

# Evaluation the Effect of Addition of Plasma Treated Polypropylene Fiber and Silanized Silicon Dioxide Nanoparticles Composite on Some Properties of Heat-Polymerized Polymethylmethacrylate

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## ABSTRACT

**Background:** Polymethylmethacrylate (PMMA) is the most commonly used material in denture construction. This material is far from ideal in fulfilling the mechanical requirements, like low impact and transverse strength and poor thermal conductivity are present in this material.

The purpose of this study was to study the effect of addition a composite which include 1%wt silanized silicone dioxide nano fillers (SiO<sub>2</sub>) and 1wt% oxygen plasma treated polypropylene fiber (PP) on some properties of heat cured acrylic resin denture base material (PMMA).

**Materials and methods:** One hundred (100) prepared specimens were divided into five groups according to the tests, each group consisted of 20 specimens and these were subdivided into two groups (unreinforced heat cured acrylic resin as control group) and reinforced acrylic resin with ( 1%wt Nano SiO<sub>2</sub> and 1% wt oxygen plasma treated polypropylene fibers) group. The transverse strength, impact strength, indentation hardness (shoreD), surface roughness and water sorption and solubility were investigated. The results were statistically analyzed using descriptive and t-test.

**Results:** The results of this study show that a highly significant increase in impact strength (10.4939 KJ/m<sup>2</sup>), surface hardness (89.9375) surface roughness (0.9498) and water sorption (0.0171mg/cm<sup>2</sup>) was observed with the addition of 1%wt silanized (SiO<sub>2</sub>) nanoparticles and 1%wt oxygen plasma treated polypropylene fibers to (PMMA) , also significant decrease in transverse strength (103.4753 N/mm<sup>2</sup>), nonsignificant decrease occurred in water solubility which was (0.0005mg/cm<sup>2</sup>).

**Conclusion:** The incorporation of 1%wt silanized SiO<sub>2</sub> nanoparticles and 1%wt oxygen plasma treated polypropylene fiber to heat cure PMMA form a composite improves the impact strength, surface hardness and surface roughness of acrylic resin, at the same time this addition increase the water sorption and decrease water solubility; while significant decrease in transverse strength.

**Key words:** silicon dioxide nano filler, polypropylene fiber, plasma treatment. (J Bagh Coll Dentistry 2016; 28(4):1-8)

## INTRODUCTION

The goal of dentistry has been to replace or restore the lost or damaged tooth structure for satisfying esthetic and functional requirements. Dentures remain the most common choice of prosthetic devices. Dentures made from resin based polymeric systems were common because of their ability to be molded with outstanding esthetic appearance and appropriate mechanical characteristics in most clinical conditions <sup>(1)</sup>.

This material is not ideal in every respect and it is the combination of virtues rather than single desirable property that accounts for its popularity and usage. Regardless of the satisfying esthetic demands it is far from ideal in fulfilling the mechanical requirements of prosthesis. However, a polymer requires some modifications in its structure or physical properties to get a greater range of functions.

One modification technique is adding fillers to the polymer in order to produce a composite with enhanced properties, such as improvement in mechanical strength, electrical conductivity or thermal stability <sup>(2)</sup>.

The filler materials include organic, inorganic, and metallic particulate materials in both micro and nanosizes. Several types of polymers and polymer-matrix composites reinforced with metal particles have a wide range of manufacturing applications <sup>(3)</sup>.

Organic-inorganic hybrid nanocomposite materials have been studied in recent years, with the expectation that nanocomposite material will serve a significant and evolutionary means of achieving properties that cannot be realized with single material <sup>(4)</sup>. Nanocomposites have the potential to be implemented as a new high strength matrix in a composite <sup>(5)</sup>. These composites are desired due to their low density, high corrosion resistance, simplicity of fabrication, and low cost <sup>(6)</sup>.

