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WATER SORPTION AND SOLUBILITY OF MODIFIED HEAT CURED ACRYLIC DENTURE BASE MATERIAL WITH SILVER AND ZIRCONIA NANOPARTICLES

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ABSTRACT

Objective: Evaluation of water sorption and solubility of heat-cured acrylic denture base material incorporating silver nanoparticles (Ag-NPs) and zirconia nanoparticles (ZrO_2 -NPs). **Materials And Methods:** A total number of 18 specimens were used in this study**.** The specimens were divided into three groups (n=6); Group I (conventional heat-cured acrylic resin "powder and liquid" as a control group), Group II (0.5wt% Ag-NPs incorporated heat-cured acrylic resin powder mixed with commercial heat-cured acrylic resin liquid) and Group III (7.5 wt.% ZrO_2-NPs incorporated heat-cured acrylic resin powder mixed with commercial heat-cured acrylic resin liquid). The water sorption and water solubility were evaluated. One-way analysis of variance (ANOVA) was used to analyze the data. Statistical significance is established at the probability level of 0.05. **Results:** Although the control group (Group I) recorded the highest mean values of water sorption, and solubility, followed by Ag-NPs incorporated heat-cured acrylic resin (Group II) , there was no statistical significant difference among all groups**. Conclusion:** Modification of heat-cured acrylic resin with 0.05wt. % silver nanoparticles or with 7.5wt. % ZrO2 nanoparticles has no significant effect on the water sorption and solubility.

KEY WORDS: Water sorption, Solubility, Denture Base, PMMA, Nanoparticles, Zirconia Nanoparticles, Silver Nanoparticles.

INTRODUCTION

Polymethyl methacrylate (PMMA) has become the most commonly used material for the fabrication of dentures. It is the main component of the denture base material. PMMA has been used as a denture base material because of its desirable characteristics such as ease of processing, chemical stability, durability, moderate cost, lightweight, colour matching, and stability [1-4].

However, PMMA has the tendency to absorb water; this water absorbed by the acrylic resin can act as a plasticizer and cause softening, discoloration, and loss of mechanical properties of acrylic resin such as hardness, transverse strength, and fatigue limit [5,6]. Water sorption has detrimental effects on the colour stability of the denture base and can cause dimensional changes in denture base resin [7, 8].

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Solubility of PMMA, when immersed in an aqueous medium such as saliva, nasal secretion, water or cleansing agents, is of great importance. When PMMA is immersed in such solutions, plasticizers and other soluble components may leach out over extended periods and may cause several drawbacks in physical and mechanical properties [9].

Nanomaterials have many unique properties, such as ultra-small size, large surface area to mass ratio, extensive thermal stability, and high reactivity, so they have physicochemical properties different from bulk materials of the same composition [10].

Different types of nanomaterials like copper, zinc, titanium, magnesium, gold and silver have been developed and used in the dental field for their antimicrobial activity and the improvement of physical & mechanical properties [11].

Silver nanoparticles are widely used in the reinforcement of many dental materials because of their unique physical and mechanical properties such as ductility, electrical and thermal conductivity, antimicrobial activity, low toxicity, good biocompatibility with human cells and long-term antibacterial activity [12-15].

Zirconia is one of the biocompatible dental ceramics that improve mechanical properties. In addition, it has been widely used because it possesses good surface properties, biocompatibility and biological properties, thus making it a beneficial material in the reinforcement of dental materials [16].

Despite many researchers studying the effect of adding different nanoparticles on the properties of the resin materials, there is still little data available regarding the impact of adding silver and zirconia nanoparticles on water sorption and solubility of acrylic resin. Therefore, this invitro study aimed to evaluate the effect of silver and zirconia nanoparticles incorporation on water sorption and solubility of heat-cured acrylic denture base material.

MATERIALS AND METHODS

The materials used in this study were conventional heat-cured acrylic resin (Vertex^{tm} Rapid simplified), silver nanoparticles (Ag-NPs) with a concentration of 0.5wt. % and zirconia nanoparticles (ZrO_2-NPs) with a concentration of 7.5 wt. % (Nano-gate Company, Egypt).

