The effect of the addition of silanized Nano titania fillers on some physical and mechanical properties of heat cured acrylic denture base materials

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ABSTRUCT

Background: Polymethyl methacrylate (PMMA) is the most commonly used material in denture fabrication. The material is far from ideal in fulfilling the mechanical requirement. The purpose of this study was to evaluate the effect of addition of 3% wt of treated (silanized) Titanium oxide Nano filler on some physical and mechanical properties of heat cured acrylic denture base material.

Materials and methods: 100 specimens were constructed, 50 specimens were prepared from heat cure PMMA without additives (control) and 50 specimens were prepared from heat cure PMMA with the addition of TiO_2 Nano fillers. Each group was divided into 5 sub groups according to the test performed which was mixed by probe ultra-sonication machine.

Results: A highly significant increase in impact strength and transverse strength was observed with the addition of (TiO_2) Nano particles to (PMMA). A significant increase in surface hardness and in surface roughness. The water sorption and solubility were significantly decreased when compared with the control group.

Conclusions: The addition of TiO_2 Nano particles to heat cure acrylic resin improve the impact strength, transverse strength and surface hardness of heat cure acrylic resin at the same time this addition decrease water sorption and solubility. On the other hand there was an increase in surface roughness with the addition of 3% wt of silanized TiO_2 Nano particles.

Keywords: NanoTiO_2, TMSPM, PMMA. (J Bagh Coll Dentistry 2015; 27(1):86-91).

INTRODUCTION

PMMA is the most commonly used material due to its satisfactory mechanical and physical properties, compatibility with oral tissue, esthetics, ease of repair and low cost [(1)].The main problem associated with PMMA as a denture base material, is poor strength particularly under fatigue failure inside the mouth caused by occlusal biting force and impact failure outside the mouth by dropping the dentures [(1)].

To overcome their physical and mechanical limitations, polymers had been reinforced by adding different metallic oxide [(2)]. Recently, much attention have been directed toward the incorporation of inorganic Nano particles into PMMA to improve its properties [(3)].

The properties of polymer Nano composites depend on the type of incorporated Nano particles, their size and shape, as well as the concentration and interaction with the polymer matrix [(3)]. TiO_2 had been a low cost material with chemical stability and non toxicity [(4,5)].

This study was conducted to use inorganic TiO_2 Nano fillers that were added to heat cure PMMA and test the effect of this addition on the some mechanical and physical properties of heat cured acrylic denture base material.

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MATERIALS AND METHODS

Silanization Process

According to Arkle's equation, the minimum amount of silane required to create monolayer of silane coating on the fillers as follows: Amount of silane (g)= Amount of filler (g) × Surface area of fillers (m^2/g) /Minimum coating area of silane coupling agent (m^2/g).

One hundred milliliter of ethanol aqueous solution (70 vol %) was prepared using 99.8 vol% ethanol and de-ionized water (30 % vol), and adjusted to pH of 4.5 using PH meter through titrating with 99.9% acetic acid. (TMSPM) silane coupling agent were added respectively into each ethanol aqueous solution, and stirred. Hunderd (100) grams of titania Nano particles were added into each TMSPM solutions.

The mixture was stirred with magnetic stirrer for 20 minutes, then the mixture was sonicated with probe sonication apparatus for 30 minutes, then the solution was left to dry at room temperature for 14 days [(6)]. The (FTIR) spectrophotometer was used to determine whether or not functional group of the TMSPM have been attached to Nano filler by analyzing characteristic vibrations of functional groups [(6)].

Specimens grouping

One hundred (100) specimens were prepared. The specimens were divided into 2 groups, 50 heat cure PMMA specimens without additives (control) and 50 heat cure PMMA with silanized

TiO_2 Nano fillers addition (Experimental). Each group of specimens was sub divided into 5 groups according to the test selected.

Preparation of the test specimens

a- Transverse strength test, hardness test, and surface roughness test: a bar shaped specimens with dimensions of 65 mm length, 10 mm width, and 2.5 mm thickness[7]

b- Impact strength test: a bar shaped specimen with dimension of 80 mm x10 mm x4 mm length, width and thickness respectively [8].

c- Water sorption and solubility test: circular shaped specimen with diameter 50 mm and thickness 0.5 mm [7]

The mould was prepared by using separating medium for coating the plastic pattern. The lower portion of the metal flask was filled with dental stone. The plastic patterns were inserted to approximately one half on their depth, the upper half of the flask was filled with stone.