The addition of inorganic fillers like alumina into polymers is primarily aimed at the cost reduction and stiffness improvement <sup>(7)</sup>. Fiber reinforced polymer composites (FRPCs) have

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generated wide interest in various engineering field because of high specific strength, high modulus, low density and better wear resistance<sup>(8)</sup>. The concept of combining nanocomposites as matrix material with fiber reinforcement in a new three-phase composite reinforcement has been shown to be very successful. Lighter, thinner, stronger, and cheaper structures are the goals of materials science and engineering applications nowadays<sup>(9)</sup>.

## MATERIALS AND METHODS

Before starting the tests PP fiber and nano silica should be under go surface treatment separately to increase the adhesion with PMMA matrix. The silica would be silanised using a coupling agent to guarantee the interfacial adhesion between the filler and matrix<sup>(10-11)</sup>, while PP fiber undergo surface treatment by oxygen plasma<sup>(12)</sup>

One hundred acrylic specimens were constructed by conventional flasking technique using heat cure acrylic resin, the samples were divided into five groups according to the using tests and each group sub divided into two subgroups: unreinforced heat cured acrylic resin as control group and reinforced acrylic resin with (1%wt Nano SiO<sub>2</sub> and 1% wt oxygen plasma treated polypropylene fibers) group

### Mechanical and physical tests

#### A. Impact strength test

The specimens were prepared with dimensions (80mm x 10mm x 4mm) (ISO 179, 2000) for unnotched specimens. Specimens were stored in distilled water at 37 °C for 48 hours before being tested<sup>(13)</sup>. The impact strength test was evaluated following the procedure recommended by the ISO 179 with impact testing device. The specimens were supported horizontally at each end and struck by freeswinging pendulum of 2 Joules. The scale readings give the impact energy in Joules. The charpy impact strength of unnotched specimens was calculated in Kilo joules per square meter by the following equation:

$$\text{Impact strength} = X103 \text{ (ISO, 2000) (kJ/m}^2\text{)}$$



E : The impact energy in Joules,

b: the width of the specimens in millimeters,

d :the depth of the specimens in millimeters

#### B. Transverse strength test

Specimens were prepared with dimensions (65mm x 10mm x 2.5 + 0.1mm). All specimens stored in distilled water at 37 °C for 48 hours before being tested<sup>(13)</sup>. The test was performed using Instron universal testing machine (WDW-200 E), each specimen was positioned on the

bending fixture which consist of two parallel supports (50 mm apart), the fullscale was 50 Kg and the load was applied with across head speed of 1mm/min. by a rod placed centrally between the supports making deflection until fracture occurs.

#### C. Surface hardness test

Specimens of heat cure acrylic resin were prepared with a dimension (65mm x 10mm x 2.5+ 0.1mm). All specimens were stored in distilled water at 37°C for 48 hours before being tested<sup>(13)</sup>. Surface hardness was determined by using (Shore D) durometer hardness tester which is appropriate for acrylic resin material.

The apparatus consist of spring-loaded indenter (0.8mm in diameter), the indenter is attached to digital scale that is graduated from 0 to 100 units. The standard method is to press down firmly and quickly on the indenter and record the reading. Three readings were done on each specimen (one in the center and other at each end) then the mean of three readings was calculated.

#### D. Surface roughness test

Specimens with dimensions (65mm x 10mm x 2.5 + 0.1mm) were prepared to be used for surface roughness test. All the specimens were stored in distilled water at 37 °C for 48 hours before being tested<sup>(13)</sup>. The profilometer device was used to study the effect of fiber and nano particles reinforcement on the microgeometry of the test surface.

This device is supplied with sharp stylus surface analyzer from a diamond to trace the profile of the surface irregularities by recording of all the peaks and recesses which characterized the surface by its scale. The acrylic specimen was placed on its stable stage and the location of the tested area was selected (The specimen was divided into four parts) then the analyzer was traversed along the tested area and the mean of four readings was calculated by this equation:

$$\text{Transverse strength(S)} = 3 PL / 2bd^2$$

Where, P = Fracture load

L = Span length

b = Sample width

d = sample thickness.

