.A. Specification No.12. with a dimension of 65mm (length) x 10 mm (width) x 2.5mm (thickness) for flexure strength test.
The water sorption test mold was made in the shape of a disk by A.D.A. specification No. 12, 50 mm. in diameter and 0.5 mm in thickness. And measured by a digital caliper for dimensions checking (SERIES 500, Mitutoyo Corporation, Japan).
The metal patterns were coated with a thin layer of petroleum jelly (Evasiline, Eva company, Egypt) before preparing flasks for packing of acrylic resin for easy separation from stone without stone deformity. The flask base was poured with dental stone (Deniston, type IV, Turkish design), which was mixed following the manufacturer's instructions then; metal patterns were invested into the flask and the investing material was left to set. After a complete set of the stone in the flask, the metal patterns were removed from the investing stone. The bar cavities were used as molds for the packing of acrylic resin specimens. Fig-2

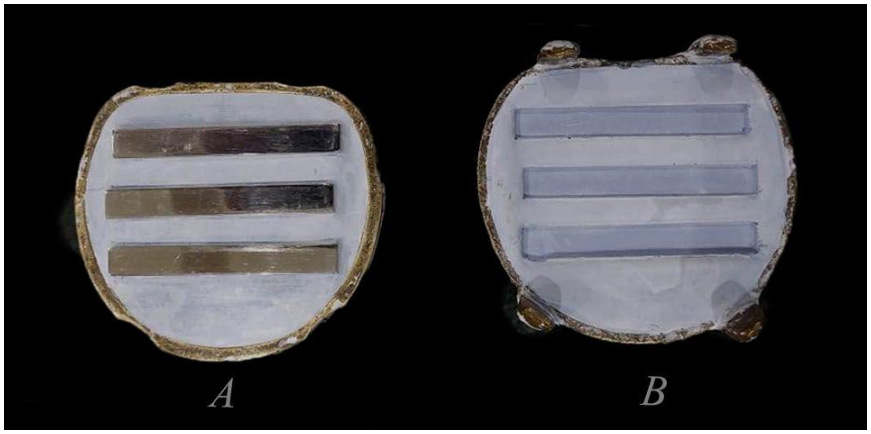

Figure 2: Proportioning and mixing of acrylic resin

An appropriate amount of heat cure acrylic resin was weighed with the help of an electronic weighing machine (Black Horse scale, Shenzhen Veiyuxing Trading CO, LTD) and mixed with monomer in the ratio 1: 2.5 by weight and 1% of benzoyl peroxide (initiator) was added to the powder. Addition of Nps to acrylic powder: Two concentrations (0.5% and 1%) from each of ZrO_2Nps, and **Ag_2O**Nps were added to a polymer of heat-cured acrylic resin. Each nanoparticle and acrylic resin powder were thoroughly mixed using mortar and pestle for initial blending followed by meticulously stirring for 30 minutes to ensure the homogeneity and distribution of the mix. According to nanoparticles, samples were divided into five groups.

Deflasking, finishing, and polishing of specimens
The flasks were allowed to slow cooling in a water bath at room temperature before deflasking. Then, after deflasking the acrylic Specimens were trimmed with a tungsten bur and ground to the final dimension with a silicon Carbide abrasive paper. Pumice was used for final polishing. The specimens were immersed in water at 50ºC for 1 hour for excess residual monomer removal and stored in water at room temperature for 48 hours until the testing was performed. Fig-3a,b