Proportioning and mixing of acrylic

Table 1: Proportioning and mixing of acrylic

TiO_2 conc	Amount of TiO_2	Amount of polymer	Amount of monomer
0%	0 g	50	22.7 ml
3%	1.5 g	48.5	22.7 ml

Addition of silanized titania Nanofillers:

Nano TiO_2 fillers were added to the monomer, the fillers were well dispersed in the monomer by ultra sonication, by using a probe sonication apparatus **(120 W, 60 KHz)** for 3 minutes[9].Then the conventional stages of packing, clamping and finishing of the specimens were followed[7].

Mechanical and physical tests:

Impact strength test: Specimens were construced,stored in distal water at 37 C for 48 hrs in incubator.The impact strength test was performed with impact testing device N43-1(Tinius Olsen,USA).

Impact strength= $E/B.D \times 10^3$ [8]

E: is the impact absorbed energy in joules.

B: is the width of the specimens in millimeter.

D: is the thickness of the specimens in millimeter.

Transverse strength test: All the specimens were immersed in distilled water in the incubator at 37°C for (48) hours.Test was performed using a universal Instron testing machine(Tinius Olsen,USA). Each specimen was positioned on the bending fixture, the load was applied with a cross head speed of 1mm/min by a rod placed centrally between the supports making deflection until fracture occurs.

Transverse strength= $3PL/2bd^2$

P: is the peak load

L: is the span length(50mm)

b: is the sample width

d: is the sample thickness [10].

Surface roughness test

The Profilometer device (surface roughness tester, TH 210, CHINA) was used.3 locations were selected, one in the middle and two at the periphery then the analyzer pass along the specimen surface and the mean of three readings were recorded for each specimen [7].

Surface hardness test

Test was performed using durometer hardness tester TR 220, CHINA (shore D hardness).The usual method was to press down firmly and quickly on the indenter and to record the maximum reading. Five measurements were recorded on different areas of each specimen and an average of these five readings was recorded [7].

Water sorption and solubility test: Water sorption and solubility measurement was done according to ADA Specification No12 [10].

RESULTS

FTIR Result The results of infra red (IR) Spectra were obtained by analyzing the characteristic vibrations of functional groups in Nano-TiO_2, modified Nano-TiO_2 help to clarify the interaction of Nano-TiO_2 with TMSPM.

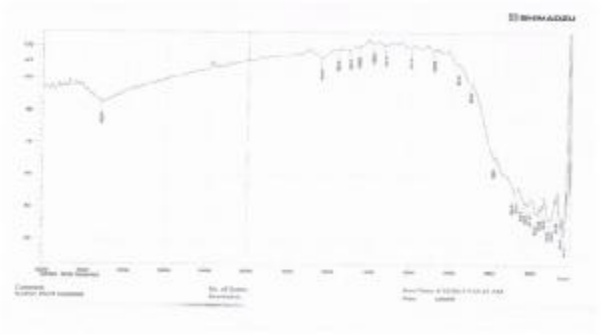

Figure 1: IR of non silanized nano TiO_2

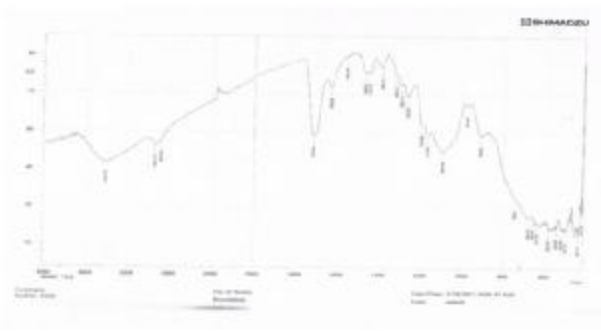

Figure 2: IR of silanized nano TiO_2

SEM Result: the result of SEM showed that + p-p-Nano titania fillers were less than 50 nm in size

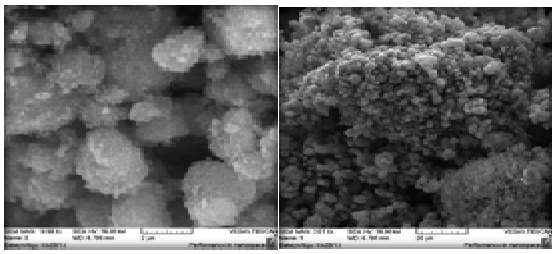

Figure 3: Nano titania filler under SEM

Mean values, standard deviation, standard error, maximums and minimums of the impact strength test results with t-test parameters are presented in Table 2, 3, and figure 4.