#### E- Water sorption and solubility test

Acrylic disc specimens were prepared by using plastic pattern having dimensions of (50mm+1mm in diameter and 0.5 mm ±0.1 mm in thickness). The specimens were dried in desiccators containing freshly dried silica gel .The desiccator was stored in an incubator at a 37 °C ±2 °C for 24 hours after that the specimens were removed to room temperature for one hour then

weighted with electronic balance with accuracy of (0.0001g). This cycle of weighting was repeated every day until a constant mass (M1) (conditioned mass) was reached <sup>(14)</sup>. All discs of all groups were immersed in distilled water for 7 days at 37 °C ± 2 °C <sup>(14)</sup>. The discs were removed from the water with a dental tweezers wiped with a clean dry towel until free from visible moisture, waved in the air for 15 seconds and weighted; this mass was

recorded as (M2). The value of water sorption was calculated  $WSP = \frac{M2 - M1}{S}$  from the following equation:

WSP = Water sorption in mg/cm<sup>2</sup>

M2 = The mass of the disc after immersion in distilled water (mg)

M1=The mass of the disc before immersion in distilled water (conditioned mass) (mg).

S = Surface area of the disc (cm<sup>2</sup>)

In order to obtain the value of water solubility the discs were again reconditioned to a constant mass in the desiccator at 37 °C ± 2 °C as done in the first time for sorption test and the reconditioned mass was recorded as (M3). The solubility during immersion was determined for each disc by the following equation:

$$WSL = \frac{M1 - M3}{S}$$

WSL= Water solubility in mg/ cm<sup>2</sup>.

M1= the conditioned mass (mg).

M3= the reconditioned mass (mg).

S= the surface area of the disc (cm<sup>2</sup>).

## RESULTS

The results obtained from the measured data were computerized using SPSS system version 20for statistical analysis and classified according to the followings experimental groups:

- Group (A) Control group
- Group (B) Acrylic resin+SiO<sub>2</sub> 1% wt + PP 1 % wt

### A-Impact strength test

Mean value, standard deviation, maximum and minimum of the impact test results are presented in table (1). Group B (study group) exhibited the highest impact strength mean value (10.4939

Kj/m<sup>2</sup>), while the A (control group) shows the lowest one (8.3313Kj/m<sup>2</sup>).

### B-Transvers strength test

Mean value, standard deviation, maximum and minimum of the transverse test results are presented in table (3). As shown in table (3) group A (control group) exhibited the highest transverse strength mean value (112.6651N/mm<sup>2</sup>), while the B (study group) shows the lowest one (103.4753N/mm<sup>2</sup>)

### C-Hardness number test

Mean value, standard deviation, maximum and minimum of the surface hardness test results are presented in table (5). Group B (study group) exhibited the highest hardness mean value (89.9375), while the A (control group) shows the lowest one (85.0200).

### D-Surface Roughness test

Mean value, standard deviation, maximum and minimum of the surface roughness test of hot cure acrylic resin results are presented in table (7).Group B (study group) exhibited the lowest roughness mean value (0.9498 μm), while the A (control group) shows the highest one (1.4402 μm).

### E-Water sorption

Mean value, standard deviation, maximum and minimum of the water sorption test of hot cure acrylic resin results are presented in table (9).Group B (study group) exhibited the highest water sorption mean value (0.0171 mg/cm<sup>2</sup>), while the A (control group) shows the lowest one (0.0138 mg/cm<sup>2</sup>).

### F-Water solubility

Mean value, standard deviation, maximum and minimum of the water sorption test of hot cure acrylic resin results are presented in table (11).Group B (study group) exhibited the lowest water solubility mean value (0.0005 mg/cm<sup>2</sup>), while the A (control group) shows the highest one (0.0006 mg/cm<sup>2</sup>).

