

# Comparison of Some Mechanical Properties of Silanated SiO<sub>2</sub> and Polyester Fiber Composite Incorporation into Heat Cured Acrylic Resin.

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## ABSTRACT

**Background:** improving polymethylmethacrylate (PMMA) resin properties is the challenge nowadays, this can be done by adding several forms and types of nano- and micro-particles to the powder or to the monomer, the aims of the study is to investigate some mechanical properties of the acrylic resin after the addition of SiO<sub>2</sub> nano-particles in combination with polyester fibers.

**Materials and Methods:** The research includes 160 samples divided into four groups, four tests investigated in the study: Transverse flexural strength, impact strength, surface hardness and surface roughness (n=10). Group I is the control (No addition), group II with the addition of SiO<sub>2</sub> 5% by wt. nano-particles, group III in which polyester fibers 3% by wt., 6 mm length added and group IV contains a combination of SiO<sub>2</sub> 5% by wt. nano-particles and polyester fibers 3% by wt., 6 mm length. The data analyzed by ANOVA Table and multiple comparison post hoc Tukey's tests.

**Results:** Show that a mark increase in the impact and flexural strength when combination of 5% by wt. silanated SiO<sub>2</sub> and 3% by wt. and 6 mm length polyester fiber incorporated into PMMA resin; while flexural strength and surface hardness tests show that there was no significant differences among the groups after ANOVA Table inferential statistical analysis application. Surface roughness comparison among the groups revealed that group containing silanated SiO<sub>2</sub> only gives the highest rough surface while group containing polyester fiber only shows the lowest value of roughness.

**Conclusion:** Within the limitation of this study, incorporation of silanated SiO<sub>2</sub> and polyester fiber combinations in a significant weight and fiber length lead to enhancement in the impact and flexural strength of PMMA resins and with no or little effect on the surface hardness and roughness properties.

## KEYWORDS

Silanated SiO<sub>2</sub>, Polyester fibers, PMMA, Acrylic resins, Nanoparticles.

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## مقارنة لبعض الخواص الميكانيكية لخليط Silanated SiO<sub>2</sub> وألياف البوليستر المضافة إلى الراتنج الأكريلي الحراري

### المستخلص

تحسين خصائص PMMA (polymethylmethacrylate) الراتنج الأكريلي الحراري هو التحدي في الوقت الحاضر، وهذا يمكن أن يتم عن طريق إضافة عدة أشكال وأنواع نانوية وجسيمات دقيقة إلى مسحوق الراتنج الأكريلي، و الهدف من الدراسة هو تحسين بعض الخواص الميكانيكية من راتنج الأكريلي بعد إضافة جزيئات SiO<sub>2</sub> النانوية المخلوطة مع ألياف البوليستر ووجد ان ضمن محدودية هذه الدراسة اضافة البوليستر المخلوط مع حبيبات SiO<sub>2</sub> النانوية يحسن من الخواص الميكانيكية ولا يؤثر على خشونة الاسطح.

## INTRODUCTION

Acrylic resin has been advocated for use as denture base materials for many years, new techniques and additions were employed to enhance the physical and mechanical properties of this material. PMMA resins are still more commonly used for the fabrication of removable prosthesis, due to their properties of good aesthetics, applicable processing, and ease of repair when fractured (1,2).

Various attempts have been made to resolve the problems of denture fracture and to advance the mechanical properties of dental polymers. Reinforcing of the prosthesis with metal wires is one of the general strengthening means; on the other hand adhesion failure between PMMA and metal wire makes this procedure weak (3). Two method of improving the impact strength of rigid PMMA by adding wire or

cast metal plate (4) or by adding polypropylene fiber (5,6). A further technique is by reinforcing dentures with fibers, for examples glass fibers, polyester and carbon (7).

Alamel 2014 added SiO<sub>2</sub> nano fillers to the acrylic resin and no significant increased in surface roughness was found when the percentages of SiO<sub>2</sub> 3% and 5% by wt. (8).

Research has been devoted to the development of a new industrial process that produce a composite materials from nanoparticles, fibers and PMMA to synthesized a new form of PMMA that offers the strength of the Nano-oxides, and flexure of fibers in addition to polymer flexibility (9).

