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EFFECT OF COPPER OXIDE NANOPARTICLES INCORPORATION ON THE MECHANICAL AND SURFACE PROPERTIES OF HEAT POLYMERIZED DENTURE BASE RESIN: AN IN VITRO STUDY

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ABSTRACT

Purpose: This study aimed to examine the flexural strength, surface roughness, and surface hardness of heat-polymerized denture base material incorporated with copper oxide (CuO) nanoparticles.

Materials and Methods: Forty heat-polymerized acrylic resin specimens with the dimensions of (65x10 x 2.5mm) were prepared according to the manufacturer's instructions. Four groups (n=10 each) were examined. Unmodified denture base material is the control group, the other groups were incorporated with 1%, 3%, and 5% CuO nanoparticles to the heat-polymerized acrylic resin powder. Flexural strength was examined using Universal Testing Machine. A non-contact optical profilometer was used for surface roughness testing, and a Vickers digital testing machine to measure the surface hardness. One-way ANOVA test followed by Tukey's post-hoc test was used to compare different groups.

Results: Regarding flexural strength, there was statistically significant difference between the nanoparticle-added groups and the control group. The highest strength was detected in the 1% CuO nanoparticles group. The surface hardness was significantly increased in comparison to the control group. The surface roughness was not significantly altered except for the 5% concentration.

Conclusion: Within the limitations of this study, it was concluded that incorporating CuO nanoparticles to heat-polymerized denture base resin improved flexural strength. The highest strength was with the 1% concentration of the nanoparticles. The addition of CuO nanoparticles also improved the surface hardness and did not affect the surface roughness in concentrations below 5%.

KEYWORDS: denture base material, flexural strength, surface roughness, surface hardness

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INTRODUCTION

Polymethyl methacrylate PMMA is commonly used in the prosthodontic management of completely and partially edentulous cases. This is because it offers many advantages such as; its pleasing aesthetics, reasonable cost, simple laboratory procedures, light weight, lack of toxicity, and being stable in the oral environment.[1]

However, poor flexural strength and low fracture resistance are known to be its main drawbacks, which usually result in denture fracture during function.[2] Various methods have been used to improve the strength of PMMA including giving more bulkiness to the material in the susceptible areas, co-polymerization, cross-linking,[3,4] and the addition of different reinforcing fibers.[5]

Recently, many studies have investigated the reinforcement of PMMA denture base materials through incorporating different types and concentrations of nanoparticles such as Silicon Dioxide (SiO_2) ,[6,7,8] Titanium Dioxide (TiO_2) ,[8,9,10] Silver (Ag),[10,11,12] Aluminium Oxide (AL_2O_3),[12,13] Zirconium Dioxide (ZrO_2),[14,15] Zinc oxide (ZnO),[16] and Copper Oxide (CuO). [17]

According to these studies, the properties of the reinforced material depend mainly on the size and concentration of the used nanoparticles. The addition of the appropriate concentration could result in improved mechanical properties of PMMA denture base material.

Improving the mechanical properties should not be associated with adverse effects on the surface properties. Some studies reported that the addition of proper concentration of nanoparticles enhanced the surface properties of acrylic resin denture base materials.[9,14,16]

Based on the literature reviews, limited studies have investigated the effect of incorporating copper oxide nanoparticles on the mechanical and surface properties of denture base materials. Therefore, this study aimed to examine the flexural strength, surface roughness, and surface hardness of heat-polymerized denture base material after the addition of 1%, 3%, and 5% CuO nanoparticles.

MATERIALS AND METHODS:

In this study, conventional heat-polymerized PMMA powder (Trevalon Hi Dentsply G-7, Saket, New Delhi) was modified by the addition of CuO nanoparticles 99.9 % purity 20 nm particle size acquired from (Nanografi Nanotechnology, Ankara, Turkey)

The experimental groups included in this study were:

Group 1: unmodified heat-polymerized PMMA (Control group)

Group 2: 1 wt% CuO nanoparticles + 99 wt% PMMA

Group 3: 3 wt% CuO nanoparticles + 97 wt% PMMA

Group 4: 5 wt% CuO nanoparticles + 95 wt% PMMA

Forty (n=10 for each group) heat-polymerized acrylic resin specimens were fabricated with the dimensions of (65 mm long, 10 mm wide, and 2.5 mm thick) according to the ISO 1567 standards. [18] A special metal mold was customized with five spaces according to the specified dimensions to be used for specimens' preparation. (Figure 1)