A total number of 18 specimens were used in this study; the specimens were divided into three groups:

Group I: contained six specimens of conventional heat-cured acrylic resin as a control group.

Group II: contained six specimens of heat-cured acrylic resin modified with 0.5 wt % Ag-NPs.

Group III: contained six specimens of heat-cured acrylic resin modified with 7.5 wt. % ZrO_2-NPs.

Specimens' fabrication

Two stainless-steel metal plates were used; each one was milled into disk forms with 20 mm diameter and thicknesses of 1 mm. Each stainless-steel pattern was painted with a separating medium and then flasked into a metal flask using dental stone. After the complete set of dental stone, the top half of the metal flask and the stainless-steel metal pattern was removed, leaving spaces in the dental stone of the bottom half of the flask with the same dimensions of each stainless-steel metal pattern. Then, the mold was painted with a separating medium for the application of heat-cured acrylic resin of each group.

Group I specimens

The PMMA powder and monomer liquid were mixed according to the manufacturer's instructions (polymer/monomer ratio was 2.5:1 by weight) in a glass jar until reaching the dough stage. Then, the formed dough resin was packed in specially designed stone mold (that was formed through the previous step) after painting by a separating medium. Then, the two halves of flask were closed together and placed under hydraulic pressure, which

was slowly applied to the flask to get the flow of the resin dough throughout the mold space.

The mold was then immersed in a temperature-controlled curing water bath for six hours to allow polymerization. The curing cycle involved increasing the temperature to 60°C, over 1 hour and maintaining this temperature for 3 hours. After this time, the temperature was increased to 95°C, over an additional 2 hours to complete the heat polymerization cycle. The mold was removed from the curing bath and cooled slowly for 30 minutes at room temperature. The mold was then opened, and the samples were removed [17]. The samples were then trimmed using a tungsten carbide bur to remove any remaining small scratches, finished and polished.

Group II and Group III specimens

The nanoparticles powder used were Ag-NPs (for Group II) and ZrO_2-NPs (for Group III).

The silane coupling agent was added to nanoparticles to create reactive groups on the surface, which allow for adequate adhesion between nanoparticles and the resin matrix. To achieve this, 0.3 g of silane coupling agent was dissolved in 100 ml of acetone to ensure that it would evenly coat the surfaces of the nanoparticles.

Thirty grams of nanoparticles (Ag-NPs or ZrO2-NPs) were added to the solution, which formed from mixing of silane coupling agent and acetone, and stirred with a magnetic stirrer (Thermo Fischer Scientific, Waltham, MA, USA) for 60 min. Subsequently, a rotary evaporator was used to remove the solvent under vacuum at 60°C and 150 rpm for 30 min. When the nanoparticles were dried, they were heated at 120°C for two hours and naturally cooled to obtain the surface-treated nanoparticles.

The silanized Ag-NPs were measured with an electronic analytical balance device (Sartorius, Sartorius AG. Germany) and added a concentration of 0.5 wt. % into the PMMA powder. Similarly, the silanized ZrO_2-NPs were measured with the same device and added in a concentration of 7.5 wt.% into the PMMA powder.

Each modified powder was stirred with an electric mixer at a rotating speed of 400 rpm at room temperature for 30 seconds to obtain a more homogenous mixture and an equal distribution of the nanoparticles. Then, each modified powder was mixed with MMA liquid according to the manufacturer's instructions (polymer/monomer ratio was 2.5:1 by weight) in a glass jar until the dough stage. The preparation of the specimen was completed, as mentioned before.

Water Sorption and Solubility Test

The water sorption and water solubility were evaluated for all samples. After removing specimens from the molds, they were transferred to a desiccator containing silica gel maintained at 37°C to allow the materials to dry completely. The specimens were weighed daily using digital analytical balance until a constant mass was achieved (M1).

