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EVALUATION OF FLEXURAL STRENGTH, MICROHARDNESS, AND COLOR STABILITY OF POLYAMIDE AND HEAT-CURED ACRYLIC RESINS MODIFIED WITH TITANIA NANORODS

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ABSTRACT

Objectives: The present study was designed to evaluate flexural strength, microhardness, and color stability of polyamide and conventional heat-cured acrylic resins after the addition of titania nanorods.

Methods: Polyamide (Group I) and heat-cured acrylic (Group II) resins were used in this study and both were modified with titania nanorods at different concentrations as follows: 0 wt%, 0.1wt%, 0.5wt%, 1 wt% for the subgroups A, B, C, D respectively. Flexural strength, microhardness, and color stability have been investigated for all specimens of Group I and II. The data were statistically analyzed by one-way ANOVA and Tukey's HSD analysis at a significance factor of $\alpha = 0.05$.

Results: For the flexural strength and microhardness test the highest value was for heat-cured acrylic resin specimens modified with 1% titania nanorods (Group II subgroup D), while the lowest value was for polyamide specimens without modification (Group I subgroup A). For color stability, the lowest color change value was for polyamide specimens modified with 0.1% titania nanorods (Group I subgroup B), while the highest color change value was for heat-cured specimens modified with 1% titania nanorods (Group II subgroup D), all color changes were in the acceptable range (slight change).

Conclusions: Within the limits of this study, titania nanorods shave increased both flexural strength and microhardness of both polyamide and heat-cured acrylic resins as the concentration increase. Titania nanorod shave to affect the color stability of heat-cured acrylic resin more than that of polyamide resin as the concentration increase. Heat-cured acrylic resins showed higher flexural strength and microhardness than polyamide ones.

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INTRODUCTION

Denture bases can be fabricated using various materials such as polymethylmethacrylate (PMMA) that is commonly used, however, in recent years other materials than PMMA such as polyamides (nylon), polycarbonate, acetal resins were developed and these materials are processed by thermoplastic and are suitable for nonmetallic clasps and short-term dentures.[1] Nowadays esthetics for patients is of importance. Consequently, denture basesfabricated from materials that are processed by thermoplastic technology in place of traditionally used heat-cured acrylic resins have been introduced as another method of treatment to eliminate undesirable metal exposure.[2] Polyamide exhibits high flexibility, so it can prevent gum from being rubbed leading to better chewing efficiency and increased capability of undercuts engagement. It has high impact strength and high biocompatibility since it is free of monomer and metal, so gingival inflammation.[3] It has a low modulus of elasticity so it cannot be used as occlusal rests or rigid parts such as the major connector of a removable denture.[3] Polyamides are used primarily for temporary applications and tissue-supported removable dentures and where more comfort, higher flexibility, and impact strength are requested, also cases where there is monomer allergy.[1] It is frequently designated for definite clinical situations where flexibility is preferred such as protuberance, extremely bulging alveolar processes, protuberance, and particularly in the maxillary anterior area. Polyamide is also useful for patients with bruxism and compromised neuro-motor function. Moreover, it could be used as obturators and for rehabilitating the anomalies such as ectodermal dysplasia.[4,5] However polyamide is contraindicated for the unilateral distal extension, low vertical dimensions and closed bites.

Prosthetic esthetic appearance is an important request for the satisfaction of patients. Furthermore, color stability is an essential clinical property, and color changes may be a sign of material aging or damage.[6,7] Many reasons can result in discoloration such as; ingredients dissolution, surface roughness, accumulation of stains, watersorption.[8] PMMA have low impact strength, also flexural one isn't optimal.[9,10] Denture bases fracture represents a challenge and is considered the main problem. Repeated masticatory forces lead to denture base fracture that frequently takes place at the midline due to flexural fatigue failure caused by repeated deformation of denture base.[11-13] Flexural strength of denture base materials is commonly evaluated by three-point bend test since it simulates stress type that the denture base subjected to it during mastication.[13,14] Hardness of denture base materials is also a necessary mechanical property that indicates the resistance of denture base materials to scratching and irreversible deformation.[15,16] Hardness affects finishing, polishing that reduces scratches which by its role can lead to decreased fatigue strength and produce premature failure. Finishing and polishing are extremely important to achieve ideal esthetics and good oral hygiene.[15,17] The microhardness of denture base resin's surface is directly interrelated to their longevity, where the superior values of their microhardness, the greater resistance of it to wear, abrasion and cracking of the denture base material. So it is of importance to be evaluated.[16,18]

