

## CORRELATION BETWEEN THE WATER SORPTION AND THE FLEXURAL STRENGTH IN HEAT-CURED ACRYLIC RESIN WITH DIFFERENT FILLERS

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### ABSTRACT

**Purpose:** The purpose of this study was to correlate the water sorption and flexural strength of acrylic resin reinforced with three different fillers.

**Materials and Methods:** A Flexural strength test using a universal testing machine was done for 132 heat-cured acrylic resin specimens reinforced with titanium oxide nanoparticle, aluminum oxide nanoparticle, silanated, and non silanated glass fibers (n=12). Another 132 discs with the same fillers were evaluated for water sorption at  $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$  using a four-digit electric balance. Specimens were stored in distilled water for 120 days. Statistical analysis was done using Pearson correlation ( $p < 0.05$ ) to correlate the water sorption and the flexural strength at each nanoparticle concentration.

**Results:** The correlation between the water sorption and the flexural strength was positive for the titanium oxide and aluminum oxide nanoparticles, and negative for the glass fibers specimens. However, these correlations were non-significant.

**Conclusion:** Water sorption of heat-cured acrylic resin has no effect on the flexural strength with the addition of titanium oxide nanoparticle, aluminum oxide nanoparticle, silanated and non silanated E-Glass fiber.

**KEYWORDS:** Flexural strength, Glass fibers, Heat cured acrylic resin, Nanoparticles, Water sorption.

### INTRODUCTION

Acrylic resin polymers are commonly used in prosthodontics as a denture base material due to their ease of processing, cost efficiency, lightweight, and ability to match the oral mucosa with color.

However, this material is not ideal in its properties. Different fillers have been incorporated into it to improve its physical and mechanical properties<sup>(1)</sup>. Fiber-reinforced composites are commonly used in dentistry as periodontal splints, partial fixed

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dentures, endodontic posts, and orthodontic tools. The fiber reinforcement offers better biomechanical efficiency for the composite structure due to its superior tension and flexure properties. Due to the chemical resistance and low cost, the E-glass fiber is currently the most commonly used glass filler in dentistry <sup>(2)</sup>. Reinforcement of complete denture with 4.3 % E-glass fiber mesh has been reported to improve the acrylic denture base's mechanical properties compared to the metallic one. The glass mesh increased the fracture load by 125%, while the metal mesh raised it by just 109%<sup>(3)</sup>. Finite element analysis showed that the glass fiber reinforced denture base behaves as same as the metallic base when used as an implant overdenture <sup>(4)</sup>.

Nanotechnology has many daily life applications extended to the medical and dental field due to their unique structures and physical, mechanical, and biological properties. They are used in implants, bleaching agents, caries preventive agents, restorative materials, and bone substitutes <sup>(5)</sup>. Aluminum oxide is commonly used in dentistry as a sandblasting material, removing casting investment materials and increasing the surface area to enhance porcelain or cement's mechanical retention strengths <sup>(6)</sup>. It has been reported that the inclusion of aluminum oxide powder by weight from 5 % to 20% in the heat-cured denture base resin results in an improvement in the flexural strength and thermal diffusivity <sup>(7)</sup>. The initial use of titanium in dentistry has been started in the '60s. Titanium is a metallic element known for its several attractive characteristics such as biocompatibility, excellent corrosion resistance, and high mechanical strength. It provides a favorable biological response when used in contact with the living tissues. It is used in implants, crowns, and partial denture frameworks <sup>(8)</sup>. Titanium oxide has improved the mechanical properties and the elastic modulus and hardness of the composite resins (BisGMA/TEGDMA) by increasing the degree of vinyl resin conversion by 7%. The shear bond strength to the dentin bonding agents was enhanced by adding titanium oxide

nanoparticles <sup>(9)</sup>. The null hypothesis that there is no correlation between the flexural strength and water sorption with the addition of glass fiber, titanium oxide, and aluminum oxide nanoparticles.

