



ISSN NO. 2320-5407

Journal Homepage: - [www.journalijar.com](http://www.journalijar.com)

**INTERNATIONAL JOURNAL OF  
ADVANCED RESEARCH (IJAR)**

Article DOI: 10.21474/IJAR01/9798  
DOI URL: <http://dx.doi.org/10.21474/IJAR01/9798>



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Journal DOI: 10.21474/IJAR01

### RESEARCH ARTICLE

#### EFFECT OF SILVER NANOPARTICLES FILLERS ADDITION ON FLEXURAL STRENGTH, FRACTURE TOUGHNESS, IMPACT STRENGTH, COMPRESSIVE STRENGTH AND HARDNESS OF HEAT-POLYMERIZED ACRYLIC RESIN.

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#### Manuscript Info

##### Manuscript History

Received: 16 July 2019

Final Accepted: 18 August 2019

Published: September 2019

#### Abstract

**Objectives:** Polymethyl methacrylate, PMMA, is widely used in prosthodontics for fabrication of removable prostheses. This study was undertaken to investigate the effect of adding silver nanoparticles (AgNPs) to PMMA at 0.5% and 2% concentrations on flexural strength, fracture toughness, impact strength, compressive strength and hardness of heat-polymerized acrylic resin.

**Materials and methods:** The silver nanoparticles (AgNPs) were mixed with heat-cured acrylic resin with different concentrations at 0.5 and 2 wt% 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 (50 × 30 × 30 mm) for the flexural strength, fracture toughness, cylindrical samples (25×38 mm) were prepared for compressive strength, rectangular shaped specimens (60 x 7 x 4 mm) were prepared for impact strength testing and (30 × 10 × 2.5 mm) were fabricated for measuring hardness. PMMA without additives was prepared as a test control.

The recorded values of flexural strength in (MPa), fracture toughness in (MPa.m<sup>1/2</sup>), impact strength in (KJ/m<sup>2</sup>), compressive strength in (MPa) and hardness (VHN) 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 ≤ 0.05.

**Result:** Addition of silver nanoparticles (AgNPs) to PMMA significantly increased the flexural strength, fracture toughness, impact strength, compressive strength and hardness. Conclusion: These results indicate that of silver nanoparticles added to PMMA has a potential as a reliable denture base material with increased flexural strength, fracture toughness, impact strength, compressive strength and hardness. According to the results of the present study, the best mechanical properties were achieved by adding 2% wt AgNPs concentration.

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**Introduction:-**

Denture bases can be fabricated using various materials, including metals and heat cured acrylic resins.

Metal denture base is not preferred due to several disadvantages, including lack of retention because of heavy denture, poor esthetic features, cost, difficulty in tissue replacement in severely resorbed alveolar ridge and inability to relin [1]. Acrylic resins have been used widely because of their good esthetics and favorable characteristics such as easy handling and biocompatibility [2]. These materials account for approximately 95% of the denture base materials used in prosthodontics. The majority of prosthetic acrylic resins consist of PMMA, polyethyl methacrylate (PEMA), and additional copolymers. Polymethyl methacrylate, PMMA, is commonly used in dentistry for different purposes such as trial base plates, orthodontic functional appliances and denture bases [3].

PMMA is the most popular denture base material currently available.4 Almost all the dentures are fabricated with this type of polymer [4]. Although the characteristics of this material are not ideal in every aspect, it has many desirable features that make it very favorable. Acrylic resins have excellent esthetic properties, sufficient strength, low water sorption, low solubility, and biocompatibility [5].

In a survey to compare ten types of denture base resins, nearly 70 percent of dentures broke in the first three years of delivery[6,7].In a study evaluating the fracture of the prosthesis, 33% of the repairs have been caused by debonded/detached teeth, 29% due to midline fractures more frequently found in the upper dentures, and the remainder by other types of breaks.

In another study the authors reported that the Mandibular partial denture was the most commonly needing repair [8]. So, the measuring of mechanical properties of the denture base materials is important to evaluate the effect of adding different strengthening materials [9].