Little studies had been reported on how the addition of these composite materials (Nanoparticles and fibers) to PMMA could affect its properties,

this study was conducted to use silanated SiO<sub>2</sub> nanoparticles in a combination with polyester fiber added to heat cured PMMA and study the effects of this addition on some mechanical properties over pure heat cured PMMA and silanated SiO<sub>2</sub> only added to PMMA.

## MATERIALS AND METHODS

### Specimens' grouping:

160 samples included in the study, four mechanical tests applied for each group (Impact strength, transverse flexural strength, surface hardness and surface roughness tests), 10 samples were selected for each test. The samples grouped as follow:

- Group I: Control (PMMA) only
- Group II: 5% silanated SiO<sub>2</sub> nanoparticles incorporated into PMMA <sup>(8)</sup>.
- Group III: 3% (6 mm length) Polyester fiber incorporated into PMMA <sup>(10)</sup>.
- Group IV: Composite of 5% silanated SiO<sub>2</sub> and 3% (6 mm length) Polyester Fiber.

### Test specimen's preparation:

Two different metal patterns used to prepare the test specimens. The first pattern with the dimension of 65 mm\*10 mm\*2.5 mm (length, width and height respectively) was used to prepare the samples to

**Table 1: Shows the ratio of materials mixing for each group included in the study.**

Group	Amount of SiO <sub>2</sub> (g)	Amount of Polyester fiber (g)	Amount of polymer (g)	Amount of monomer (ml)
Group I (Control)	0g	0g	44g	20 ml
Group II	2.2g (5%)	0g	41.8g	20 ml
Group III	0	1.32g (3%)	42.68g	20 ml
Group IV	2.2g (5%)	1.32g (3%)	40.48	20 ml

In group II a silanated SiO<sub>2</sub> mixed with monomer by probe sonication device for 2 min. and immediately mixed with PMMA to prevent aggregation of nanoparticles while in group III the polyester fiber first cut into small thread of 6 mm length by using ruler and scalpel, then immersed in monomer for 10 min. in a container and allowed to dry and mixed with polymer <sup>(12)</sup>.

For group IV, the Silanated SiO<sub>2</sub> mixed with monomer by sonicating probe for 2 min. and the polyester fiber mixed with PMMA powder then added together.

Then wait the acrylic to reach dough stage and loaded into the mold which coated with separating media to prevent sticking of the sample to the dental stone, then a pressure of 100 bar applied and the access materials was removed, the flask and its holder

be tested by transverse flexural strength, surface hardness and surface roughness test. The other form of pattern with dimension of 80 mm\*10 mm\*4 mm (length, width and height respectively) was used to prepare samples for impact strength test (ANSI/ADA specification No. 12, 1999) <sup>(11)</sup>.

The mold prepared by pouring a dental stone type III (Zhermach, Italy) in the flask lower half after placing the flask over the vibrator then the metal pattern immersed into the stone to about half of its thickness for easy of removal after complete set of the dental stone, a layer of petroleum jelly painted over the set stone and metal pattern then the upper member of the flask placed over the lower half with the flask placed over the vibrator and another layer of dental stone poured and leaved to set then opened and the metal pattern removed.

the acrylic resin used in the study (Vertex, Netherland) mixed according to the manufactural instruction (2.2 g Powder / 1 ml liquid) and the mixing ratio of each group calculated for polymer , monomer , SiO<sub>2</sub> and Polyester fiber by using a sensitive electronic balance with accuracy of 0.0001g and illustrated in the Table 1.

placed in water path, the temperature increase from 20-100°C for 3 hr. and then for 30 min. in 100 °C according to manufactural instructions <sup>(11)</sup>.

### Testing the specimens:

#### A. Impact strength test:

After immersing the samples in a distilled water for 48 hrs. at 37 °C <sup>(11)</sup>, the samples tested by using charpy type impact testing instruments with a 2 joules testing capacity and the impact energy absorbed read on a scale which represent the energy required to fracture the specimen. The impact strength can calculated by applying the following formula:

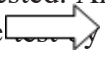
$$\text{Impact strength} = \frac{E}{B \cdot D} \times 10^3 \Rightarrow \text{KJ} / \text{m}^2 \quad (13)$$

E: is the impact absorbed energy in joules.