## MATERIALS AND METHODS

### Sample grouping

A power analysis was performed on a previous study [10] using G\*Power software (v3.1.9.2) to calculate the sample size. The power value was 95%, and the  $\alpha$  level of significance was 0.05. The results showed an effect size of  $f = 0.78$ , no centrality parameter of 14.62, and a critical F of 4.3. The calculated number of specimens for each group was 12. The samples were classified into a control group free from nanoparticles, and another ten groups according to the type of nanofiller (Table 1). Nanoparticles were prepared in a laboratory (Nanotech laboratory, Giza, Egypt) by sol-gel method, followed an electron microscope analysis and X-Ray diffraction analysis to confirm the size of nanoparticles (<20 nm). The nanoparticles were added to the heat-cured acrylic resin monomer with the desired concentration by volume %.

Table (1): Different groups in this study according to the type of nanoparticle

Group No	Type of Nanoparticle filler
Control Group	No nanoparticles
Group I	1% Aluminum-oxide nanoparticles
Group II	5% Aluminum-oxide nanoparticles
Group III	1% Titanium nanoparticles
Group IV	5% Titanium nanoparticles
Group V	5% Glass fiber
Group VI	5% Silanated glass fiber
Group VII	10% Glass fiber
Group VIII	10% Silanated glass fiber
Group IX	15% Glass fiber
Group X	15% Silanated glass fiber

### Preparation of specimens

A metallic mold was prepared to standardize the size of the wax pattern of all samples. Wax pattern samples with the specified dimensions were prepared and coated by a separating medium and then invested by plaster in a flask. After setting, the wax was eliminated by immersion of the mold in boiling water for 4 minutes. Heat cured acrylic resin (Vertex Regular, Vertex Dental B.V, Soesterberg, Holland) was measured according to the manufacturer's instructions. The polymer ratio to the monomer containing nanoparticles was (3:1) by volume. The E-glass fiber (Al-Ahram Glass Fiber, Al-Qalyubia, Egypt) was measured and added to the acrylic resin powder in Group V, Group VII, and Group IX. The powder of the glass fiber was coated with the silane coupling agent (Porcelain primer Pre-Hydrolyzed Silane primer, Bisco, Schaumburg, Illinois) before addition to the powder in Group VI, Group VIII, and Group X. The material was mixed until reaching the dough stage then packed inside the mold. The material was cured by inserting the flasks in a 75° C water bath for 2 hours. Then, the temperature was raised to 100°C for 1 hour and 30 minutes (Fig 1). After the setting of the specimens, they were removed from the mold for finishing and polishing.

### Flexural strength measurement:

A Total number of 132 specimens were prepared (n=12). The samples were rectangular with a dimension of 65mm in length, 10mm in width, and 3.3 mm in thickness. They were stored in distilled water 37°C ± 1°C for 120 days in multiple separate containers after their construction. The samples were removed from the water, then blot-dried using a filter paper, and waved in air for 15 seconds to remove any apparent moisture. The samples were mounted at the universal testing machine's horizontal support with a span length of 50 mm between the support rods. Specimens were loaded by 5kN load at the center using a computer-

controlled universal testing machine (Model LRX plus, Ametek instruments, Berwyn, Pennsylvania, USA) with a crosshead speed of 5 mm/min. The data was recorded using computer software (Nexygen-MT; Lloyd Instruments, Sussex, United Kingdom). The calculation of Flexural strength (FS) in Mpa was done using the following formula:  $FS = 3F(L)/2wh^2$  Where; F is the maximum load at the point of fracture, L is the span length between support, w is the width of the sample, and h its height.