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) [10] or by incorporating a rubber phase [11], metal fram [12], metal oxides [13], or fibers [14]. Despite these efforts to improve the fracture resistance of PMMA has obtained promising results [15]. The reinforcement of polymers used in dentistry with metal-composite systems has been a prime interest [16].

The antimicrobial properties of silver, especially when the nanoparticles are added to the denture have been reported in several studies [17]. Chladek and colleagues showed that silver nanoparticles inhibit the fungus *Candida albicans* growth and adding this material to the dentures could reduce oral diseases among edentulous patients [18]. The antibacterial effect of silver is even more noticeable when used as nano-particles [19].

Silver particles have been used as an addition to acrylic resin in order to improve its mechanical properties [20]. Although adding 25% silver powder to denture base increases its thermal conductivity more than 4 times, it results in a significant decrease in the mechanical properties of acrylic resin, making denture more susceptible to breaking by an impact [21].

In the past, micrometer-sized particles were used to improve the resin characteristics; however, these particles presented several drawbacks. Regarding advances in nanotechnology sciences and benefits of adding silver nanoparticles to the acrylic base, which leads to better processing and smoother surface compared to micrometer-sized silver powder, the use of silver nanoparticles is preferred. Among various nanofillers available the silver nanoparticles are the most widely used nanoparticles because of their ductility, electrical and thermal conductivity and antimicrobial activity [22-25].

On the other hand, decoloration of resins and high costs can restrict their use.The effect of of of the addition of silver nanoparticles at concentrations of 0.5 and 2 wt percent on flexural strength, strength, strength, fracture resistance, impact strength, compressive strength and hardness of heat - polymerized acrylic resin was evaluated in this study.

**Objectives:-**

Polymethyl methacrylate, PMMA, is widely used in prosthodontics for fabrication of removable prostheses. This study was undertaken to investigate the effect of adding silver nanoparticles (AgNPs) to PMMA at 0.5% and 2%

concentrations on flexural strength, fracture toughness, impact strength, compressive strength and hardness of heat-polymerized acrylic resin.

### Materials and methods:-

An in vitro study was conducted to evaluate the effect of silver nanoparticles (AgNPs) with a diameter of <35 nm (Model number: SP – A00601, Top Nano Technology Co., Ltd., Iran) at 0.5 and 2 wt% concentrations on flexural strength, fracture toughness, impact strength, compressive strength and hardness of heat-polymerized acrylic resin (PMMA) was used as the control (Acrostone (A), Anglo-Egyptian Company. Hegaz, Cairo, Egypt, Batch No.505/04).

Silver nanoparticles (AgNPs) were 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) 150 bar shapes specimens were prepared to be used in this study. 30 specimens were used for each test [flexural strength (group A), fracture toughness (group B), impact strength (group C), compressive strength (group D) and hardness (group E)].

Grouping of the specimens:

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

Table 1: Classification and grouping of the specimens.

Groups	Subgroups	Description	No. of Specimens
Group A	Group A1	Heat-cure acrylic resin (PMMA) without additives as control.	10 specimens
	Group A2	PMMA with 0.5% silver nanoparticles (AgNPs)	10 specimens
	Group A3	PMMA with 2 % AgNPs.	10 specimens
Group B	Group B1	PMMA without additives as control.	10 specimens
	Group B2	PMMA with 0.5% AgNPs.	10 specimens
	Group B3	PMMA with 2% AgNPs.	10 specimens
Group C	Group C1	PMMA without additives as control.	10 specimens
	Group C2	PMMA with 0.5% AgNPs.	10 specimens
	Group C3	PMMA with 2% AgNPs.	10 specimens
Group D	Group D1	PMMA without additives as control.	10 specimens
	Group D2	PMMA with 0.5% AgNPs.	10 specimens
	Group D3	PMMA with 2% AgNPs.	10 specimens
Group E	Group E1	PMMA without additives as control.	10 specimens
	Group E2	PMMA with 0.5% AgNPs.	10 specimens
	Group E3	PMMA with 2% AgNPs.	10 specimens
Total			150 specimens

### 1. Flexural Strength

Specimens were tested by 3-point bend test on Lloyd universal testing machine (model LRX plus II, Fareham, England)[ figure 1] at a cross head speed of 1 mm/min. For the 3 point bend test, a fixture was fabricated with the dimensions (65 mm length x 10 mm width x 2.5 mm thickness) according to International Standards Organization (ISO) Specification No.1567.

