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Effect of Incorporation of Henna and Silver-nanoparticles on Surface Properties of PMMA Acrylic Resin

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KEYWORDS

Heat-cured PMMA, Henna, Silver-nanoparticles, Surface properties, Surface hardness.

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ABSTRACT

Aim: This study was aimed to evaluate the effect of Henna fillers and silvernanoparticles (Ag-Nps) fillers on surface properties of PMMA acrylic resin. **Subjects and methods:** One type of conventional heat-cured acrylic resin (Vertex tm Rapid simplified), Henna with a concentration of 1% by weight (Egyptian Local industry), and silver nanoparticles (Ag-Nps) with a concentration of 0.5% by weight (Nano-gate Company Egypt); were selected for this study. The different fillers were added to the polymer powder and was stirred with an electric mixer before mixing with monomer, mixing, packing, and processing was done using the conventional water bath method. After processing, all specimens were finished and polished, then kept in distilled water till tested. All modified and unmodified (control) specimens were used to measure surface hardness and surface roughness. Statistical data analysis was conducted via SPSS program . **Results:** Incorporation of Henna and Ag-Nps significantly decrease surface hardness of PMMA acrylic resin. While they insignificantly decrease its surface roughness. **Conclusion:** Surface hardness of PMMA acrylic resin adversely affected by the incorporation of fillers, while surface roughness improved.

INTRODUCTION

PMMA acrylic resin is the most commonly used material for the fabrication of complete and partial denture bases which may be attributed to its ease of fabrication, easy repair, low cost, lightweight, and color matching ability ⁽¹⁻⁴⁾. However, this material is not fulfilling the all ideal requirements of denture base material and has many shortcomings which can cause prosthesis failure ^(5,6).

PMMA dentures have many mechanical and physical drawbacks such as low flexural strength ⁽⁷⁾, weak impact strength ⁽⁸⁾, low hardness, surface porosity ⁽⁹⁾, residual monomers, dimensional instability, water sorption, and solubility ⁽¹⁰⁾.

To overcome these drawbacks, several researchers have been reported about the improvement of mechanical and physical properties PMMA denture base polymers by incorporation of different reinforcing materials such as metal fillers, glass and polymeric fibers, and nanoparticles such as Ag-Nps^(1,3,11,12).

It was found that the size, shape, and distribution of filler particles in the polymer matrix, strong adhesion at the matrix/filler interface and degree of polymerization play a significant role in the mechanical properties of filled polymer composites ^(3,13). The lower degree of polymerization conversion is usually associated with greater number of residual unreacted monomers which can act as a plasticizer and reduces the mechanical properties of the resulted polymer ^(3,14,15).

Recently, various researches attempted to study the influence of incorporation of natural powder fillers such as *Lawsonia inermis* (Henna), pomegranate peels, seed powder of dates Ajwa, and neem on biological, physical, and mechanical properties of PMMA acrylic resin ⁽¹⁶⁻¹⁹⁾.

Numerous studies have been reported about the incorporation of Ag-Nps and Henna powder into conventional heat-cured PMMA acrylic resin that may act as good antimicrobial agents ^(20,21). However, the effect of incorporation of Ag-Nps and Henna powder as reinforcement fillers has not been substantiated and very limited literature is available on the effect of these materials on the physical and mechanical properties of PMMA acrylic resins. ^(3,19).

The previous studies found that the minimum antimicrobial inhibitory concentration of Ag-Nps and Henna fillers which incorporated in acrylic resins without significant adverse effect on their mechanical properties was 1wt.%, 0.5wt.% for Henna and Ag-Nps respectively ^(18,19,22-24). So, in this study, we choose these minimum inhibitory concentrations as a base-line in evaluating their effect on surface hardness and surface roughness of heat-cured PMMA acrylic resin.

This study was essentially designed to evaluate and compare the influence of the addition of Ag-Nps and Henna fillers on surface properties of PMMA heat-cured acrylic resin.

MATERIALS AND METHODS

A power analysis of sample size revealed that a total of 132 specimens (n=11) was required. This study involved an in-vitro investigation of 132 specimens of heat-cured acrylic resin were prepared according to; American Dental Association (ADA) specification specimens' dimensions for each test, $(65 \times 10 \times 2.5 \text{ mm})$ for flexural strength ⁽²⁵⁾, $(20 \times 3 \text{ mm})$ for hardness and surface roughness ⁽²⁶⁾, and $(20 \times 1 \text{ mm})$ for water sorption and solubility test⁽²⁷⁾.