### Water sorption measurement

Another 132 discs were prepared (n=12) with 50±1 mm diameter and 2±0.1 mm thickness in the same manner as the rectangular specimens. Following their construction, the disks were removed from the mold with caution. Any flashes were cut away using a sharp blade (# 15) to avoid loose particles from being attached to the samples during weighting or immersion. The disks were put into a glass desiccator containing dehydrated silica gel (Desi-Pak, Sud Cheme, Belen, New-Mexico, USA) at 37° C ± 2°C for one hour at the room temperature. The disks were weighed using an electronic balance with four digits precision (BS150, ST Instruments. Taiwan). This process was repeated until a constant value of weight was found. Then, each specimen was stored in distilled water at 37°C ± 1°C for 120 days in multiple separate containers. The samples were removed from the water, blot-dried using filter paper, and waved in air for 15 s to remove any apparent moisture. The final weight was recorded after 1 minute from the time of removal from water. Water sorption was calculated by the weight changes in percent (%). The water sorption percentage was computed as follows:  $\text{Water sorption}\% = (\text{weight gained} - \text{original weight}) / \text{original weight} \times 100$ .

### Statistical Analysis

The results were analyzed for normality using the Kolmogorov Smirnov test. Data showed normal

(parametric) distribution. The data presented as mean and standard deviation (SD) values. Pearson correlation was done to correlate the flexural strength and the water sorption at the different nanoparticle concentrations. The significance level was set at  $P \leq 0.05$ . Statistical analysis was performed with IBM SPSS© Statistics for Windows, Version 20.0. (Armonk, New-York: IBM Corp.)

## RESULTS

The flexural strength and water sorption results are shown in table (2) and figure (1). The highest flexural strength value was in Group IX, and the

lowest flexural strength value was in Group II. All specimens showed a lower flexural strength value than the control group except in Group IX. All the results of water sorption were lower than the control group except in Group II. The highest amount of water sorption was in group II, and the least amount was in Group VIII. Pearson correlation for the glass fibers showed a weak-positive correlation between the control group and groups (V, VI, VII, and VIII). In both groups (IX, X,) the correlation was moderately positive. The correlation was negatively strong in groups II and III and weak in group I and IV. All these correlations in this study were found to be non-significant ( $p < 0.05$ ) (Table 2).

TABLE (2): Mean and SD of flexural strength and water sorption with Pearson correlation in all groups.  $p (< 0.05)$

Group	Number of specimens for each test	Flexural strength	Water sorption	Pearson Correlation	Sig. (2- tailed)*
		Mean $\pm$ SD	Mean $\pm$ SD		
Control	12	64.404 $\pm$ 17.622	2.095 $\pm$ 0.435	.299	.261
Group I	12	50.733 $\pm$ 7.581	1.323 $\pm$ 0.482	-.089	.911
Group II	12	41.084 $\pm$ 5.869	3.205 $\pm$ 0.727	-.891	.109
Group III	12	51.825 $\pm$ 11.451	1.323 $\pm$ 0.482	-.884	.116
Group IV	12	48.665 $\pm$ 8.003	2.069 $\pm$ 0.193	-.076	.924
Group V	12	49.468 $\pm$ 11.591	1.308 $\pm$ 0.210	.193	.491
Group VI	12	56.029 $\pm$ 13.434	1.903 $\pm$ 0.216	.587	.21
Group VII	12	57.828 $\pm$ 13.586	1.913 $\pm$ 0.533	.269	.332
Group VIII	12	56.069 $\pm$ 8.9146	0.204 $\pm$ 0.133	.149	.470
Group IX	12	80.063 $\pm$ 27.299	1.480 $\pm$ 0.712	.593	.071
Group X	12	55.078 $\pm$ 23.047	1.977 $\pm$ 1.323	.447	.195

P-value significant at  $p < 0.05$

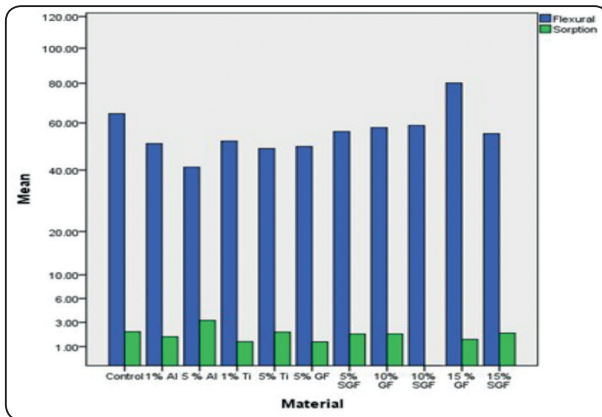


Fig. (1) Bar chart of the flexural strength and water sorption of all groups.