On top of the fixture two plates were welded at a distance of 15 mm from the center on either side. A customized “T” shaped stress applicator rod with the dimension of 80 × 20 mm was fabricated, by which stress can be applied in the center of the specimen. The specimen was placed on the rollers in such a way that the center of the specimen coincided with the center of the distance between the two rollers.

This whole unit was mounted on the lower jaw of the universal testing machine and the stress applicator rod was fixed on the upper jaw. A load was applied with “T” shaped rod on the center of the specimen until fracture occurred and peak force (F) values were recorded at this point in Newton [26].

The maximum force (F) necessary to produce fracture of the specimens was recorded in Newton. The flexural strength Q was calculated in (MPa) for all specimens from the "Equation (1)":

$$Q = \frac{3FI}{2BH^2}$$

"In this formula, "F" is the maximum load or force which is applied to the center of the specimen to fracture it (N); "I" is the distance between the two rests on the surface under the tensile force (mm); "B" is the width (mm) and "H" is the height of the specimen between the surfaces under the tensile and compressive forces (mm)."

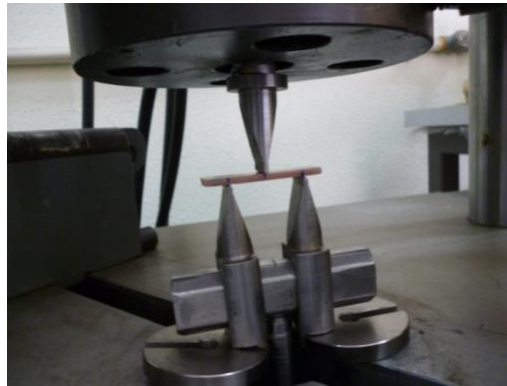


Fig.(1): A photograph of Flexural Strength

## 2. Fracture Toughness

For fracture toughness testing, specimens were fabricated with the dimensions of (65 mm length x 10 mm width x 2.5 mm thickness) according to International Standards Organization (ISO) Specification No.1567. After all specimens were stored in distilled water at 37°C for 24 hours, a notch was made in the middle of each specimen on one edge with 2.5 mm lengths using sand paper disk. Fracture toughness tests were performed on Lloyd universal testing machine (model LRX plus II, Fareham, England) with a cross-head speed of 1 mm/min, and peak load to fracture was recorded. The recorded data were used to determine the fracture toughness ( $K_{Ic}$ ) in MPa.m<sup>1/2</sup> according to the "Equation (2)" [26]:

$$K_{Ic} = pc/bw^{1/2} \cdot F(a/w)$$

Where  $pc$  is the maximum load (kN) prior to crack advance,  $b$  is specimen thickness (cm),  $w$  is the width of the specimen (cm),  $a$  is crack length (cm) and  $F$  is calculated from the following Equation (2):

$$(2+a/w) (0.886 + a/w - 13.32 a^2/w^2 + a^3/w^3 - 5.6 a^4/w^4)$$

$$F(a/w) = \frac{\quad}{(1-a/w)^{3/2}}$$

## 3. 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 [27]. 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.

#### 4. Compressive strength

The compressive strength testing of the PMMA was carried out in accordance with the International Organization for Standardization ISO 9917 standards [28]. Moulds were made with sets of cylindrical samples, each sample with a diameter of 4 mm and a height of 6 mm. These were filled to excess with freshly mixed PMMA and then covered with acetate sheets. The moulds were then sandwiched between two stainless steel plates, clamped and then incubated at 37 °C for at least 1 h.