B: is the width in millimeters of the specimens.

D: is the thickness in millimeters of the specimens.

## B. Flexural transverse strength test:

40 samples included in the test 10 samples for each group, the samples stored in a distilled water for 48 hrs. at 37 °C <sup>(11)</sup> before tested. An instron testing machine used to perform the  applying a load of 50 kg with cross head speed 1 mm /min., once the sample fractured the maximum reading in Newton (N) obtained and by applying the following formula the bending strength of the material calculated:

$$\text{Transverse strength} = \frac{3Pl}{2bd^2} \quad \text{N/mm}^2 \quad (13)$$

P: is the peak load (N)

l: is the span length(mm)

b: is the sample width (mm)

d: is the sample thickness (mm)

## C. Surface hardness test:

After storing the specimen in distal water for 48 hrs at 37°C <sup>(11)</sup>, all samples included in this test divided into five equal parts by drawing lines using ruler to obtain five reading for each sample by using shore D test for hardness, which is special for plastic

materials hardness testing, the average of the five reading calculated. The device consists of indenter read from 0-100 units, connected to a digital scale meter, the device set to give the maximum reading after pressing down quickly and firmly.

## D. Surface roughness test:

Ten specimens for each group included in the test and the test performed after storing the samples for 48 hrs. at 37 °C <sup>(11)</sup>, in distilled water, the sample tested by using profilometer which can measure up to 1 mm surface variation and three points selected for each sample then the average of the three reading calculated for each sample in micrometer (µm).

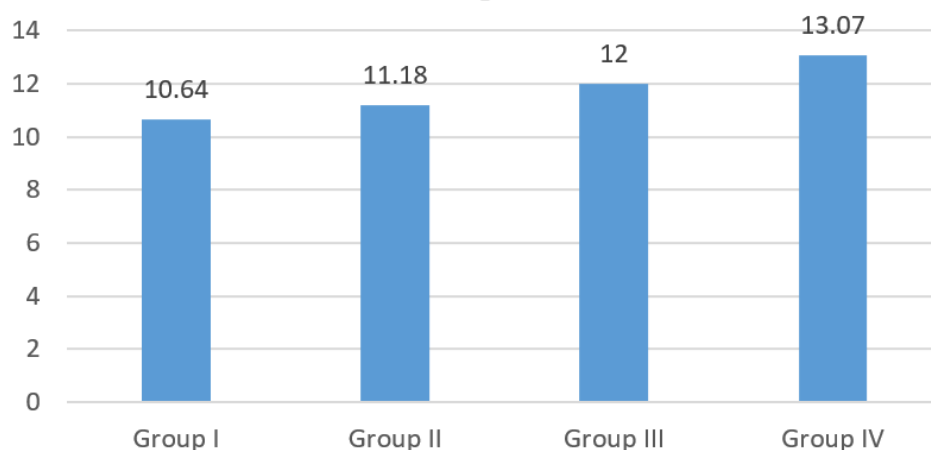
The data statistically analyzed by using descriptive and inferential statistical analysis using ANOVA Table and post hoc Tukey test P<0.05.

## RESULTS

Descriptive statistical analysis including means and standard deviations for all groups have been represented in Table 2, in addition bar chart representing the means for all groups included in each test (Fig. 1,2,3 and 4).

**Table 2: Represent means and Standard Deviation for all groups and tests included in the study.**

	N	Impact strength (KJ/m <sup>2</sup> )		Flexural Transverse strength (N/mm <sup>2</sup> )		Surface Hardness		Surface Roughness (µm)	
		Mean	SD	Mean	SD	Mean	SD	Mean	SD
Group I	10	10.64	0.58157	117.78	8.457	84.57	2.9181	0.6555	0.04211
Group II	10	11.18	0.3743	119.21	2.552	85.93	2.3485	0.6713	0.02231
Group III	10	12	1.78730	120.052	4.05785	84.88	1.3315	0.5694	0.02465
Group IV	10	13.07	0.67338	122.148	9.09777	86.60	1.5656	0.6462	0.02877