Specimens processing:

To prepare the specimens, the powder was measured using a digital weighing machine (Denver Instrument, Gottingen, Germany). The 1%, 3%, and 5 % of CuO nanoparticles were incorporated within the polymer powder to bring it to obtain 100% powder. The polymer powder with the incorporated nanoparticles was homogenized using an electric mixer (President Dental, Germany) in a 400 rpm cycle at room temperature for 20 minutes.[13]

The powder/liquid ratio used for mixing was 24 g/10 ml, as recommended by the manufacturer. The metal mold was painted with a thin coating of separating medium to allow easy removal of the acrylic resin specimens. While in the dough stage, the mix was properly kneaded and packed into the customized metal mold. The mold was placed in a water bath (KaVo Elektrotechnisches Werk GmbH) and processed for 8 hours at 74± 1°C, and then boiled for 2 hours. The mold was allowed for bench cooling. The specimens were carefully separated and polished with silicon carbide papers (Waterproof silicon carbide paper, English Abrasives Ltd., London, England) for 5 minutes under water cooling. [19]

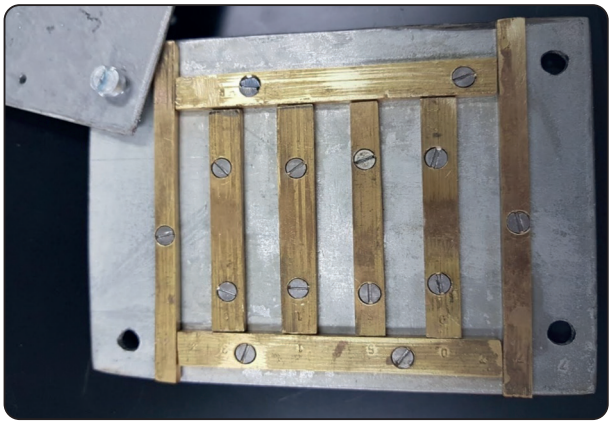

Fig. (1) Custom metal mold used for specimens fabrication.

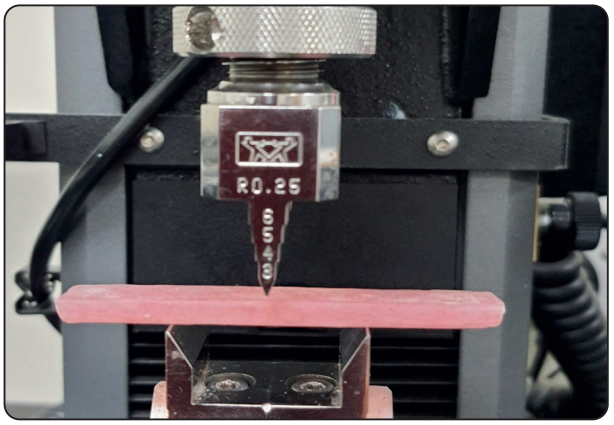

Fig. (2) Flexural strength testing using a 3-point bending machine.

Testing the specimens

1- Flexural strength testing

Before testing the specimens were preserved in distilled water at 37˚ C for a week. The test was conducted by a three-point bending test on a universal testing machine (5567 Universal Testing Machine; Instron Ltd., USA) (Figure 2). A load was exerted with a crosshead perpendicular to the specimen center with a speed of 5 mm/min till fracture of the specimen occurred. Data were obtained by using a software program (Bluehill v1.5; Instron Ltd). The highest load applied to cause specimen failure was measured in Newtons (N). Flexural strength (FS) was calculated according to the following equation: [20]

$$FS\ (MPa) = 3\ PL/\ (2bd^2\ )$$

Where **P** is the maximum load, **L** is the span length (50 mm), **b** is the specimen width (10 mm), and **d** is the specimen thickness (2.5 mm).