**Table 1: Descriptive parameters of impact test Kj/m<sup>2</sup>**

Group	N	Minimum	Maximum	Mean	S.D
Group A (Control)	10	7.23	9.13	8.3313	.63685
Group B (Study)	10	9.03	13.58	10.4939	1.53140

**Table 2: t-test analysis of impact test**

Groups	Standard error	Mean differences	t-test	p-value	Significance
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Group A (Control)	.52448	-2.16258	-4.123	.001	HS
Group B (Study)					

**Table 3: Descriptive parameters of transverse test N/mm<sup>2</sup>**

Groups	N	Minimum	Maximum	Mean	S.D.
Group A (Control)	10	102.90	123.40	112.6651	7.02951
Group B (Study)	10	89.59	126.28	103.4753	9.92008

**Table 4: t-test analysis of transverse strength test**

Groups	Standard error	Mean differences	t-test	p-value	significance
Group A (Control)	3.84476	9.18982	2.390	.028	S
Group B (Study)					

**Table 5: Descriptive parameters of hardness number test**

Groups	N	Minimum	Maximum	Mean	S.D.
Group A (control)	10	84.30	85.95	85.0200	.48959
Group B (study)	10	88.88	90.88	89.9375	.70072

**Table 6: t-test analysis of hardness number test**

Groups	Standard error	Mean differences	t-test	p-value	significance
Group A (Control)	.27031	-4.91750	-18.192	.000	HS
Group B (Study)					

**Table 7: Descriptive parameters of surface roughness test um**

Groups	N	Minimum	Maximum	Mean	S.D.
Group A (control)	10	1.32	1.59	1.4402	.08386
Group B ( study)	10	.90	1.02	.9498	.04335

**Table 8: t-test analysis of surface hardness test**

Groups	Standard error	Mean differences	t-test	p-value	significance
Group A (control)	.02985	.49034	16.425	.000	HS
Group B (study)					

**Table 9: Descriptive parameters of water sorption test mg/cm<sup>2</sup>**

Groups	N	Minimum	Maximum	Mean	S.D.
Group A (control)	10	.01	.02	.0138	.00094
Group B (study)	10	.02	.02	.0171	.00153

**Table 10: t-test analysis of water sorption test**

Groups	Standard error	Mean differences	t-test	p-value	significance
Group A (control)	.00057	-.00337	-5.936	.000	HS
Group B (study)					

**Table 11: Descriptive parameters of water solubility test mg/cm<sup>2</sup>**

Groups	N	Minimum	Maximum	Mean	S.D.
Group A (control)	10	.00	.00	.0006	.00061
Group B (study)	10	.00	.00	.0005	.00062

**Table 12: t-test analysis of water solubility test**

Groups	Standard error	Mean differences	t-test	p-value	significance
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Group A (control)	.00028	.00006	.230	.821	NS
Group B (study)					

## DISCUSSION

Physical and mechanical properties of polymer are critical in attaining clinical success and longevity of the material. The denture base material must be able to tolerate high impact force as well to normal masticatory forces<sup>(15)</sup>. One of the approaches to resolve the fracture problem in acrylic denture base was the incorporating of some types of reinforcement material into the polymer. The idea of new three-phase composite reinforcement from combining a nano particles and fibers as a filler with the material to be reinforced as a matrix has been shown to be very successful<sup>(9)</sup>.

In this in vitro study with its limitation in performing this concept, some mechanical and physical properties of acrylic denture base material were evaluated after the addition of silanized SiO<sub>2</sub> nanoparticles and oxygen plasma treated polypropylene fiber in order to improve the mechanical and physical properties with minimum effect on other properties.

### A-Impact strength test

The results of impact strength test as shown in table (1) has shown that the addition of 1%wt silanized SiO<sub>2</sub> nanoparticle and 1%wt oxygen plasma treated polypropylene fibers increased the mean values of impact strength (10.4939 KJ/m<sup>2</sup>) compared with the control group (8.3313 KJ/m<sup>2</sup>).