**Fig. 1: Bar chart shows all the groups' means for Impact strength Test (KJ/m<sup>2</sup>)**

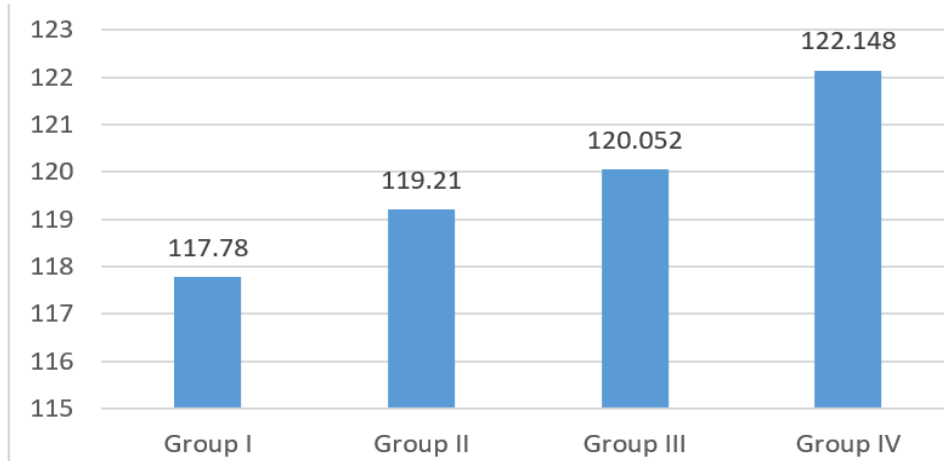


Fig. 2: Bar chart shows all the groups' means for Flexural strength Test (N/mm<sup>2</sup>).

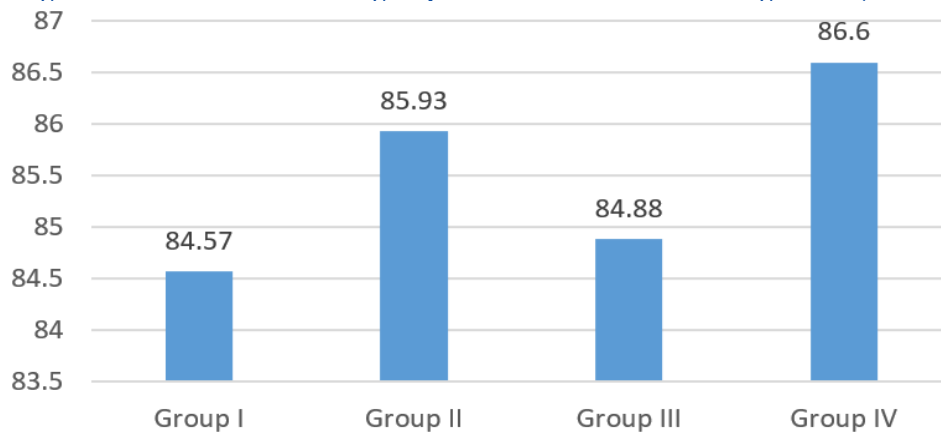


Fig. 3: Bar chart shows all the groups' means for surface hardness Test.

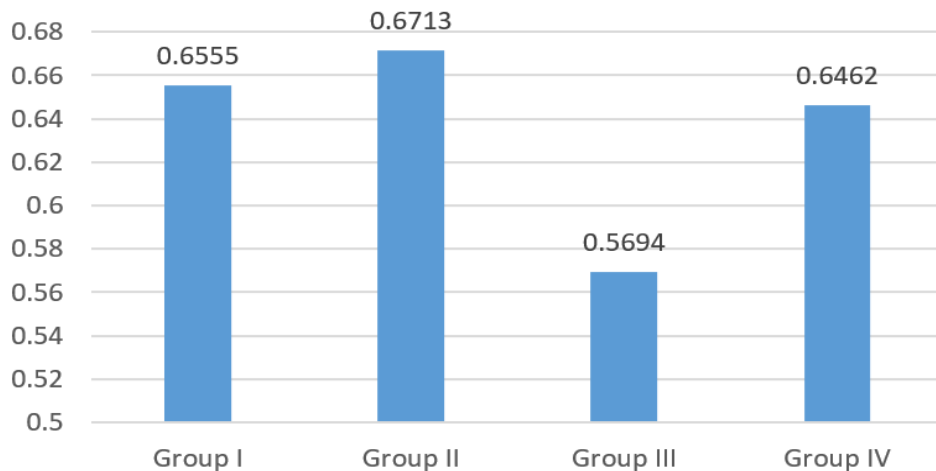


Fig. 4: Bar chart shows all the groups' means for surface roughness Test (μm).