Nano is a Greek word (nanus) meaning dwarf, one nanometer is a unit of length equal to one billionth of a meter. Materials in the nanoscale have different properties from that in molecular form. They have a much greater surface area that leads to enhanced surface energy which in turn affects the interaction of the elements and nanoparticles. Nanomaterials have also different biological, chemical, and physical properties including color, thermal, electrical, and properties that differ from the microscaled materials.[19,20] TiO_2 nanoparticles are progressively used due to their good properties such as lack of toxicity, chemically inertness, not expensive, antibacterial effect, high microhardness and resistance to corrosion.[21,22]

MATERIALS AND METHOD

Specimens grouping

A total number of 120 specimens were prepared and divided into two main groups: Group I: 60 specimens constructed of the polyamide resin (Dentiflex, Multipress injection system, Rokodent, Poland), Group II: 60 specimens were made up of heat-cured acrylic resin (Vertex-Dental. Johan van. Olden-bamevelten, Netherlands). These two groups were modified with titania nanorods (Prepared in national research center-Cairo-Egypt) at different concentrations. Each group was divided into four subgroups according to the concentration of titania nanorods as follows**:** 0 wt%, 0.1wt%, 0.5wt%, 1 wt% for the subgroups A, B, C, D respectively.

Preparation of Titania nanorods

A hydrothermal process was employed for the synthesis of TiO_2 nanorods using a chemical process. Briefly, anatase TiO_2 white powder was placed into a Teflon-lined autoclave. NaOH aqueous solution was added up to 80% of the total volume of a sealed stainless steel tank maintained at 200 °C for 24 hrs in the autoclave without shaking or stirring during heating. After heating in an autoclave, the specimens were cooled to room temperature and washed with an aqueous solution of dilute HCl, deionized water, and absolute ethanol several times. Specimens drying was done at 70°C for 6 hrs. Finally, the soft white-colored fibrous powder was produced.[23] The morphologies of titania nanorods were analyzed with a transmission electron microscope (TEM) at different magnifications as shown in Figure 1.

Mixing titania nanorods with polyamide & heat-cured acrylic resin

Titania nanorods were added to grains of flexible resin cartridges with concentrations: 0%, 0.1%, 0.5%, and 1% within polyamide manipulated according to manufacturer's instructions at 265^0C, specimens are shown in Figure 2. Heat cured acrylic resin specimens were prepared by mixing titania nanorods with the powder of acrylic resin. After they have been mixed, the addition of monomer was applied following the manufacturer's instructions as shown in Figure 3.[24,25]

Flexural strength

A total number of 40 rectangular-shaped specimens (20 for each type of resin) were prepared according to the ISO 1567 with the dimensions: 64 × 10 × 2.5 mm^3. The flexural strength of each specimen was measured through a 3-point bending strength test using an Instron universal testing machine (WDW-100D, Dahometer, USA). A crosshead speed of 5 mm/min was applied with a distance of 50 mm between the supports. The load was applied perpendicular to the center of the specimen until fracture occurred. Flexural strength was calculated using the following equation:[26]