Sample Grouping:

This study has been divided into three main groups according to additives incorporated in PMMA;

Group 1: A total number of 44 specimens of conventional PMMA heat-cured acrylic resin (unmodified) as (**Control Group**) (n=44).

Group 2: A total number of 44 specimens PMMA containing 1wt. % Henna particles that incorporated into PMMA acrylic resin (n=44).

Group 3: A total number of 44 specimens PMMA containing concentrations of 0.5 wt.% Ag-NPs that incorporated into PMMA acrylic resin (n=44).

Each main group was subdivided into four subgroups according to the type of test (n=11) (flexural strength, micro-hardness, surface roughness, and water sorption and solubility).

Specimens Fabrication:

A stainless-steel mold with internal dimensions of $(65 \times 10 \times 2.5 \text{ mm})$ for flexural strength test, $(20 \times 3 \text{ mm})$ for micro-hardness and surface

roughness tests, and $(20\times1\text{mm})$ for water sorption and solubility test were used to fabricate 132 wax specimens, thirty-three specimens for each test (n=33). Modified polymers powders are prepared by addition Henna and Ag-Nps fillers to the powder of PMMA resin with a proportion of 1 and 0.5wt.% respectively using a digital balance (Precisa 205A; Moosmattstrasse, Dietikon, Switzerland), and was stirred with an electric mixer before mixing with monomer.

According to type of test; each stainless-steel metal plates painted with a separating medium, and then flasked with plaster into a metal flask, after complete setting of plaster, the top half of the metal flask and the stainless-steel metal plates removed leaving spaces in plaster of the bottom half of flask with the same dimension of each stainless-steel metal plates. Then, the mold painted with a separating medium for the application of PMMA acrylic resin of different main groups (Figure 1). Then, the unmodified and modified acrylic resin powders were mixed with monomer liquid according to the manufacturer's instructions (1:1 by volume). The specimens then were packed into the specially designed stone mold in the dough stage and flasked. Then, the specimens were polymerized through immersion in a temperature-controlled curing water bath for 6 hours (28,29).



Fig. (1) Stainless-steel patterns invested in dental stone.

The finishing and polishing procedures of all specimens were done using a tungsten carbide bur for trimming, followed by ground with an emery paper120, 200, 400, and 600 grain respectively, to remove any remaining small scratches and to get a smooth, highly polished surface ⁽²⁸⁻³⁰⁾ (**Figure 2-4**).



Fig. (2) Finished specimens of conventional PMMA acrylic resin.



Fig. (3) Finished specimens of PMMA modified by 5%Henna.



Fig. (4) Finished specimens of PMMA modified by Ag-Nps.

Testing procedures:

1- Surface Hardness Test:

Surface Micro-hardness of the specimens was determined using Digital Display Vickers Micro-Hardness Tester (Model HVS-50, Laizhou Huayin

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Testing Instrument Co., Ltd. China) with a Vickers diamond indenter and a 20X objective lens. A load of 200g was applied to the surface of the specimens for 20 seconds. Three indentations, which were equally placed over a circle and not closer than 2 mm to the adjacent indentations, were made on the surface of each specimen. The diagonals length of the indentations was measured by a built-in scaled microscope and Vickers values were converted into micro-hardness values ^(31,32). Micro-hardness was obtained using the following equation ⁽³²⁾.

Where **HV**: Vickers hardness in kgf/mm²; **F**: is the indentation load in kgf; and **d** is the arithmetic mean of the two diagonals in (mm).

2- Surface Roughness Test:

Specimens were photographed using a USB Digital microscope with a built-in camera (Scope Capture Digital Microscope, Guangdong, China) connected with an IBM compatible personal computer using a fixed magnification of 90X (**Figure 6**). The images were recorded with a resolution of 1280 x 1024 pixels per image. Digital microscope images were cropped to 350×400 pixels using Microsoft office picture manager to specify and standardize area of roughness measurement. The cropped images were analyzed using WSx-M software.