2- Testing the surface roughness

Specimens were further polished using a mechanical polisher (Metaserve 250 grinder-polisher, Buehler) at a speed of 100 rpm for 5 minutes under wet conditions to ensure surface standardization. A noncontact optical profilometer (Contour Gt-K1 optical profiler; Bruker Nano, Inc., Tucson, AZ, USA) was used to determine the surface roughness of the same acrylic resin specimens after being fractured. [21]

The profilometer was used to assess the surface roughness (*Ra*) at a resolution of 0.01 mm with a standard camera at 2.5 × (Figure 3), the gained images were interpreted using a software program (Vision 64, Bruker Nano). The average *Ra* value (μm) of three recorded readings was calculated for each specimen. [22]

3- Testing the surface hardness

Surface hardness was measured using a Vickers digital microhardness tester (MicroMet 6040, Hardness Tester, Buehler, USA) with diamond

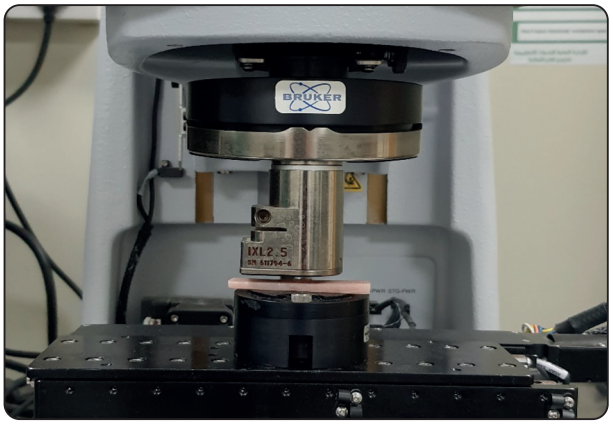

Fig. (3) Measuring surface roughness using a noncontact optical profilometer.

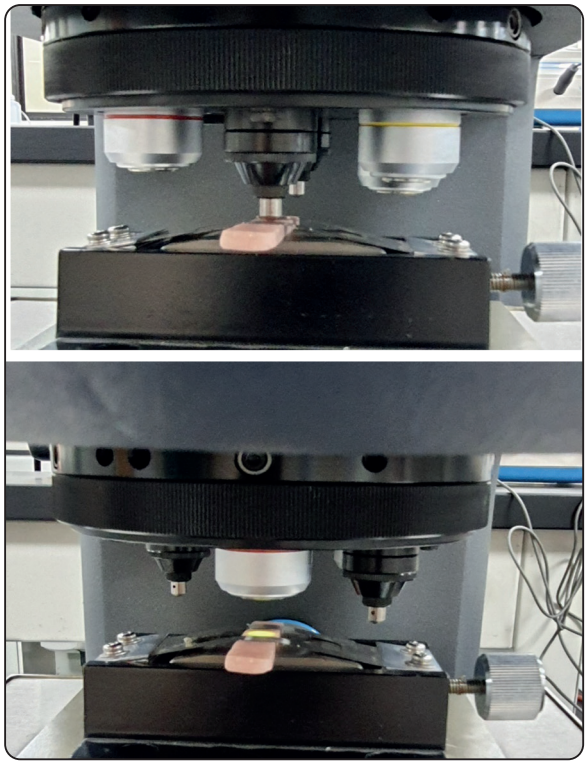

Fig. (4) Measuring surface hardness using a microhardness digital testing machine showing the diamond indenter, and objective lens.

indenter and 10 × objective lens (Figure 4). A 100 g load was exerted perpendicular to the specimen surface for 15 seconds. The hardness was digitally evaluated at three distant areas per specimen, and the mean values were then calculated. [23]

Statistical Analysis

Quantitative data were demonstrated as mean and standard deviation (SD) values. One-way ANOVA test followed by Tukey's post-hoc test was used to compare the different groups. The significance level was set at $P \leq 0.05$. Statistical analysis was carried out using IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.

RESULTS

The flexural strength mean values for the different groups are shown in Table 1. The results showed a statistically significant difference between the groups (P-value <0.001, Effect size = 0.572). Pair-wise comparisons between the groups revealed that 1% CuO nanoparticles showed the statistically significantly highest mean flexural strength. Although there was some decrease in the flexural strength showed by the 5% CuO nanoparticles concentration, but this decrease was statistically non-significant.