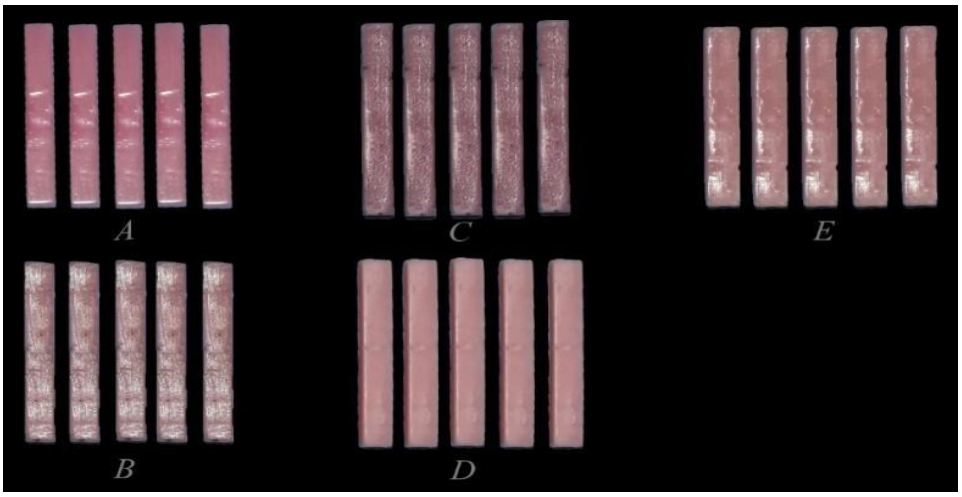

Figure 3a: Acrylic resin specimens for impact strength test (A) (control), (B) specimens containing 0.5% Ag_2ONps., (C) specimens containing 1% Ag_2ONps.), (D) specimens containing 0.5% ZrO_2NPs (E) specimens containing 1% ZrO_2NPs

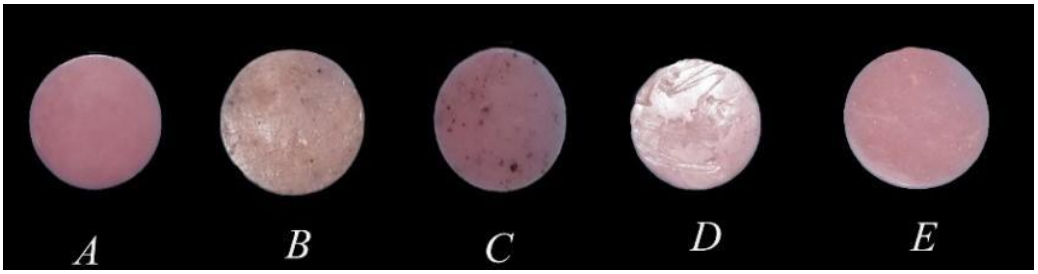

Figure 3b: Acrylic resin specimen for water sorption test: (A) specimen without nanoparticles, (B) specimen containing 0.5% **Ag_2O**Nps.(C) specimen containing 1% Ag_2ONps., (D) specimen containing 0.5% ZrO_2NPs (E) specimen containing 1% ZrO_2NPs.

Test procedure

The impact strength test will be determined by using an Izod Impact Tester (Brooks, Model IT 14 Press 1,68 Kg) Fig-4

The energy (E) required for breaking the test specimens was calculated from the following formula.

$E = W R (\cos \beta - \cos \alpha) - L$

E: Energy required for breaking the test specimen in joule,

W: Weight of pendulum in Newton,

R: Distance from the axis of rotation to the center of gravity of the pendulum, in meters,

B: Angle of the rise of the pendulum after breaking the specimen,

***α*:** Angle of fall of the pendulum, and

L: Loss of energy due to friction, in joule.

Where E is dividing the cross-section area at the notch. Charpy's impact strength machine; (A) specimen placed horizontally where the un-notched side faces the hummer; (B) load released in a pendulum action to fracture the specimen and impact strength digitally recorded.

Figure 4. Mounting the sample on the (Izod Impact Tester).

The flexure strength test

Specimens' number, preparation, curing, finishing, and storage of were done as described for the impact strength test. All samples were individually and horizontally mounted in a custom-made loading fixture three-point bend test assembly; two parallel stainless-steel rods with a span length of 50 mm supporting the specimen, with the damage site centrally located on the tensile side] on a computer-controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA), Fig 5,

with a load cell of 5 kN and data were recorded using computer software (Instron® Bluehill Lite Software). Then the samples were statically compression loaded until fracture at a crosshead speed of 1 mm/min. The stress-strain curves were recorded with computer software (Instron® Bluehill Lite Software). FS represents the limiting stress at which failure or instability is imminent. The test was achieved by a Universal Testing Machine. The transverse strength was calculated from the following formula.

S = 3*P*I / b*d

S: Transverse strength, in MPa,
P: Maximum load before fracture, in Newton,
I: Distance between supports, in mm,
b: Width of the specimen, in mm, and
d: Thickness of the specimen, in mm.