Within the WSx-M software, all limits, sizes, frames and measured parameters are expressed in pixels. Therefore, system calibration was done to convert the pixels into absolute real-world units. Calibration was made by comparing an object of known size (a ruler in this study) with a scale generated by the software.

Subsequently, a 3D image of the surface profile of the specimens was created. Five 3D images were collected for each specimen, in the central area and in the sides at an area of 10 μ m × 10 μ m ^(33,34). WSx-M software was used to calculate the average of heights (Ra) expressed in μ m, which can be assumed as a reliable index of surface roughness.

Statistical Analysis

All collected data were analyzed with one-way ANOVA analysis of variance using SPSS V20 SPSS Chicago, IL, USA.

RESULTS

1- Vickers Hardness:

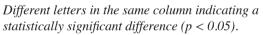
The statistical analysis results of the surface hardness of all tested groups exhibited that; there were *statistically significant* differences between all tested groups as indicated by One-way ANOVA test. It was found that the incorporation of Henna and Ag-Nps *significantly* decrease surface hardness of conventional heat-cured PMMA acrylic resin. Among the groups (fig.5); Tukey's pair-wise posthoc test showed a *non-significant statistical* difference (p>0.05) between modified groups. Therefore, both of Henna and Ag-Nps have the same adverse effect on hardness of heat-cured PMMA acrylic resin (**Table 1**).

 Table (1) Comparison of Vicker's hardness test

 results among all tested groups.

| Variables | Mean | SD | <i>P</i> -value |
|-----------|--------------------|------|-----------------|
| Group 1 | 33.85 ^A | 1.79 | |
| Group 2 | 30.63 ^в | 1.71 | 0.031* |
| Group 3 | 31.49 ^в | 2.16 | |

*; *significant* (*p* < 0.05).



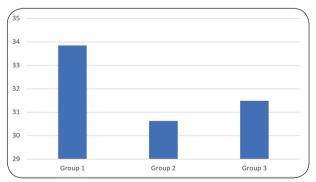


Fig. (5) Vicker's hardness test Mean values for all tested groups.

2- Surface Roughness:

The statistical analysis results of surface roughness of all tested groups (Fig.6) revealed that; there were *statistically non-significant* differences between all tested groups as indicated by One-way ANOVA test. It was found that the incorporation of Henna and Ag-Nps showed a *non-significant* decrease in surface roughness when compared to conventional heat-cured PMMA acrylic resin (**Table 2**).

Table (2) Comparison of surface roughness testresults between all tested groups.

| Variables | Mean | SD | <i>P</i> -value |
|-----------|--------|-------|-----------------|
| Group 1 | 0.2592 | 0.003 | |
| Group 2 | 0.2536 | 0.001 | 1 |
| Group 3 | 0.2563 | 0.002 | |

*; *significant* (*p* < 0.05).

Different letters in the same column indicating a statistically significant difference (p < 0.05).

Followed by Tukey's test. Statistical analysis was done with (SPSS Chicago, IL, USA) with a significant level at *P*-value <0.05.

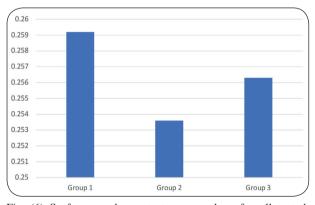


Fig. (6) Surface roughness test mean values for all tested groups.

DISCUSSION

PMMA resin has many advantages such as an excellent esthetic characteristic, low water sorption and solubility, adequate strength, low toxicity, easy repair, and a simple molding processing technique⁽³⁵⁻³⁸⁾. However, it has some disadvantages such as polymerization shrinkage, relatively low flexural strength, lower impact strength, and low fatigue resistance ⁽³⁸⁾. The failure of PMMA denture base material is most likely in the form of fracture either due to flexural fatigue or impact forces^(5,11,39,40).

Several attempts have been made to improve the mechanical, physical and biological properties of the acrylic resin ⁽⁴¹⁾. The aim of researches in these areas is to modifying the composition or reinforcing the PMMA with other stronger material for developing new material with better properties ^(11,41).

Chemical modification and the addition of some rubber-like substances, fibers, fillers, and nanoparticles (NPs) are different methods introduced and commonly used in an attempt to improve the mechanical and physical properties of PMMA-based materials ^(11,41-43).