The highly significant increased value in impact strength may be related to the effect of interaction between silanized SiO<sub>2</sub> nanoparticles and polymer depending on the size of the contacting surfaces (specific surface area of the filler), Smaller particles have larger surface area; lead to increase strength. This agreed with Tanasă et al<sup>(16)</sup>. Also this increase value in the impact strength may be due to the interfacial shear strength between matrix and nano filler which is high due to the formation of supra molecular bonding or cross-links which shields or cover the nanofillers which in turn inhibit cracks propagation. This also suggested by Sun et al<sup>(17)</sup>.

The addition of silanized SiO<sub>2</sub> Nano filler may form efficient network (3D dimensional network) of PMMA and SiO<sub>2</sub> nano particles. PMMA chain totally transferred in to 3D-network like chains at 1% of Nano filler thus lead to decrease the segmental motion and increase the impact strength<sup>(18)</sup>.

In the other hand, the functional group in addition to rough surface due to oxygen plasma treatment of poly propylene fibers may be lead to

increase fiber-matrix bonding which may lead to prevent the crack propagation and change in direction of cracks resulting in minor cracks between the fibers, this can be correlated to the improved impact strength of fiber-reinforced specimens compared to the control group where there is unobstructed crack propagation. These findings are in agreement with results obtained by Mowade et al<sup>(2)</sup> and Mohammed<sup>(19)</sup>.

Polarization of the surface by the presence of functional groups due to oxygen plasma treatment of polypropylene fibers may increase the surface energy of the fibers and its compatibility with other materials which may improve the impact strength, in combination with high surface energy of nano particles may contribute in increase impact strength. These results are in agreement with results obtained by Hocker<sup>(12)</sup> and Mohammed<sup>(19)</sup>.

So the combination of both silanised SiO<sub>2</sub> nano particles and oxygen plasma treated polypropylene fibers incorporating in 2%wt lead to increase the impact strength of the composite.

### B-Transverse flexural strength test

According to the results of this study it has shown that there were statistically a significant decrease in transvers strength of PMMA reinforced with Nano SiO<sub>2</sub> 1%wt and 1%wt PP fiber as shown in Table (3).

The flextural failure of the denture base material is considered the primary mode of the clinical failure. The flexural three-point bending test is convenient in comparing the materials as it simulates the type of stress that applied to the denture during the mastication<sup>(20)</sup>.

Possible explanations for this may be due to:

1. The stress concentration at the sharp corners of irregular SiO<sub>2</sub> particles.
2. Too many filler particles can lead also to changes in mode of crack propagation through the specimen and the modulus of elasticity of the resin due to an increased amount of fillers.
3. Low percentage of nano particle surface treatment with silane coupling agent which may lead to insufficient nanoparticle-matrix bond.
4. Aggregation of silanized SiO<sub>2</sub> nanoparticles because of higher surface energy, this aggregation result in micro-fracture that weakened the nanocomposites at this percentage, so this study in agreement with Andrew et al<sup>(21)</sup> who suggested that "single individual Nano filler exhibited no distinct fracture. In contrast the

nanoclusters (aggregated) fillers showed multiple fractures"

Such defects can speed up the failure process and might be an area in which crack propagation is started. On the other hand, this may be related to the random orientation of fibers permit only small portion of the strengthening to be directed perpendicular to the applied stress. Also may be due to the internal voids formed in the fiber-resin composite caused by poor wetting of fibers with resin (maybe not all the using fibers will undergo changes from plasma treatment), these voids were oxygen reserves that permitted oxygen to inhibit radical polymerization of the acrylic resin inside composite. This could result in higher residual monomer content of fiber composite and affect strength. This result is agree with Vallittu et al (22).