The samples were then removed from the moulds and placed in distilled water and then incubated at 37 °C for 1 day before compression testing was carried out. The samples for compression testing were then loaded on Lloyd universal testing machine (model LRX plus II, Fareham, England) using a 5 kN load cell at a crosshead speed of 1 mm/min.

The compression strength for each sample was then calculated according to the following Equation (3):

$$C = \frac{4\rho}{\pi d^2}$$

Where **C** is compressive strength (Mega Pascals), **ρ** is maximum applied load (Newtons) as measured by the Instron and **d** is diameter of the sample (millimetres).

#### 5. Hardness

For Hardness testing, specimens were fabricated with the dimensions of 30 mm length x 10 mm width x 2.5 mm thickness according to International Standards Organization (ISO) Specification No.1567. Surface hardness was determined using Digital Display Vickers Microhardness Tester [figure 2] (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd. China) which is suitable for acrylic resin material. With a Vickers diamond indenter and a 20× objective lens. A load of 20 gram was applied to the surface of the specimens for 15 sec. five indentations were equally placed over a specimen and not closer than 1 mm to the adjacent indentations or to the margin of the specimens were made on the surface of each specimen [figure 3]. The diagonal length of the indentations was measured by built in scaled microscope.

Surface microhardness calculation:

Vickers microhardness was obtained using the following Equation (4):

$$VHN = 1.854 L / d^2$$

Where:

*VHN*: Vickers hardness in Kg/mm<sup>2</sup>.

*L*: Load in Kg.

*d*: Length of the diagonals in mm.



Fig.(2): A photograph of Vickers microhardness tester.

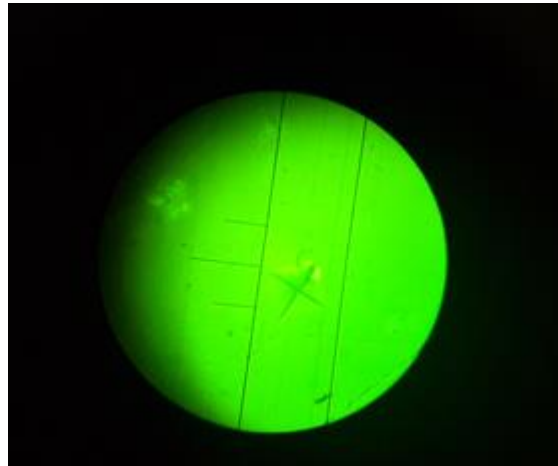


Fig.(3): A photograph of specimen under Vickers microhardness tester.

### Statistical analysis:

The recorded values of flexural strength, fracture toughness, impact strength, compressive strength and hardness 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  $\leq 0.05$ .

### Result:

#### 1. Flexural Strength

Both Table 2 and Figure 4 show a comparison between mean flexural strength in (MPa) of the tested groups of PMMA. ANOVA test showed statistically significant difference between all groups.

PMMA specimen with 2% silver nanoparticles (AgNPs) (group A3) showed significantly highest mean flexural strength followed by PMMA specimen with 0.5% AgNPs. There were significant differences ( $P < 0.05$ ) between studied groups. PMMA specimen without any additives (control group) showed significantly lowest mean flexural strength.

Table 2: Comparison between mean flexural strength (MPa) of the tested groups of PMMA

Material	Mean	SD	P-value
PMMA without additives (group A1)	85.54 <sub>c</sub>	1.145	0.000*
PMMA with 0.5 AgNPs (group A2)	116.04 <sub>b</sub>	3.028	
PMMA with 2% AgNPs (group A3)	123.72 <sub>a</sub>	1.96	