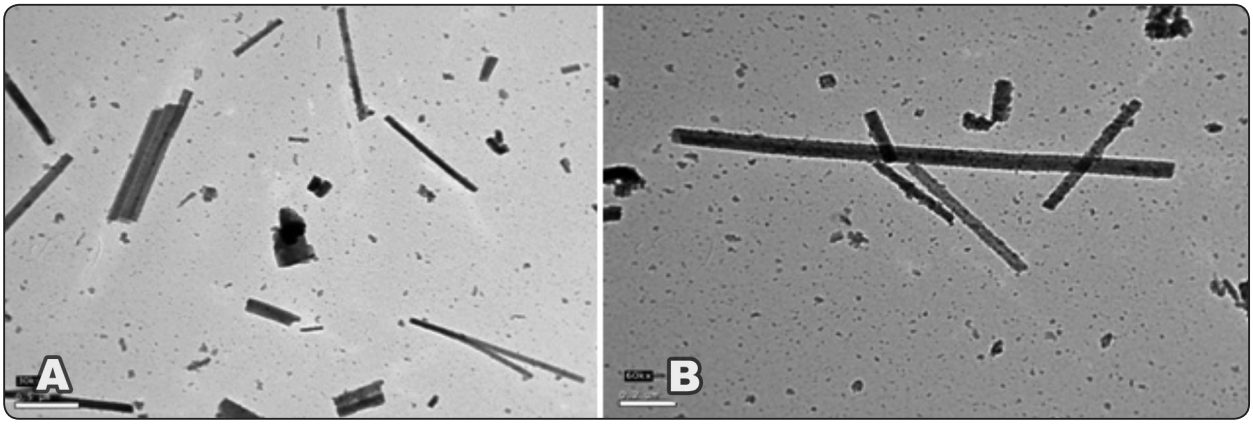

Fig. (1) Titanianorods by TEM at magnification powers of 30kx (A), 60 kx (B)

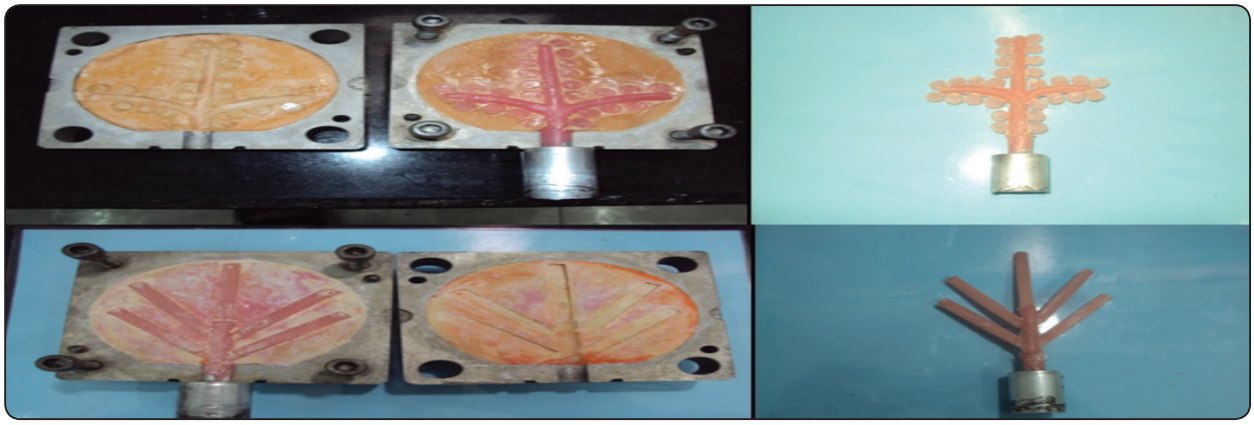

Fig. (2): Specimens of polyamide resins

Fig. (3): Specimens of heat-cured acrylic resins

$$FS=3FL/2bd^2$$

where; FS is the flexural strength in (MPa), F is the load or force at fracture in (N), L is the span length of the specimen between two supports (50 mm), b is the width (10 mm), and d is the thickness (2.5 mm).

Microhardness

A total number of 40 disk-shaped specimens (20 for each type of resin) with dimensions: 13 mm diameter × 2mm thickness were prepared. The Vickers hardness test was employed to measure surface hardness by using an indenter point in the shape of a square-based pyramid. The test was performed using a microhardness tester (Micromet 2001, Model 1600-4981, Buehler, USA) manufacturing with an applied load of 2.942 N at a 15-s dwell time at room temperature. For each specimen, three Vickers hardness indentations were made at different points along with the specimen. The mean hardness was calculated and further used for the statistical analysis.[27]

Color stability

A total number of 40 disk-shaped specimens (20 for each type of resin) were prepared in a Teflon mold with the dimensions of 13 mm diameter × 1 mm thickness. The color of the specimens was determined using the datacolor 3881 spectrophotometers (Shimadzu-Spectrophotometer, Japan). Ultraviolet scanning was carried out at a wavelength of 380-780 nm to record specimens color (CIE L*,a*,b* system) that is a three-

dimensional color coordinates and the color change was calculated according to the following formula as reported by **Gurdal P, et al**.[28]

$$\Delta E = [\ (\Delta L)2 + (\Delta a)2 + (\Delta b)2\ ]^{1/2}$$

Where ΔE refers to color change, ΔL refers to the lightness coordinate and its value ranges from 0 for perfect black to 100 for perfect white., Δa refers to the chromaticity coordinate in the red-green axis, Δb refers to the chromaticity coordinate in the yellow-blue axis. To better correlate ΔE to the clinical implications according to the National Bureau of Standards (NBS), the ΔE values were converted to NBS units by the following formula:

NBS unit = ΔEx0.92

The values of NBS were interpreted as follows: A value of (0-0.5) of NBS units is considered as an extremely slight color change, (0.5-1.5) is a slight change, (1.5-3.0) is a perceivable change, (3.0-6.0) is an appreciable change, (6.0-12.0) is much appreciable and above 12.0 is considered a change to another color.[29]

Statistical Analysis

It was carried out using a statistical package for the social sciences (SPSS) version 20. All data were investigated by one-way ANOVA and Tukey's HSD test for pairwise comparison with a significance factor of $\alpha = 0.05$.

RESULTS

Regarding the flexural strength test, Table 1 showed that for group I there was a significant difference between subgroup D and all subgroups, however, there was no significant difference between other subgroups. As shown in Table 2 for group II there was a significant difference between subgroup D and subgroups A and B, however, there was no significant difference between subgroups A, B, and C, also between subgroups C and D.

Concerning the microhardness test, as demonstrated in Table 3 for group I there was a significant difference between subgroup D and subgroups A and B, however, there was no significant difference between subgroups A, B, and C, also between subgroups C and D. Table 4 showed that for group II there was a significant difference between subgroup D and subgroups A and B, also between subgroup C and subgroups A and B, finally between subgroups C and D, however, there was no significant difference between subgroups A and B.

For the color stability test, as revealed in Table 5 for group I there was a significant difference between subgroup B and subgroups C and D, however, there was no significant difference between subgroups C and D. Table 6 showed that for group II there was a significant difference between all subgroups.

TABLE (1): Means and standard deviations (SD) of Flexural strength (MPa) of Group I

Subgroups	Mean	SD	F value
Sub group A	85.3[a]	1.35	11.092
Sub group B	85.7[a]	1.60	
Sub group C	87.1[a]	1.78	
Sub group D	90.9[b]	2.04	

Means with similar superscript capital letter in one row and similar superscript small letter in one column are not significantly different (P >0.05).

TABLE (2): Means and standard deviations (SD) of Flexural strength (MPa)of Group II

Subgroups	Mean	SD	F value
Sub group A	122.4[a]	1.74	8.51
Sub group B	123.3[a]	1.68	
Sub group C	124.8[ab]	1.64	
Sub group D	127.5[b]	1.76	

Means with a similar superscript capital letter in one row and similar superscript small letter in one column are not significantly different (P >0.05).

TABLE (3): Means and standard deviations (SD) of microhardness (HV) of Group I

Subgroups	Mean	SD	F value
Sub group A	12.92[a]	0.34	6.31
Sub group B	13.02[a]	0.38	
Sub group C	13.32[ab]	0.58	
Sub group D	13.98[b]	0.31	

Means with a similar superscript capital letter in one row and similar superscript small letter in one column are not significantly different (P >0.05).

TABLE (4): Means and standard deviations (SD) of microhardness (HV) of Group II

Subgroups	Mean	SD	F value
Sub group A	18.28[a]	0.51	32.85
Sub group B	18.46[a]	0.58	
Sub group C	19.34[b]	0.42	
Sub group D	20.92[c]	0.30	

Means with a similar superscript capital letter in one row and similar superscript small letter in one column are not significantly different (P >0.05).

TABLE (5): Means and standard deviations (SD) of color stability(ΔE)of Group I

Subgroups	Mean	SD	F value
Sub group B& control	1.04[a]	0.05	19.84
Sub group C& control	1.2[b]	0.07	
Sub group D& control	1.3[bc]	0.07	

Means with a similar superscript capital letter in one row and a similar superscript small letter in one column are not significantly different (P >0.05).