All specimens were transferred to separate glass vessels containing distilled water and stored in an incubator at 37°C for seven days. After water storage, the specimens were removed from the water, blot dried with an absorbent paper, waved for 15 seconds in the air and weighed until the maximum wet mass (M2) was obtained.

Then the specimens were placed in a desiccator containing silica gel and weighed daily to obtain a constant dry mass (M3) to determine the mass loss. The water sorption and solubility (mg/mm^3) were calculated for each specimen using the following equations [18]:

$$\text{Water sorption} = M2 - M3/V$$

$$\text{Solubility} = M1 - M3/V$$

Where; M1: the mass of the specimen (μg) before immersion in water, M2: the mass of specimen (μg) after immersion in water, M3; the mass of specimen (μg) after immersion and desiccation, V: the volume of the specimen (mm^3).

Volume of the specimen (mm^3) was calculated using the following equation: $V=\pi r^2 h$

Where π: 3.14, r: radius of the specimen (mm), and h: the thickness of specimen (mm).

Data were collected, tabulated and statistically analyzed using SPSS ® statistics software version 20. Numerical data were described as mean and standard deviation. Data were compared using the one-way-ANOVA test. The level of significance was set at $P<0.05$.

RESULTS

Water sorption test:

The informative statistical analysis showing mean values and SD of water sorption test results measured in (μg/mm3) for all tested groups (Table 1).

The statistical analysis of water sorption of all tested groups revealed that; there was a non-statistical significant difference between all tested groups as indicated by the One-way ANOVA test (F=2.782, P=0.10166).

The control group "conventional heat-cured PMMA group" (Group I) recorded the highest mean value of water sorption (1.321±0.094 $\mu g/mm^3$); followed by PMMA/Ag-Nps group (Group II) (1.224±0.090 $\mu g/mm^3$).

TABLE (1) Comparison of water sorption test results among all tested groups.

Variable	Mean ±SD	ANOVA	
		F	P-Value
Group I	1.321±0.094		
Group II	1.224±0.090	2.782	0.10166 ns
Group III	1.187±0.091		

**; significant (p< 0.05) ns; non-significant (p>0.05*

Water solubility test:

The informative statistical analysis showing mean values and standard deviation of water solubility test results measured in $\mu g/mm^3$ for all tested groups (Table 2).

The statistical analysis of water solubility of all tested groups revealed that; there was a non-statistical significant difference between all tested groups as indicated by the One-way ANOVA test (F=0.2538, P=0.7798).

The highest mean value of water solubility was recorded with the control group "conventional heat-cured PMMA group" (Group I) (1.273±0.329 $\mu g/mm^3$); followed by PMMA/Ag-Nps group (Group II) (1.222±0.302 $\mu g/mm^3$).

TABLE (2) Comparison of water solubility test results among all tested groups.

Variable	Mean± SD	ANOVA	
		F	P-Value
Group I	1.273±0.329		
Group II	1.222±0.302	0.2538	0.7798 ns
Group III	1.135±0.300		

**; significant (p< 0.05) ns; non-significant (p>0.05)*

DISCUSSION

Complete or partial dental prostheses usually have acrylic resin bases to enhance prosthesis support and keep the artificial teeth in position [19]. These bases contribute to aesthetics and functional rehabilitation and may interfere in patient acceptance of the service [20].

Acrylic resin is considered a good base material because it is cheap, maybe relined, has a good aesthetic appearance and low density [21].

Today's, the most frequently preferred denture base material is the heat-cured PMMA based acrylic resin [22]. However, it has some relatively

poor mechanical properties that affect the dentures' longevity [23-25]. Dentures will deteriorate after years of use due to constant exposure to external stresses caused by poor fit [25].

Several attempts have been made to improve the properties of the denture base material, such as the chemical modification of PMMA or reinforcement with other materials, such as fibers, and use of macro-and nano-fillers [26].

Recently, studies have investigated the effect of incorporating inorganic nanoparticles into PMMA to improve its properties [27-29]. The size, concentration, and interaction of these nanoparticles with a polymer matrix determine the properties of a polymer nanocomposite mixture [28].