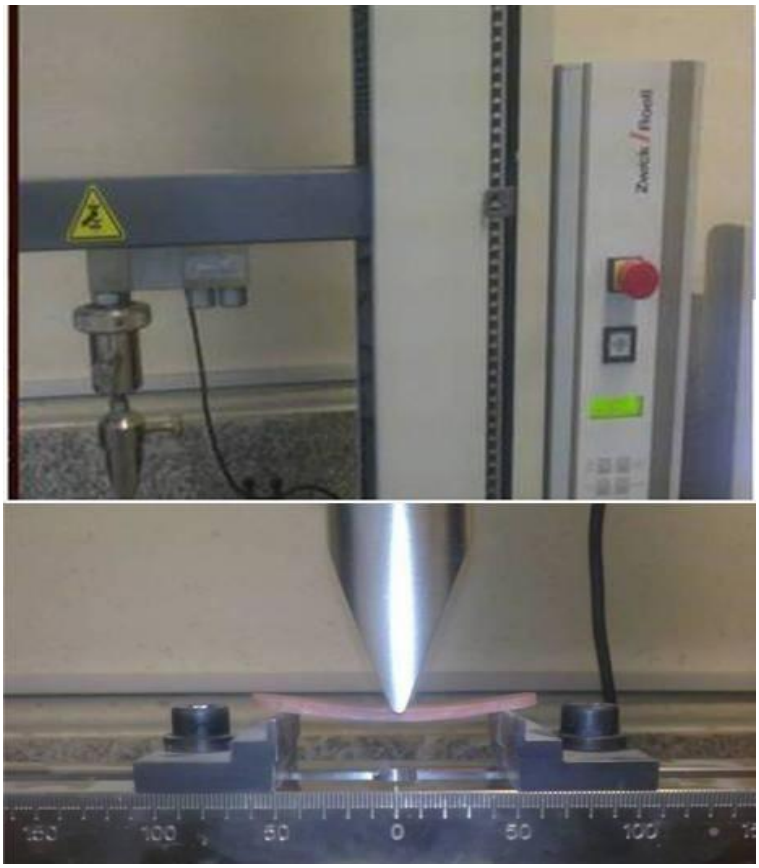

Figure 5: Universal testing machine (Instron).

Water sorption testing

Twenty-five disc-shaped specimens were placed inside a desiccator (Wheaton, Millville, N.J.) at 37oC ± 1oC for 24 hours, then, allowed to stand at ambient temperature for 1 hour. Each specimen was weighed by an electronic balance (Mettler Instrument AG, Greifensess, Switzerland), Fig-6, and the previously described cycle was repeated until the specimens reached constant mass. Specimens were immersed in distilled water at 37oC ± 2oC for 7 days, then, they were removed from the water, weighed, and reconditioned to constant mass in the desiccator. Water sorption (WR) was calculated in mg/cm2. The role of water sorption is calculated as follows for each disk.

$$\text{Water sorption } \left(\frac{\text{mg}}{\text{cm}^2}\right) = \frac{(m_1) - (m_2)}{(a)}$$

(m_2): Mass after immersion, (m): Conditioned mass, and (a): Surface area

Figure 6: Specimen weighed in electrical balance.

Statistical analysis

Statistical analysis was done by SPSS v28 (IBM Inc., Armonk, NY, USA). Shapiro-Wilks test and histograms were used to evaluate the normality of the distribution of data. Quantitative variables were presented as mean and standard deviation (SD) and compared between the three groups utilizing the ANOVA (F) test with post hoc test (Tukey). A two-tailed P value < 0.05 was considered statistically significant.

RESULTS

(A) Impact strength:

The highest impact strength mean value was recorded in Group C (39.218 kJ/m^2) followed by Group E (38.612 kJ/m^2) followed by Group B (37.662 kJ/m^2) then Group D (37.262 kJ/m^2) while the lowest impact strength mean value was recorded for Group A (control) (34.446 kJ/m^2). This difference between studied groups was statistically non-significant as proved by the One-way Repeated ANOVA test p=0.135>0.05). **Table** (1) &**Figure 7**

Table 1: Descriptive statistics of impact strength test results (Mean values ±SD) for acrylic resin groups with/without different concentrations of metal oxide modifications