Descriptive statistics of the surface roughness (Ra) of the tested materials is illustrated in Table 2. According to the results, the 5 % CuO nanoparticles group showed the statistically significantly highest mean Ra. Nevertheless, there was no statistically significant difference between the control, 1%, and 3% CuO nanoparticles groups.

Descriptive statistics of the surface hardness of the tested materials is listed in Table 3. There was a statistically significant difference between the modified and unmodified acrylic resin groups (P-value <0.001, Effect size = 0.663). However, there was no statistically significant difference in surface hardness between the different added concentrations; all showed significantly higher mean hardness than the control group.

TABLE (1) Descriptive statistics and results of one-way ANOVA test for comparison between flexural strength values (MPa) in different groups

Group (n = 10)	Mean	SD	*P*-value	*Effect size (Eta squared)*
Control	79.2 [C]	3.5	<0.001*	0.572
1 % CuO	118.5 [A]	6.4		
3 % CuO	96.6 [B]	7.3		
5% CuO	90.8 [B]	10.1		

**** Significant at P ≤ 0.05, Different superscripts indicate statistically significant difference between groups according to Tukey's test***

TABLE (2) Descriptive statistics and results of one-way ANOVA test for comparison between surface roughness Ra values (μm) in different groups

Group (n = 10)	Mean	SD	*P*-value	*Effect size (Eta squared)*
Control	0.142 [B]	0.003	0.001*	
1% CuO	0.149 [B]	0.002		
3% CuO	0.153 [B]	0.003		
5% CuO	0.225 [A]	0.008		

****Significant at P ≤ 0.05, Different superscripts indicate statistically significant difference between groups according to Tukey's test***

TABLE (3) Descriptive statistics and results of one-way ANOVA test for comparison between hardness values (VHN) in different groups

Group (n = 10)	Mean	SD	*P*-value	*Effect size (Eta squared)*
Control	19.6 [B]	2.2	0.001*	
1% CuO	29.3 [A]	4.8		
3% CuO	28.8 [A]	4		
5% CuO	29.3 [A]	3.4		0.663

****: Significant at P ≤ 0.05, Different superscripts indicate statistically significant difference between groups according to Tukey's test***

DISCUSSION

This study investigated the effect of incorporating different concentrations of CuO nanoparticles to PMMA denture base material on flexural strength, surface roughness, and surface hardness.

Copper oxide nanoparticle was selected for this study as it is a metal oxide with antibacterial properties that could inhibit of Candida albicans growth, the main causative organism for denture stomatitis. [24] Moreover, a study revealed that the addition of copper oxide nanoparticles improved the shear bond strength of denture adhesives without cytotoxicity.[25]

In addition, reinforcing denture base materials with copper nanoparticles can decrease the water sorption of acrylic resin, and accordingly enhance the durability of dentures.[26] However, the effect of copper oxide nanoparticles on the mechanical and surface properties of denture base materials is limited in the literature.

Many studies realized that low concentrations of nanoparticles caused positive effect, however high concentrations could have unfavorable results. [6,9,27] In addition, it was reported that nanoparticle concentrations more than 7% may yield a noticeable color change in the modified denture base material. [28] So, the concentrations used in this study were 1%, 3%, and 5 % to represent low and high concentrations without affecting the color of the modified resin.

Some studies incorporated the nanoparticles into the monomer [9,29], while other researches added them into the polymers.[6,8] In the current study, the nanoparticles were mixed with powder in order to avoid the possibility of nanoparticles agglomeration in the monomer.

The results obtained from this study disclosed a significant increase in the flexural strength at the 1% concentration of CuO nanoparticles as compared to the control group. The increase in flexural

strength could be explained by the homogeneity of the nanoparticles being embedded within the acrylic resin when added with low concentrations. Furthermore, it has been proposed that the small nanoparticles could be dispersed to fill in the spaces of the resin interpolymeric chain, and this limits the particles movement, and improves the flexural strength.

Among the CuO nanoparticles groups, there was some decrease in flexural strength with the 5% concentration. This could be due to the fact that as the nanoparticles concentration increases, the susceptibility to agglomeration increases, which leads to the development of stress-concentration areas, decreasing the flexural strength.