Hardness is an important mechanical property most frequently used to characterize the wear resistance of the material that means; the material with higher surface hardness considered to be more wear-resistant ⁽⁴⁴⁾.

It is also, provides a possible indication of the abrasive resistance of the PMMA acrylic resin material which is the characteristic of the ease of finishing the material as it is resistant to in-service scratching during cleansing or handling ⁽⁴⁵⁻⁴⁷⁾.

Improvement of acrylic resin hardness is one of the researcher's concerns. Since; a recent study showed that the surface of complete denture bases must be polished to provide comfort to patients, as well as adequate aesthetics, prosthesis hygiene and low level of biofilm retention ⁽⁴⁸⁾.

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The results of surface hardness of conventional heat-cured PMMA acrylic resin significantly decreased after the addition of Henna and Ag-Nps. This may be due to the interference of Henna and Ag-Nps with monomer conversion which leads to incomplete polymerization process of PMMA acrylic resin, resulting in a large amount of residual monomer ^(15,49-51).

Additionally, it was found that there is no chemical reaction between "Ag-Nps / Henna" fillers and acrylic resin, hence, they can act as impurities within the resin matrix and subsequently weakening the materials and affecting their mechanical properties^(18, 52,53).

The surface roughness of denture base material is important as it affects the oral health of the tissues in direct contact with the denture ^(54,55). Surface texture can significantly increase or decrease microbial adhesion, colonization, and biofilm maturation as well as it can cause tissue microtraumas ^(56,57). According to **Consani et al.,** ⁽⁴⁷⁾ the lower surface roughness, the lower stain catching of acrylic denture base material.

The results of the current study showed that Henna and Ag-Nps fillers, as an additive to the heatcured PMMA acrylic resin, *insignificantly* reduced the surface roughness (Ra) of the acrylic resin specimens. This, may be due to lake of their chemical reaction with PMMA acrylic resin which allow the filler to scattered on the matrix surface and thus closed the gaps within the resin matrix ^(58,59). In addition, surface roughness concerned with the outermost layer of the specimen surface and measured only from the outer surface and not the inner surface of the acrylic resin specimen ⁽⁶⁰⁾.

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تأثير دمج الحناء والجسيمات النانوية الفضية على الخصائص السطحية لراتنج الاكريليك المعالجة بالحرارة

جمال مختار الوافي (1) ، مصطفى إبراهيم فياض (1,2), رامي عبد الله عبد الرحيم (3) ، أسامة أبو هلال (1)

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الملخص :

الهدف: هدفت هذه الدراسة إلى تقييم تأثير حشو الحناء والجسيمات النانوية الفضية على الخصائص السطحية لراتنج أكريليك البولي ميثيل ميثاكريلات .

المواد والأساليب: تم استخدام نوع واحد من راتينج الأكريليك المعالج بالحرارة التقليدي , الحناء بتركيز 1⁄2 بالوزن (الصناعة الخلية المصرية), والجسيمات النانوية الفضية بتركيز ٪0.5 بواسطة الوزن (شركة نانو جيت مصر) : تم اختيارهم لهذه الدراسة. تمت إضافة مواد الحشو الختلفة إلى مسحوق البوليمر وتم تقليبها بخلاط كهربائي قبل الخلط مع المونومر , والخلط , والتعبئة , والمعالجة باستخدام طريقة الحمام المائي التقليدي. بعد المعالجة , تم الانتهاء من جميع العينات وصقلها , ثم حفظها في الماء المقطر حتى اختبارها. تم استخدام جميع العينات المعدلة وغير المعدلة لقياس صلابة السطح وخشونة السطح. تم إجراء تحليل البيانات الإحصائية عبر برنامج SPSS

النتائج: إن دمج الحناء ووالجسيمات النانوية الفضية يقلل بشكل كبير من صلابة سطح ارتنجات أكريليك البولي ميثيل ميثاكريلات في حين أنها تقلل من خشونة السطح بشكل ضئيل.

الخلاصة : الصلابة السطحية لراتنجات أكريليك البولى ميثيل ميثاكريلات تتأثر سلبًا بتضمين الحشوات ، بينما تتحسن خشونة السطح.

الكلمات المفتاحية: لراتنجات أكريليك البولي ميثيل ميثاكريلات ، الحناء، الجسيمات النانوية الفضية, خواص السطح. الصلابة السطحية.