Groups	**Impact strength test**						**ANOVA**	
	Range			**Mean**	**±**	**SD**	**F**	**P-value**
Control group	30.21	-	39.15	34.446	±	3.720	1.991	0.135ns
0.5 % Silver oxide group	34.2	-	41.3	37.662	±	2.953		
1 % Silver oxide group	36.4	-	43.04	39.218	±	2.593		
0.5 % Zirconium oxide group	34.11	-	40.21	37.262	±	2.350		
1 % Zirconium oxide group	36.15	-	42.92	38.612	±	2.799		

TUKEY'S Test				
	Control	**0.5 % Silver**	**1 % Silver**	**0.5 % Zirconium**
0.5 % Silver	0.433			
1 % Silver	0.112	0.914		
0.5 % Zirconium	0.559	0.999	0.825	
1 % Zirconium	0.201	0.985	0.997	0.947

There is a significant at P-value< 0.05 (*), ns: nonsignificant at P-value > 0.05.

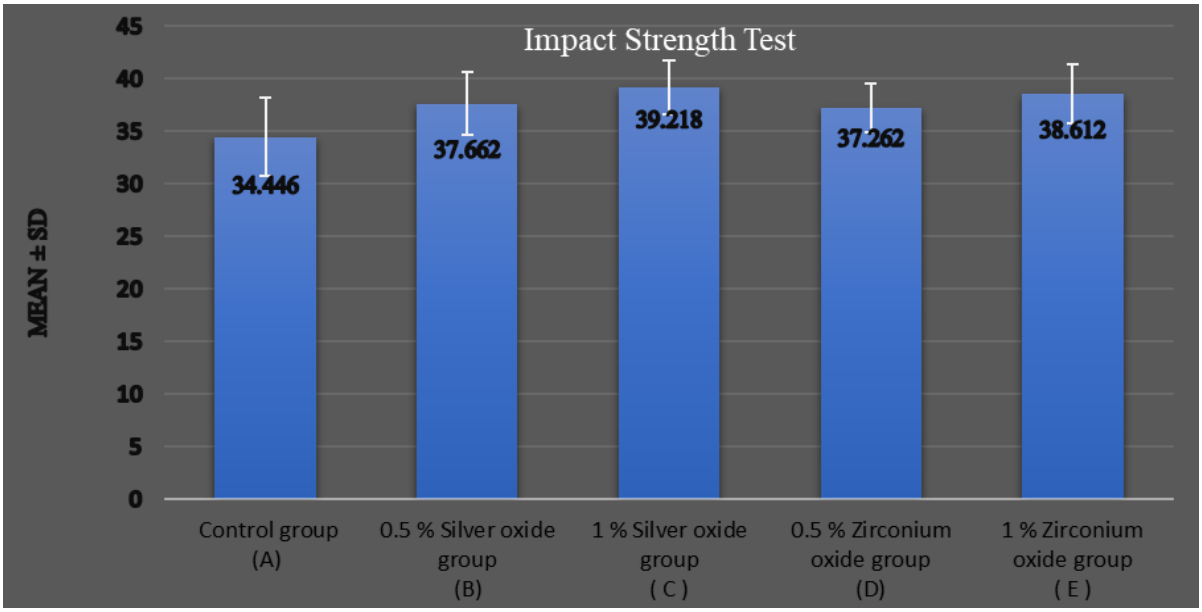

Figure 7: impact strength mean values for all groups

(B) Flexure strength

The highest flexure strength mean value was recorded in Group A (139.968MPa), followed by Group D (119.306MPa), followed by Group B (111.372MPa), followed by Group E (105.570MPa), followed by Group C (103.988MPa). This difference between studied groups was statistically significant as proved by the One-way Repeated ANOVA test p=0.029<0.05). This difference between studied groups was statistically significant as proved by the One-way Repeated ANOVA test p=0.029<0.05). **Table (2)& Figure.8**

Table 2: Descriptive statistics of flexure strength test results (Mean values ±SD) for acrylic resin groups with/without different concentrations of metal oxide modifications

Groups	**Flexure strength test**						**ANOVA**	
	Range			**Mean**	**±**	**SD**	**F**	**P-value**
Control group	123.68	-	155.56	139.968	±	11.333	3.361	0.029*
0.5 % Silver oxide group	79.51	-	129.53	111.372	±	18.871		
1 % Silver oxide group	71.46	-	123.53	103.988	±	25.065		
0.5 % Zirconium oxide group	98.76	-	133.78	119.306	±	12.717		
1 % Zirconium oxide group	83.84	-	127.87	105.570	±	18.010		

TUKEY'S Test				
	Control	**0.5 % Silver**	**1 % Silver**	**0.5 % Zirconium**
0.5 % Silver	0.124			
1 % Silver	0.034*	0.964		
0.5 % Zirconium	0.387	0.954	0.662	
1 % Zirconium	0.045*	0.985	1.000	0.743

There is a significant at P-value< 0.05 (*), ns: nonsignificant at P-value > 0.05.