TABLE (6): Means and standard deviations (SD) of color stability (ΔE) of Group II

Subgroups	Mean	SD	F value
Sub group B& control	1.24[a]	0.05	80.08
Sub group C& control	1.62[b]	0.08	
Sub group D& control	1.94[c]	0.11	

Means with a similar superscript capital letter in one row and a similar superscript small letter in one column are not significantly different (P >0.05).

DISCUSSION

PMMA possesses many benefits such as an outstanding esthetic, low sorption of water and solubility, sufficient strength, low toxicity, ease of repair, and simplicity of technique of molding. However, it has some drawbacks such as polymerization shrinkage, lower impact strength, flexural strength, and resistance to fatigue which bring about denture failure during chewing or dropping from the hand of the patient. Therefore enhancement of some properties of PMMA has been introduced recently e.g. metal wires addition, fibers, and chemical structure modification. Polyamide payed attention as a material of denture base in the current years. Polyamide resin was introduced as a material of denture base as an alternative to conventional PMMA resins.[30] Nanotechnology is now the most encouraging field to generate new applications in health care including dentistry. Nanoparticles of metal oxide are one of the most used applications of nanotechnology in dental materials such as denture bases, dental restorative materials, and dental implants.[19,20] In our study, the used resins were modified with titania nanorods at different concentrations as follows: 0 wt%, 0.1wt%, 0.5wt%, 1 wt% and were measured for flexural strength, microhardness, and color stability.

It is well known that nanoparticles addition is of significant importance nowadays on several properties. To the materials that are added. Many studies have revealed the effect of titanium dioxide

(TiO_2) addition on PMMA properties. Flexural failure of denture bases is regarded as one of the main forms of failure.[31] In our study, flexural strength has been increased by increasing titania nanorods concentration in polyamide and heat-cured acrylic resin, this is in agreement with **Karci M et al.,**[32] who concluded that the addition of 1% TiO_2 to PMMA was the ideal concentration that could increase its flexural strength. **Shibata, T et al**[33] alsorevealed that adding titania has increased flexural strength due to the distribution of TiO_2 nanoparticles in the matrix of PMMA that negatively affects the conversion degree leading to raising in the amount of residual monomer that plays the role as a plasticizer. Additionally, **Harini Pet al.**[34] have found that adding TiO_2 particles have improved the flexure strength of PMMA. On the contrary, **Sodagar et al.**[22] have found that TiO_2 additives act as impurities, especially with conventional acrylic resin. Moreover, other studies have found that TiO_2 did not enhance the PMMA flexural strength, which might be as a result of particles clustering within the matrix of resin, leading to its decrease in its strength, also modifying PMMA with TiO_2 nanoparticles causes a reduction in flexural strength.[35-37]

The microhardness measurements obtained in this study showed higher values of modified subgroups than the unmodified ones. This study was in agreement with **Naji SA et al**,[38] which revealed that microhardness of acrylic resin was enhanced with titania nanotubes addition as the concentration increased. **Porras et al.**[39] have investigated the hardness of polyethylene chitosan composite reinforced with titania nanotubes and it was revealed that well-dispersed nanotubes have enhanced the hardness of this composite. It was found that adding TiO_2 particles could improve the hardness of PMMA.[35] It is notable that the filler material used for reinforcing is supposed to enhance the mechanical properties without resulting in undesirable effects on the esthetics.[40,41] Titania nanorods have white color, so the suitable percentage of it that accomplish these requirements should be taken into consideration. It was found in this study that the specimens of conventional heat-cured acrylic resin were more affected by the white color of titania than polyamide specimens. **Aziz HK**[40] evaluated the color change after adding 3wt% of titania nanoparticles and they found that the increase in the amount of light absorbed made the specimens that were reinforced more opaque compared to non-reinforced ones and this was as a result of titania nanoparticles presence within the matrix, that leads to more absorption of light than matrix as aresult of high atomic number.[40] Further studies are needed to establish the appropriate titania nanoparticles percentage required to achieve a PMMA/TiO_2 nanocomposite with enhanced properties and acceptable aesthetics.

CONCLUSIONS

Within the limits of this study, titania nanorods shave increased both flexural strength and microhardness of both polyamide and heat-cured acrylic resins as the concentration increase. Titania nanorods have to affect the color stability of heat-cured acrylic resin more than that of polyamide resin as the concentration increase. Heat-cured acrylic resins showed higher flexural strength and microhardness than polyamide ones

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