Inferential statistics include ANOVA Table with multiple comparison Post hoc Tukey's Test used to compare the groups in each test.

The results for impact strength tests show that highly significant differences between all groups ( $P < 0.05$ ) except for group II when compared with group I and group III shows no significance differences ( $P$  Value = 0.242, 0.079 Respectively) and group III when compared with group IV also shows no significance differences ( $P$  value = 0.101) as shown in Table 3 and 4.

While comparing groups in flexural strength test show no significant differences among all groups as

shown in Table 3 and 4.

Groups' comparison in surface hardness test shows no significant differences among all groups when using ANOVA Table Test ( $P$  value = 0.142) as shown in Table 3.

Roughness test groups' comparison show that highly significant differences between Group I and III, Group II and III and Group III and IV ( $p < 0.05$ ), and no significant differences between Group I and II, Group I and IV and Group II and IV ( $P$  value = 0.655, 0.903 and 0.270 respectively) as shown in Table 3 and 4.

**Table 3: ANOVA Table for All Tests included in the study.**

		<i>Sum of Squares</i>	<i>df</i>	<i>Mean Square</i>	<i>F</i>	<i>Sig.</i>
<b>Impact Strength Test</b>	<b>Between Groups</b>	33.589	3	11.196	10.854	H.S.
	<b>Within Groups</b>	37.136	36	1.032		
	<b>Total</b>	70.725	39			
<b>Flexural Strength Test</b>	<b>Between Groups</b>	100.051	3	33.350	0.753	0.528
	<b>Within Groups</b>	1595.457	36	44.318		
	<b>Total</b>	1675.507	39			
<b>Surface Hardness Test</b>	<b>Between Groups</b>	26.441	3	8.814	1.931	0.142
	<b>Within Groups</b>	164.298	36	4.564		
	<b>Total</b>	190.739	39			
<b>Surface Roughness test</b>	<b>Between Groups</b>	0.062	3	0.021	22.173	H.S.
	<b>Within Groups</b>	0.033	36	0.001		
	<b>Total</b>	0.095	39			

**Table 4: Multiple comparison Post hoc Tukey's test for All Tests included in the study.**

	<i>Impact Strength</i>		<i>Surface roughness</i>	
	<i>Mean Difference (I-J)</i>	<i>Sig.</i>	<i>Mean Difference (I-J)</i>	<i>Sig.</i>
<b>Group I-Group II</b>	-0.54	0.242	-0.0158	0.655
<b>Group I-Group III</b>	-1.36*	S.	0.0861*	H.S.
<b>Group I-Group IV</b>	-2.43*	H.S.	0.0093	0.903
<b>Group II-Group III</b>	-0.82*	0.079	0.1019*	H.S.
<b>Group II-Group IV</b>	-1.99*	H.S.	0.0251	0.270
<b>Group III-Group IV</b>	-1.07	0.101	-0.0768*	H.S.

\* The mean difference is significant at the 0.05 level.

## DISCUSSIONS

The most important properties, which is the matter of concern for all researcher when they try to improve the acrylic resin properties are the impact strength and flexural strength properties in addition to the other mechanical characteristics tested such as surface hardness and surface roughness.

In order to improve the properties, several additives were added to the acrylic resin and the material mechanical properties examined. In this study, silanated SiO<sub>2</sub> and polyester fiber used to improve the PMMA resin properties. Some of these materials increase one or more of the properties mentioned above and other decrease them.

In this study, when adding silanated SiO<sub>2</sub> with the 5% by wt. concentration it tends to increase the impact strength as compared to control group, and decrease the impact strength as compared to group III (adding 3% by wt. and 6 mm length polyester fiber) and group IV (adding 5% by wt. SiO<sub>2</sub> and 3% by wt. and 6 mm length polyester fiber), while group III and

IV shows an improvements in the impact strength when compared with control group as shown in Table 2 and 4, this results may be due to the fact that when applying stress to measure the impact energy it will be affected by spaces results between the matrix and filler like particles' size and shape in addition to the interstitial spaces between the molecules <sup>(14)</sup>.