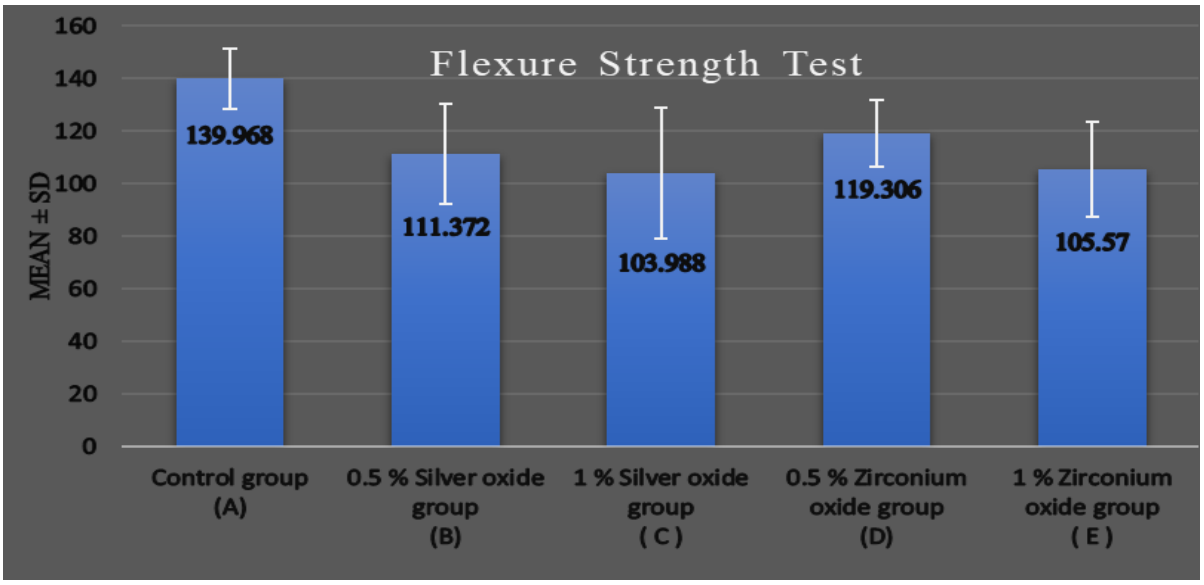

Figure 8: Flexure strength mean values for all acrylic resin groups

(C) : Water sorption test

The highest water sorption mean value was recorded in Group C (2.33±0.84mg/cm^2), followed by Group E (2.24±0.22mg/cm^2), followed by Group B (2.17±0.19mg/cm^2), followed by Group D (2.10±0.14mg/cm^2), followed by Group A (1.90±0.02mg/cm^2).

This difference between studied groups was statistically non-significant as proved by the One-way Repeated ANOVA test p=0.148>0.05). **Table (3), Figure 9**

Table 3: Descriptive statistics of water sorption test results (Mean values ±SD) for acrylic resin groups with/without different concentrations of metal oxide modifications

Groups	**Water Sorption test**						**ANOVA**	
	Range			**Mean**	**±**	**SD**	**F**	**P-value**
Control group	1.87	-	1.94	1.908	±	0.029	1.908	0.148ns
0.5 % Silver oxide group	1.88	-	2.4	2.170	±	0.198		
1 % Silver oxide group	1.55	-	2.88	2.338	±	0.486		
0.5 % Zirconium oxide group	1.89	-	2.27	2.102	±	0.144		
1 % Zirconium oxide group	1.85	-	2.36	2.246	±	0.222		

TUKEY'S Test				
	Control	**0.5 % Silver**	**1 % Silver**	**0.5 % Zirconium**
0.5 % Silver	0.530			
1 % Silver	0.112	0.848		
0.5 % Zirconium	0.770	0.994	0.624	
1 % Zirconium	0.288	0.990	0.980	0.906

There is a significant at P-value< 0.05 (*), ns: nonsignificant at P-value > 0.05.