## DISCUSSION

Acrylic resin prosthesis has been used for years as a denture base material for the edentulous patients to improve their quality of life. It has many benefits as reasonable cost, aesthetic, and ease of handling. Because of its inferior mechanical and physical properties, it is more prone to fracture or deform. Several attempts have been made to enhance the acrylic resin properties by including reinforcing material such as fibers or nanoparticles<sup>(10)</sup>. The water sorption, storage time, and the type of denture base material affect the flexural strength of heat-cured acrylic resin, polyamide, and polycarbonate resins<sup>(11)</sup>. As the water molecules penetrate acrylic resin, they embed themselves among the polymer chains and push them apart and cause two effects: First, it allows the polymerized mass to expand slightly. Second, the water molecules interact with the polymer chains and therefore act as plasticizers<sup>(12)</sup>.

It was found that the increase of the water sorption decreases the flexural strength of heat-cured acrylic resin specimens reinforced with glass fibers sticks for 21 days of water storage due to the leaching of ions or oxides from the glass fiber in water. Indeed, some elements as boron were added to the E-glass fibers to improve its properties may adversely induce hydrolytic degradation and negatively influence the

polymer system<sup>(13)</sup>. Also, adhesion failure between the glass fiber and the polymer results in the fibers debonding from the polymer matrix and induces more water entrapment. The orientation of the fibers plays a role in the hydrolysis of the glass fiber<sup>(14)</sup>. Polymerization shrinkage destroys the acrylic resin layer surrounding the surface fibers and reduces the bond between them<sup>(15)</sup>. Microscopic analysis of the acrylic resin specimens reinforced with the dental and industrial glass fibers revealed voids between the glass fibers and the polymer that indicate partial bonding between them<sup>(16)</sup>. It was found that with the higher fiber content, the amount of water sorption decreased for both compression mold and injection molded acrylics reinforced with glass fibers after 4, 25, and 30 days interval of water storage. The amount of water sorption was higher in compression molded acrylic resins<sup>(17)</sup>. It was found that the wet acrylic resin specimens showed a reduction in the flexural strength by 27 % than the dry fiber-reinforced one. The most reduction occurred during the first month of water storage and stayed at that level for 180 weeks<sup>(18)</sup>. The addition of 2% volume glass fibers has decreased the flexural strength of both injection-molded and heat-cured acrylic resin after five weeks of immersion in water. This reduction may be explained by the lateral spreading of fibers when the specimens were pressed<sup>(19)</sup>. However, another study found a significant increase in acrylic resin's flexural strength reinforced with glass fiber sticks after 50 hours and 180 days of water immersion<sup>(20)</sup>.

The previous studies showed that in the specimens reinforced with metal oxide nanoparticles, the water sorption decreased due to some nanoparticles' existence in the free spaces between the acrylic resin polymer chains. The nanoparticle approximates the resin molecules and produces a more complicated network chain with less space available for water sorption. Consequently, water sorption resistance will be increased<sup>(21)</sup>. Nanoparticles provide an additional energy-absorbing mechanism that will prevent the crack re-sharpening and propagation of