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

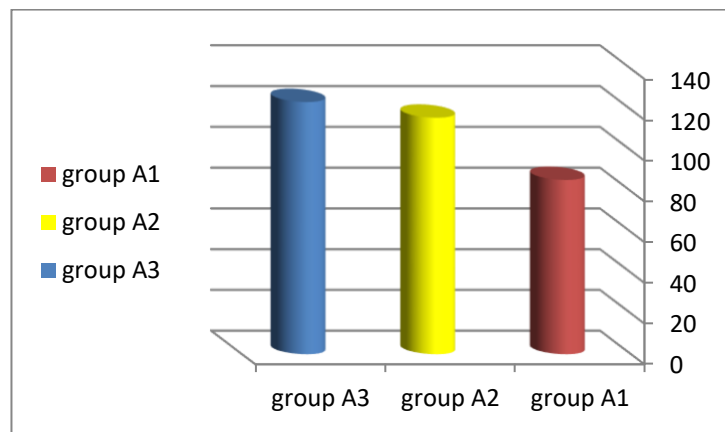


Fig. (4): Bar chart of means flexural strength (MPa) of the tested groups.

**2. Fracture Toughness**

The tensile strength data showed there was significant improvement in the tested groups which were reinforced with AgNPs (Table 3 and Figure 5).

There was significant increase in the fracture toughness for groups reinforced with (0.5% and 2 %) AgNPs when compared with control group.

Table 3. Comparison between mean fracture toughness (MPa.m<sup>1/2</sup>) of the tested groups of PMMA.

Material	Mean	SD	P-value
PMMA without additives (group A1)	2.30 <sub>c</sub>	0.158	0.000*
PMMA with 0.5% AgNPs (group A2)	2.85 <sub>b</sub>	0.37	

PMMA with 2% AgNPs (group A3)	3.95 <sub>a</sub>	0.16	
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\*: Significant at  $P \leq 0.05$ , Means with different letters are statistically significantly different according to Tukey's test

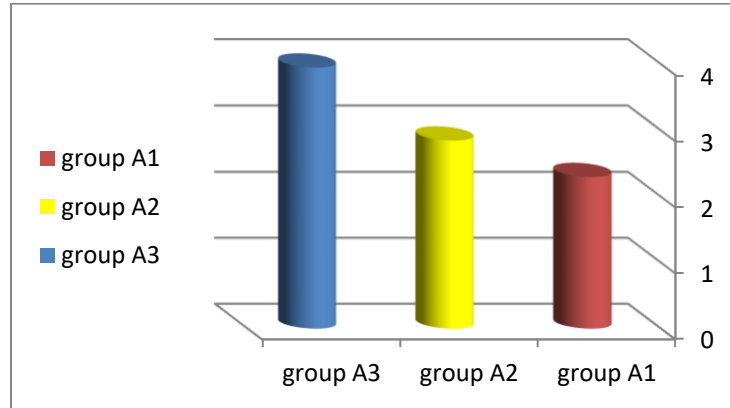


Fig. (5): Bar chart of means fracture toughness (MPa.m<sup>1/2</sup>) of the tested groups.

**3. Impact strength testing**

The impact strength data showed there was significant improvement in the tested groups which were reinforced with AgNPs (Table 4 and Figure 6).

There was significant increase in the impact strength for groups reinforced with (0.5% and 2 %) AgNPs when compared with control group.

Table 4. Comparison between mean impact strength (J) of the tested groups of PMMA.

Material	Mean	SD	P-value
PMMA without additives (group A1)	1.67 <sub>c</sub>	0.96	0.000*
PMMA with 0.5% AgNPs (group A2)	2.98 <sub>b</sub>	0.56	
PMMA with 2% AgNPs (group A3)	3.85 <sub>a</sub>	1.05	

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

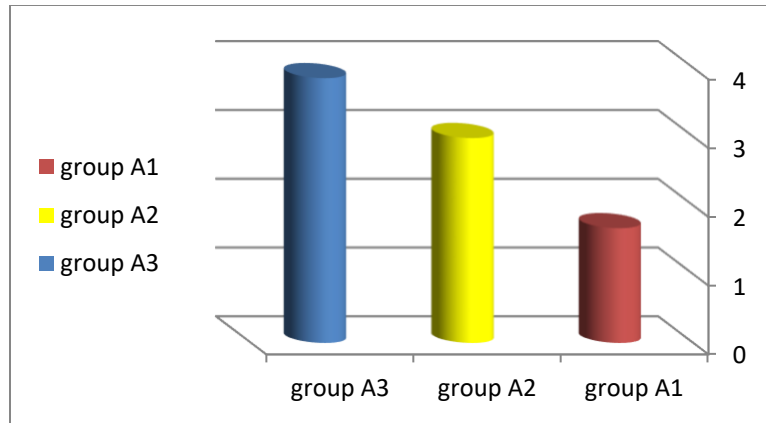


Fig. (6): Bar chart of means impact strength (J) of the tested groups.