This finding is consistent with Karci et al.[13], who found that the flexural strength of Aluminium oxide and Titanium oxide nanoparticles modified PMMA increased at 1% but decreased with increasing the concentration and that the greatest decrease was at 5% concentration. They attributed this to the difficulty of obtaining a homogeneous distribution of the particles for concentrations above 1%.

Similarly, Gad et al.[6] reported significant improvement in the flexural strength of repaired acrylic resins by adding low concentrations of nano-SiO2.

The same results were also shown by Cevik and Yildirim-Bicer[21], who attributed the decrease in the flexural strength after the addition of high concentrations of nanoparticles to alterations in surface porosity as identified by SEM analysis of the nanoparticles-incorporated specimens.

Recently, Ansarifard et al. investigated the effect of adding 0.5, 5, 50, and 500 μg/ml CuO nanoparticles to the monomer, and showed a significant increase in the flexural strength in all concentrations except the 500 μg /ml which resulted in a decrease in the flexural strength values.[30]

Regarding the effect of CuO nanoparticles on surface roughness, there was an increase in the surface roughness values. Nonetheless, this increase was non-significant in the low concentrations (1% and 3% CuO nanoparticles). This could be justified by the addition of low concentrations of small well-dispersed nanoparticles to the acrylic resin may result in few particles being involved on the surface of the specimens that could not be recognizable for roughness parameter measured on a micrometer scale.

However, increasing the concentration of the nanoparticles to 5 % increased the surface roughness significantly. This could be justified by the increased concentration of the additive particles, which may adversely affect the distribution and homogeneity of the particles within resin matrix, and increase the rate of nanoparticle agglomeration.

These results were in accordance with some previous studies. For instance, Cevik and Yildirim-Bicer [21] and Alzayyat et al.[22] reported that adding nano SiO2 increased the surface roughness. In the same context, Gad et al.[31] claimed that the incorporating concentrations above 0.5% of nano ZrO_2 and nano-SiO_2 into denture base materials may result in a damaging effect to the surface roughness that renders it to be clinically unaccepted.

Likewise, Al-Hiloh et al.[32] conducted a study in which acrylic resin was modified by 3% silanized zirconium oxide and declared that there was no change in the surface roughness.

On the other hand, Aljafery et al.[33] showed a significant decreasing effect of Ag-Zn zeolite particles added at different concentrations (0.50 % and 0.75 %) to heat-polymerized and cold polymerized acrylic resins. These controversial results may be attributed to the particle size, concentrations, filler distribution, as well as the different methodologies used.

The results of the present study demonstrated that the addition of CuO nanoparticles improved the

surface hardness, as there was a significant increase in the hardness values of the modified acrylics as compared to the control group. This increase may be related to the existence of hard CuO nanoparticles particles scattered at random in between the polymer matrix specifically on the specimen surface.

This finding is in line with many studies[22,32,33,34], which revealed an increase in the surface hardness of the denture resin after being modified with different types and concentrations of metal oxide nanoparticles.

While, Cevik and Yildirim-Bicer [21] reported that silica nanoparticles incorporation did not result in a significant change in hardness.

Conversely, da Silva et al.[35] concluded that the addition of surface-treated silica at concentrations of 0.1% to 5.0% caused decreased hardness of the modified acrylic resin. However, none of the articles investigated the effect of CuO nanoparticles on surface hardness.

According to the results of the current study, CuO nanofiller concentration played an essential role in the improvement process, as the influences were related mainly to the concentration. The preferred concentration of CuO nanoparticles that could be used for PMMA modification is 1%, resulting in improved flexural strength and hardness without adversely affecting the surface roughness.

As an in vitro study, it had limitations. The acrylic resin specimens used for the test did not simulate the structure of complete denture. Moreover, the mechanical test used did not represent the oral cavity environment. The low sample size is considered another limitation. Consequently, it is suggested to investigate the addition of CuO nanoparticles in low concentrations to acrylic resin complete denture bases in a situation that could mimic the oral cavity environment.

CONCLUSION

Within the limitations of this study, it was concluded that incorporating of CuO nanoparticles to heat-polymerized denture base resin improved flexural strength. The highest strength was with the 1% concentration of the nanoparticles. The addition of CuO nanoparticles also improved the surface hardness and did not affect the surface roughness in concentrations below 5%.

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