The results also in agreement with Chen et al <sup>(15)</sup> who found that when adding 3% by wt. 6 mm length polyester fiber, it would increase the impact strength and he concluded that the polyester fiber was the ideal fiber for acrylic reinforcement from the point of manipulation and esthetics.

The use of silanated SiO<sub>2</sub> nano-fillers will cause increase in the impact strength properties to a significant value while combining silanated SiO<sub>2</sub> with polyester fiber will improve the strength to much more value, this is in agreement with Fulga et al <sup>(16)</sup> who found that the impact strength can be increase because of increase the surface areas as the particles size decreased, when a composite materials

used (silanated SiO<sub>2</sub> and polyester fibers) another idea become clear which is the mesh action of the polyester fiber when mixed with Silica oxide which can improve the impact energy of the composite material and in turn prevent crack propagation between the molecules<sup>(17)</sup>.

This results also agreed with Alnamel and mudhafar<sup>(5)</sup> who found that the addition of silanated SiO<sub>2</sub> 5% by wt. with epoxy coupling agents will increase the impact strength due to increase the interfacial surface area available for energy dissipation, in spite of the differences in the saline coupling agents used in the silanation of the the SiO<sub>2</sub> as the SiO<sub>2</sub> used in this study already silanated by the US-Nano company (Gamma-Methacryloxypropyltrimethoxy silane coupling agent which is a methacryl-functional silane) also differences in particle size affected.

The flexural strength test, which is also called bending strength and rupture strength, shows that the lowest value of flexure obtained in group I (control) (117.78 N/m<sup>2</sup>) when only PMMA resin used without addition while in group IV when silanated SiO<sub>2</sub> used in combination with polyester fiber shows the highest value of flexural strength (122.1480 N/m<sup>2</sup>) as shown in Table 2, this may be due to the number of the microfilament in the polyester fiber and the size of each filament that act as a network to mesh the nanoparticles of SiO<sub>2</sub> and increase the flexural strength of the composite resin matrix and also may be due to the mixture homogeneity and even distribution of fibers<sup>(18-22)</sup>.

By using ANOVA Table shows no significant differences among all groups for surface hardness test however, the highest value found in group IV (86.60) and the lowest value in group I (control group = 84.57) which show a very little difference with group III (84.88) when only polyester fiber incorporated into PMMA resin, therefore the unchanged in the surface hardness of the material may be due to the accumulation of the enforcement materials within the bulk of the specimen rather than on the surface, the results in disagreement with Hachim et al<sup>(11)</sup> who stated that acrylic resin hardness decreased when using polyester fiber of both 2 mm and 4 mm length this may be due to differences in fiber length and concentration used (3% by wt and 6 mm length in the present study) that leads to more fiber enforcements and more randomized distribution of them within the bulk of the specimen.

The surface roughness test can be defined as a quantifying measurements by local deviation of the surface from perfectly flat ideal plane and according

to this measurements if it's small, the roughness is low while if it's large, the surface roughness is high<sup>(23)</sup>. In our study, group III when 3% by wt. and 6 mm length polyester fiber incorporated into the PMMA resin it shows the lowest value (0.5694 μm) and the smoothest surface than the other groups because the nature of the fibers and the number of the filaments in each fiber give this smoothness and improve the material properties, while the highest value shown in group II when 5% by wt. SiO<sub>2</sub> only incorporated into the PMMA resin, this may be due to the fact that the particles of SiO<sub>2</sub> differ in roughness than that of acrylic denture base resin and the distribution of this particles within the matrix of the specimen leads to increase the value of surface roughness and more coarse surface resulted but it still with no significant differences when compared with control group and this came to be in agreement with the results of Abdul Ameer 2006<sup>(24)</sup>, Safi 2011<sup>(25)</sup>, Alnamel 2014<sup>(5)</sup> and Esmael 2015<sup>(26)</sup>, in which they were incorporated nano particles in different concentration into the PMMA resin.

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