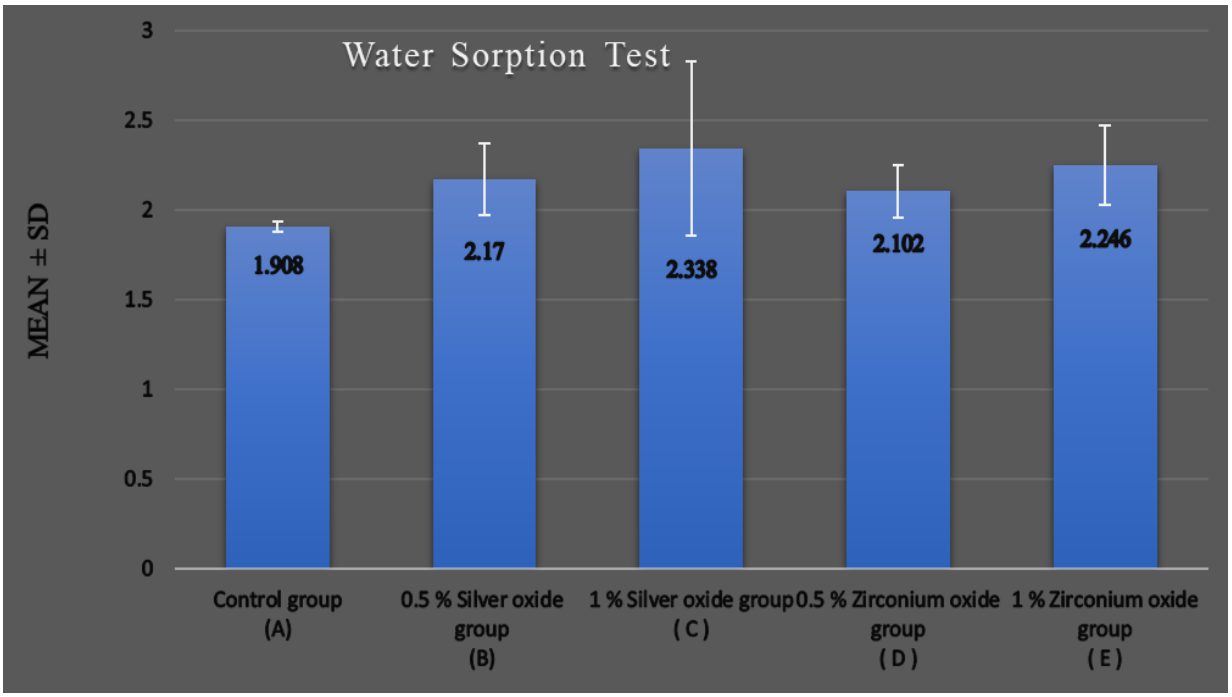

Figure 9: Water sorption means values for all acrylic resin groups.

DISCUSSION

PMMA is the most frequently used resin material due to its good biocompatibility, consistent physicochemical qualities, simplicity of manipulation, low cost, and appropriate aesthetics. Unfortunately, PMMA-based resins have clinical problems such as wear and fracture from inadequate mechanical characteristics, it is essential to improve these resins' mechanical qualities to be able to use it for long-term success in the oral environment without early failures [16].The impact strength is a measure of absorbed energy by the material before fracture. For prostheses, the impact failure can be represented by suddenly falling off dentures and colliding with the ground. Fracture energy for the prepared specimens is obtained from the impact test[17].In the present study, the mean impact strength value increases with increasing concentration of Ag_2O and **ZrO_2** nanoparticles. This result was in agreement with the previous experiments[18-20]. Also agreed with **Hamdy et al.** who found that the impact strength of the modified groups with Ag-doped CNTs (11.2 kJ/mm2) was significantly greater than that of the control groups (2.3 kJ/mm2) [21]. Coming in line with our results, **Srivastava et al.** assessed the impact strength of various types of acrylic resin. They demonstrated that there was an increase in the impact strength of acrylic resin by incorporating silver nanoparticles and zirconium oxide powder[22] . This was supported by another study that evaluated the effect of zirconium oxide nano-filler addition on transverse strength and impact strength of heat-polymerized acrylic resin, an in vitro study. They illustrated that Zirconium oxide nanofillers added to heat-cure acrylic resin (PMMA) had the potential as a reliable denture base material with increased impact strength[22]. Along the same line, a study evaluated the preparation and characterization of zirconium dioxide-reinforced aluminum metal matrix composites. They noted that the addition of nano-ZrO2 boosted and improved the material's impact strength[23].The compatibility between the polymeric matrix and the nanoparticles possibly improves the mechanical strength[23,24].