**4. Compressive strength**

compressive strengths in various groups showed that acrylic resin at 0.5% and 2% AgNPs concentrations had a significantly higher compressive strength compared with the control group ( $P < 0.05$ ), but the strength difference between the groups containing 0.5% and 2% AgNPs was not significant ( $P > 0.05$ ). The comparisons of compressive strength results of all the groups are shown in Figure 7.

Table 5. Comparison between mean compressive strength (MPa) of the tested groups of PMMA

Material	Mean	SD	P-value
PMMA without additives (group A1)	99.5 <sub>b</sub>	3.86	0.001*
PMMA with 0.5% AgNPs (group A2)	113.23 <sub>a</sub>	5.75	
PMMA with 2% AgNPs (group A3)	119.76 <sub>a</sub>	9.89	

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

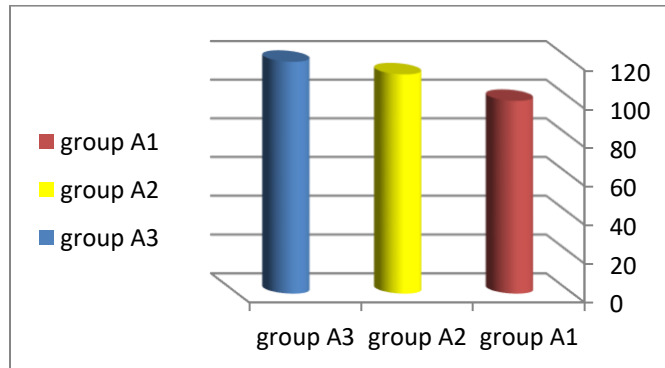


Fig. (7): Bar chart of means compressive strength (MPa) of the tested groups.

**5. Hardness**

Both Table 6 and Figure 8 show the mean hardness of tested groups. All specimens showed hardness mean values higher than that control group. PMMA specimen with 2% AgNPs (group A3) showed significantly highest mean hardness followed by PMMA specimen with 0.5% AgNPs (group A2). There were significant differences ( $P < 0.05$ ) between studies groups. PMMA specimen without any additives (control group) showed significantly lowest mean hardness.

Table 6. Comparison between mean hardness (VHN) of the tested groups of PMMA.

Material	Mean	SD	P-value
PMMA without additives (group A1)	15.95 <sub>c</sub>	0.96	0.000*
PMMA with 0.5 AgNPs (group A2)	20.60 <sub>b</sub>	0.56	
PMMA with 2% AgNPs (group A3)	23.19 <sub>a</sub>	1.05	

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

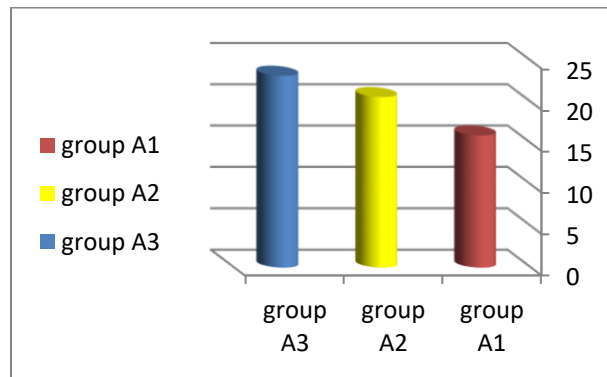


Fig. (8): Bar chart of means hardness (VHN) of the tested groups

**Discussion:**

We principally aimed to assess possible improvements in the mechanical properties of PMMA, in particular, the flexural strength, fracture toughness, impact strength, compressive strength and hardness through incorporating of AgNPs 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 [29] [30].