The increase in internal repulsive or negative forces after addition of the silanized SiO<sub>2</sub> nano particle and oxygen plasma treated polypropylene fiber to the resin samples may contribute in reduction of transverse force

The plasticization effect of oxygen plasma treated polypropylene fiber may be result in decreased transverse strength, this agree with Al-Momen (23) who found after the addition of 5% and 10% Styrene Butadiene Rubber (SBR) into acrylic resin produce a significant decrease in transverse strength was observed due to increase in flexibility of composite containing SBR. This study disagree with Mohammed (19) who found after the addition of 2.5%wt oxygen plasma treated polypropylene fiber a non-significant increase in flexural strength test.

### **C-Surface hardness test**

It has been reported that surface hardness of composite resins influenced by both the organic matrix and fillers. With regard to the organic matrix, hardness depends on the density and structure of the polymer formed and degree of conversion after polymerization (24).

It was found in this study that hardness value showed a highly significant increase in the study group compared with control group as shown in table (5).

The increased hardness of the Nano composite at 1%wt may be attributed to the accumulation of the (SiO<sub>2</sub>) particles in to the acrylic matrix especially on the surface.

Similar finding were reported by Al Momen(23), while the result of this study disagree with Abdul Ameer (25) where titanium powder lower the indentation hardness of acrylic resin. This could be due to difference in the material and particles size between SiO<sub>2</sub> used in this study and titanium powder particles size.

Other factor of increase in hardness may be due to the use of coupling agent which increases adhesion between nanoparticles and the polymer and increases interphase stiffening that leads to increase nanocomposite's elastic modulus and strength, this suggestion agree with Zakaria and Nezhad (26).

This increase also could be related to the existence of polypropylene fibers near or at the surface of the composite which very stiff and hard, these findings were in agreement with Sato and Ogawa (27), Mohammed (19), Al- Momen (23) and Salih (28).

### **D-Surface roughness**

It appears from the literature that the roughness of dental acrylic resins is mainly affected by material inherent feature, polishing technique and operator's skills (29).

Table (7) and (8) showed that the surface roughness of the acrylic denture base was highly significant decrease in the studied group compared to the control group.

It is difficult to make direct comparisons of roughness values with other studies because of methodology used, disparities in the experimental procedures as well as measuring the surface roughness procedure and differences in the type of PMMA material used.

Surface modification of SiO<sub>2</sub> nano particles with silane coupling agent and treatment of polypropylene fibers with oxygen plasma may result in increase bonding among the component of the composite may lead to decrease chipping away of the particles from the surface of the composite during deflasking and grinding, this in turn may contribute in decreasing the roughness of the composite.

Also this may be attributed to the small particle size of SiO<sub>2</sub> (20-30nm) which is accumulated at the surface of study specimen.

### **E-Water sorption and solubility tests**

From the results, there were highly significant increases in water sorption in group B (study group) compared to the Group A (control group).

The surface treatment of SiO<sub>2</sub> nano particle with silane coupling agent may change the hydrophobicity of SiO<sub>2</sub> nano particles to hydrophilic leading to increased water adsorption to the surface of the specimen.

Porosity or micro void formed during polymerization process of acrylic resin may facilitate water transportation and act as a reservoir for it.

Non reactant interfacial surfaces between silanized SiO<sub>2</sub> nano particles and polymer matrix

as a result of aggregation of SiO<sub>2</sub> nano particles may provide a room for water molecules resulting in increasing water sorption.

Also increase could be attributed to fact that plasma treatment changed totally hydrophobic surface of untreated pp fibers to hydrophilic surface due to incorporating of functional groups, this explanation also agreed with Skacelova et al (30).

Table (11) and (12) show a non-significant decrease in the values of water solubility of the specimens containing SiO<sub>2</sub> and oxygen plasma treated PP fiber.

This decrease could be attributed to the fact that SiO<sub>2</sub> is insoluble in water so that the addition of SiO<sub>2</sub> to the mass of the specimens which act as additives and their presence will lead to decrease in the solubility of acrylic resin.

Also this may be attributed to the transverse interlocking occurred between the reinforced plasma treated polypropylene fibers and acrylic resin which lead to decrease the residual monomer content subsequent lesser solubility of the polymer will occur.

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