On the other hand, The findings presented here disagree with those of **Sharanya Adhikari.,** who concluded that the impact strength of the acrylic resin significantly reduced with the addition of metal oxide nanoparticles[25]. Flexural strength is an important property for dental resins, as the major cause of prosthesis fracture in the oral cavity is related to stresses caused by repeated application of masticator forces, which induce more bending movements[26]. The findings presented here agree with other studies[27, 28-31] which discovered that all unmodified (control) specimens exhibited greater flexural strength than the modified specimens, which decreased gradually from 0.5 wt% to 5.0 wt%. Many causes can be responsible for this effect. Poor dispersion of Nps, in addition to the low concentration of **ZrO_2**Nps, will not fill the space between polymer chains homogeneously, which will interrupt the continuity of the resin matrix and create defects in the material, ultimately weakening it. One of the causes is Nps dispersion, which has been demonstrated to play an important role in reinforcing material[26].

Conversely, our results disagree with those other studies that found that flexural strength values were generally higher with the addition of nanoparticles [21],[32-34]. These differences could be explained by differences in the materials used, polymerization methods, and the size and quantity of nanoparticles. Water sorption is a process where water molecules are absorbed within the polymeric material due to its small size[36,36].PMMA absorbs water slowly over a period of time when placed in an aqueous environment which

affects the mechanical and dimensional properties of the polymer. It causes plasticization and lowers mechanical properties. According to **Anusavice.,** it has been estimated that for each 1% increase in weight produced by water absorption, acrylic resin expands 0.23% linearly[37].

In this study, we found that a slight increase in water sorption value occurred with all tested groups when using different nano-particle types, the increase of water sorption is affected by the increasing concentration of nanoparticles. These results are in agreement with many studies, that investigated the effect of reinforcing conventional heat-cured PMMA with zirconia nanoparticles on water sorption and solubility after 28 days, the findings showed that an increase in nanoparticle concentration increased water sorption [38]. According to **Chladek G et al.,** the modified acrylic resin's mean sorption values were significantly impacted by the inclusion of nanosilver. They discovered that when the concentration of nanosilver was between 0.1% and 0.4%, the rate of water sorption increased [39]. Increasing water sorption mean values could be attributed to the addition of AgNPs, with their hydrophobic nature, so the addition of AgNPs resulted in a decrease of microporosity that resulted after the polymerization process and subsequently reduced the water sorption [40]. Then again, the findings of water sorption were in disagreement with those of **Asar et al.,** who evaluated the effect of reinforcing conventional heat-cured acrylic resin with various metal oxide microparticles (**TiO_2**, **ZrO_2**, andAl_2O_3 using 1% and 2% by volume) on mechanical and physical properties such as water sorption and solubility [41]. The results showed that the incorporation of metal oxides significantly decreased water sorption and solubility, particularly with 2% of **ZrO_2**, which showed the lowest solubility compared to the conventional acrylic resins [42] .

CONCLUSIONS

Based on our findings within t h e limitation of the study; we can conclude that the addition of different concentrations of Silver and Zirconium Oxide nanoparticles to the PMMA denture base led to:

- Slight increase in the impact Strength values of the reinforced PMMA groups.
- Slight decrease in the flexure strength value of the reinforced PPM groups.
- Insignificant changes in water sorption effect in all modified PMMA groups.

Further study is recommended to have different percentages or mixing techniques for those nan particles to the PMMA.

ACKNOWLEDGEMENT

Special thanks to the Faculty of Dental Medicine, Al-Azhar University, Asuit - Egypt, the Deanship of Scientific Research (DSR), and the Faculty of Dentistry at King Abdulaziz University, Jeddah, for supporting the authors in finalizing this project.

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