Nanomaterials are known for their superior characteristics compared to conventional ones[28,29]. When these nanomaterials are incorporated into a polymer matrix as fillers, hopeful features of both constituents could integrate to improve the properties of the polymer [29].

Silver nanoparticles are among the most commonly used nanoparticles because of their ductility, electrical and thermal conductivity, and antimicrobial activity [30-32]. Therefore, incorporating silver nanoparticles into acrylic resin denture base materials was developed to enhance physico-mechanical properties [32].

Recently, zirconia nanoparticles received great attention because of their excellent biocompatibility and white colour, making it less likely to change the aesthetics than other metal nanoparticles such as silver [33].

The selection of 0.5 wt. percentage concentration for Ag-NPs and 7.5 wt. % concentration for ZrO_2-NPs, added to conventional heat-cured PMMA, is based on the previous studies, which claimed that these concentrations could be used to minimize the possible unfavourable changes in mechanical and optical properties of the denture acrylic base [32, 33]. In addition, it is based on the literature available, which supports that these concentrations are biocompatible and non-toxic to the patient [34, 35].

Good adhesion of nanoparticles with the resin matrix effectively enhance the polymer/ nanoparticles resin properties [16]. Therefore, surface modification of nanoparticles was carried out in the present study with a silane coupling agent of 97% 3-trimethoxysilyl propyl methacrylate solution that might reduce aggregation of nanoparticles and then enhance their compatibility with the polymer, which may result in the enhancement of resin properties.

The acrylic resin test specimens were prepared according to the ADA specification for heat-cured denture base acrylic resin to standardize the dimensions for all the samples during testing procedures [36].

Water sorption is determined depending on the mass increase in the unit volume, while water solubility is determined depending on the mass loss in the unite volume [37].

Since, PMMA acrylic resin absorbs water slowly in the long term due to the hydrophilic nature of PMMA [38] so, the period of keeping the specimens in water is very important. For this reason, the specimens were kept in distilled water for seven days to equalize the differences considered to exist among all tested specimens [39].

This study showed a non-significant decrease in water sorption and water solubility of the PMMA/ Ag-NPs and PMMA/ZrO2-NPs modified groups compared with the control "unmodified group".

According to the ISO 20795-1 standard, 32μg/mm^3 is the threshold value for water sorption, and 1.6 μg/mm^3 is the threshold value for water solubility of heat-cured acrylic resins [17].

The results of this study showed that the water sorption and water solubility values in all test

groups were within clinically acceptable limits as they recorded (1.321µg/mm^3,1.224µg/mm^3, and 1.187µg/mm^3) for water sorption, and (1.273 µg/mm^3, 1.222 µg/mm^3, and 1.135 µg/mm^3) for water solubility for group I , group II and group III respectively.

The decrease in water sorption and solubility in PMMA/Ag-Nps modified group (Group II) may be due to the polar interactions between the nanoparticles and C=O of the polymer matrix (weak interactions), which are adequate at low concentrations. Also, the metal nanoparticles may bond with the polymer molecules, crosslinking them between each other, thereby increasing the "pseudocrystallinity" of the polymer and forming an ordered system[40].

The decrease in water sorption and solubility in the PMMA/ZrO2-Nps modified group (Group III) can be explained in several ways. During the poe lymerization process of acrylic resins, porosity or microvoids can occur among polymer chains. A high porosity level or microvoids has been shown to facilitate fluid transport into and out of polymer by serving as sites for molecules to be sequestered and leading to enhanced solvent uptake and elution. Zirconia nanoparticles are insoluble in water and could reduce the overall volume of the absorbing polymer. The second factor could be the silane coupling agent used to treat the nanoparticles. The nanoparticles-resin interface provided by the silane coupling agent could reduce the amount of water that reached the inner layers of the polymer matrix [41].

CONCLUSION

Within the limitations of this study, it can be concluded that the incorporation of silver and zirconia nanoparticles has an insignificant effect on water sorption and solubility of heat-cured acrylic resin.

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