the nanocomposites. Thus higher energy is needed to cause the fracture of specimens <sup>(22)</sup>. The polymer network center is neutral without a surface charge except at the nanoparticles' grafted site. Indeed, the hardcore is hydrophobic, and the polyelectrolytes are weakly hydrophilic. Upon ionization of the grafted polyelectrolytes in the water bath, the ionized beads' charges will create a repelling force that prevents the water from adsorption inside the polymer. There is an inverse relationship between the degree of ionization and the contact angle of the nanocomposites. The more degree of ionization, the less contact angle occurs <sup>(23)</sup>. This study's results agree with the results of another study where the addition of 0.5% and 1% titanium oxide nanoparticles has decreased the flexural strength of the heat-cured acrylic resin without a significant difference from the control group <sup>(24)</sup>. The possible reasons for that are the nanoparticles affects the degree of conversion, so that the residual monomer will be entrapped between the polymer network and act as a plasticizer. Lack of bond between the nanoparticles and the polymer is an additional problem <sup>(25)</sup>. Indeed, the nanoparticles tend to agglomerate inside the polymer and act as stress concentration centers and voids <sup>(26)</sup>. Scanning electron microscope showed nanoparticle agglomeration inside the polymers of the maxillofacial prosthesis material. It is evident when the nanoparticle concentration was increased to a 3.0 % density <sup>(27)</sup>. A meta-analysis showed that the addition of 0.1% and 0.3% of titanium nanoparticles to the heat-cured acrylic didn't improve its mechanical properties. Indeed, no precise ideal titanium dioxide nanoparticle concentration was determined to improve the polymer's flexural strength <sup>(28)</sup>. Another study evaluated the effect of the addition of 1%, 3%, and 5% aluminum oxide nanoparticles to the heat-cured, auto polymerized, and microwave acrylic resin. Only 1% showed a significant increase in flexural strength of both heat cure and chemical cured acrylic resin and 3% in heat-cured acrylic resin only. There was a statistically

significant decrease in the flexural strength of the microwave acrylic with the addition of 3% and 5% aluminum oxide nanoparticles concentration and 5% alumina in auto polymerized acrylic. The rest of the specimens showed a statistically non-statistical difference. Scanning electron microscope analysis showed nanoparticle aggregation and pull-out outside the polymer matrix <sup>(29)</sup>. Another study showed a significant decrease in the flexural strength of heat-cured acrylic with the addition of 1%, 2%, 5% nano, and micro-sized alumina nanoparticles. The nanoparticles group showed fewer flexural strength values and more particle agglomeration and water sorption <sup>(30)</sup>.

The current study showed that the water sorption has no effect on the flexural strength of heat cured acrylic resin with the addition of E-Glass fibers, titanium and aluminum nanoparticles. For the heat cured acrylic resin without glass fibers, water immersion didn't significantly decrease the flexural strength. A study found that heat-cured acrylic resin's flexural strength did not vary significantly after 50 hours, 30 days, 60 days, and 180 days of water storage in distilled water at 37°C. Although the transverse strength showed a slight decrease after 30 days of water storage, the flexural strength increased again after 60 and 180 days of water storage <sup>(31)</sup>. Another study showed a reduction in the transverse strength of high impact, rapid heat-cured, and visible light-cured acrylic during 30, 60, and 90 days of water storage, statistically non-significant. The decrease in the first 30 days was high for high impact strength acrylic, intermediate in the rapid heat-cured acrylic resin, and low for the visible light-cured acrylic. However, all the material's transverse strength began to show an increase from 60 to 90 days of water storage <sup>(32)</sup>.

The current study has some limitations. First: only two metal oxide concentrations were selected in this study, a wide range of concentrations and materials must be covered. Second: there are a few

studies regarding the toxicity, safety, whether the amount of leaching of nanoparticles from acrylic at the concentrations in this study is less than the toxic level or not. However, the amount of nanoparticles release from the denture base is less than the denture liner and the denture adhesive irrespective of the type of nanoparticle<sup>(33)</sup>. Third: the medium in this study didn't stimulate oral fluid conditions. Fourth: The other physical and mechanical properties should be studied for a broader scope of correlation. Fifth: a longer time is needed.

## CONCLUSION

Within the limitation of this study, the correlation between the water sorption and flexural strength was non-significant in acrylic resin reinforced with E-Glass fiber, aluminum oxide, and titanium nanoparticles. Water sorption has no effect on the flexural strength of heat cured acrylic resin.

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