In recent years AgNPs have been largely investigated because of their antimicrobial activity. In particular, AgNPs are now considered antibacterial agents due to inhibition of oral pathogens and have been used in various applications [23]. There are many reports about dependence of acrylic resin's properties on nanoparticle concentrations [31]. Selection of silver as filler in this study was based on properties of this filler. The first reason is the high thermal conductivity of silver, which can improve the thermal conductivity of the denture. In addition, it has been demonstrated that silver not only has no adverse effects in the oral cavity [32], but also it can reduce the adhesion **Candida albicans** and has anti-microbial effects [19]. In addition; some studies have revealed that AgNPS can improve the mechanical properties of acrylic resin. Low concentration of silver would reduce material costs and less monomer would be needed while mixing with acrylic powder. Therefore, the mechanical properties of the final polymer would not be compromised. Chladek et al [33] reported that the

mechanical and physical properties of the composite are influenced by silver nanoparticle concentration. They also showed that mechanical properties of composites decreased by increasing silver nanoparticles. It has been demonstrated that addition of more than 5 wt% of the metal fillers into acrylic resin would reduce tensile strength [34].

Addition of silver nanoparticles to acrylic resin was found to improve mechanical properties. Fractures in an acrylic denture base are a common clinical problem. Flexural strength of denture base resin was measured in this study because it is considered the primary mode of clinical failure [35]. Fatigue failure does not require strong biting forces as relatively small stresses caused by mastication over a period of time can eventually lead to the formation of a small crack, which propagates through the denture and results in a fracture. The maximal biting forces of a patient can reach up to 700 N, but these values are reduced (100 - 150 N) [36] with the removal of dentures. Denture fractures are essentially due to stress concentration and increased flexing [37]. Many authors found that the fracture toughness seems to be a suitable measurement to demonstrate the effects of resin modifications [38].

Hardness of the polymerized resin has been found to be sensitive to the residual monomer content in the resin material. Moreover, hardness measurement have been successfully used as an indirect method of evaluating polymerization depth of resin-based composite materials [39] and the degree of conversion of conventional heat polymerizing and self-curing acrylic resins. In addition, hardness has been used to predict the wear resistance of dental materials [40].

The Results of the present study demonstrated a significant increase in flexural strength, fracture toughness, impact strength and hardness as the percentage of AgNPs fillers increased. This improvement in mechanical properties could be attributed to the high interfacial shear strength between the nanofiller 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 [41]

Improvement of hardness with the increase in concentration of AgNPs nanofillers may have be due to inherent characteristics of the AgNPs particles. AgNPs possesses strong ionic interatomic bonding, giving rise to its desirable material characteristics, that is, hardness and strength.

The results of this study are in good agreement with the findings reported by others who concluded that reinforcement of ceramics, dental restorative resins as well as acrylic resin with Zirconia nanoparticles could exhibit improvement in their mechanical properties [42]. The increase of mechanical properties was due to good bonding between nanofillers and resin matrix [43]

According to the results, AgNPs with 0.5 and 2 wt% increased the compressive strength of acrylic resins, but increasing AgNPs concentration from 0.5 wt% to 2 wt% did not improve compressive strength of acrylic resin significantly due to acrylic resin is a brittle material but at compressive conditions behaves like ductile materials [44].

The results of this article were based on an “in vitro” study; so future “in vivo” studies can be conducted to evaluate the effects of these changes in dentures on clinical performance and patient satisfaction.

### **Conclusion:**

This study was conducted to evaluate the effect of adding AgNPs to PMMA with two different weight percentages on five properties of acrylic resin.

The properties were flexural strength, fracture toughness, impact strength, compressive strength and hardness. The results showed that the effect of AgNPs significantly depends on its concentration. Based on the results adding AgNPs with proper concentrations to PMMA can improve its mechanical characteristics with effects and strongly recommended in the palatal portion of acrylic base of complete maxillary dentures.

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