Table 2: Descriptive data of impact strength

	Control	3%
Mean	7.3	8.76
SD	0.402	0.496
SE	0.127	0.156
Max	7.743	9.349
Min	6.486	7.951

Table 3: t-test analysis for impact strength

t-test	p- value	Significance
6.819	0.000	HS

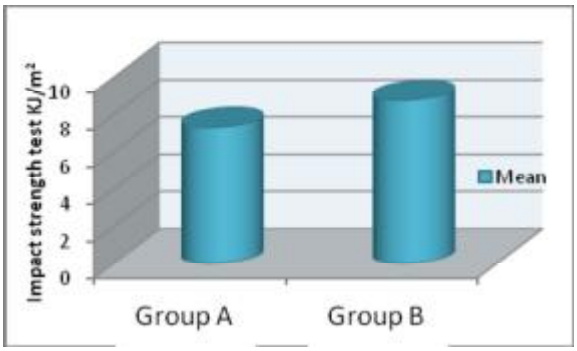

Figure 4: Bar chart of Impact strength means of studied groups

Mean values, standard deviation, standard error, maximums and minimums of the transverse strength test results with t-test parameters are presented in Table 4, 5, and figure 5.

Table 4: Descriptive data of transverse strength

	Control	3%
Mean	97.02	117.92
SD	1.392	2.92
SE	0.44	0.92
Max	99.1	122.3
Min	94.7	111.7

Table 5: t-test analysis for transverse strength

t-test	p- value	Significance
20.41	0.000	HS

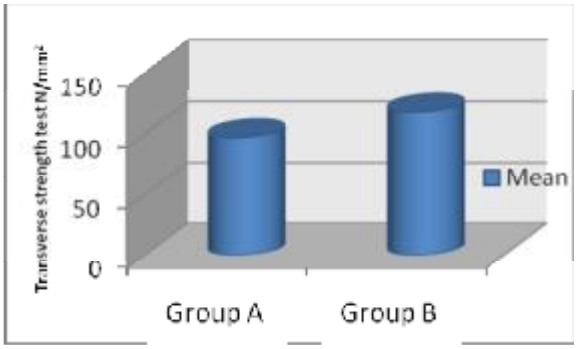

Figure 5: Bar chart of transverse strength means of studied groups

Mean values, standard deviation, standard error, maximums and minimums of the surface hardness test results with t-test parameters are presented in Table 6, 7, and figure 6.

Table 6: Decriptive data of surface hardness

	Control	3%
Mean	84.37	85.14
SD	0.95	0.61
SE	0.30	0.19
Max	85.3	86.2
Min	82.49	84.5

Table7: t-test analysis for surface hardness

t-test	p-value	Significance
2.315	0.033	S

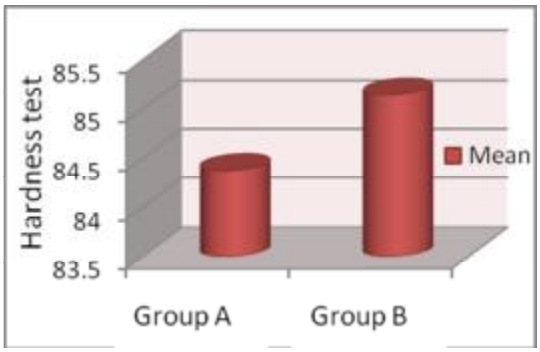

Figure 6: Bar chart of surface hardness means of studied groups

Mean values, standard deviation, standard error, maximums and minimums of the surface roughness test results with t-test parameters are presented in Table 8,9, and figure 7.

Table 8: Descriptive data of surface roughness

	Control	3%
Mean	0.28	0.38
SD	0.083	0.084
SE	0.026	0.026
Max	0.38	0.44
Min	0.12	0.23

Table 9: t-test analysis for surface roughness

t-test	p- value	Significance
2.857	0.01	S

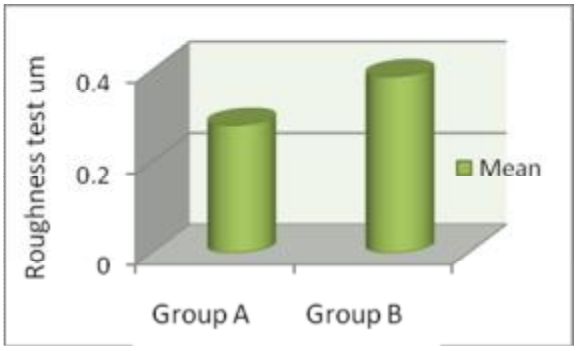

Figure 7: Bar chart of surface roughness means for studied groups

Mean values, standard deviation, standard error, maximums and minimums of water sorption test results with t-test parameters are presented in Table 10, 11, and figure 8.

Table 10: Descriptive data of water sorption

	Control	3%
Mean	0.35	0.33
SD	0.013	0.004
SE	0.005	0.001
Max	0.337	0.346
Min	0.338	0331

Table 11: t-test analysis for water sorption

t-test	p- value	Significance
3.796	0.001	HS

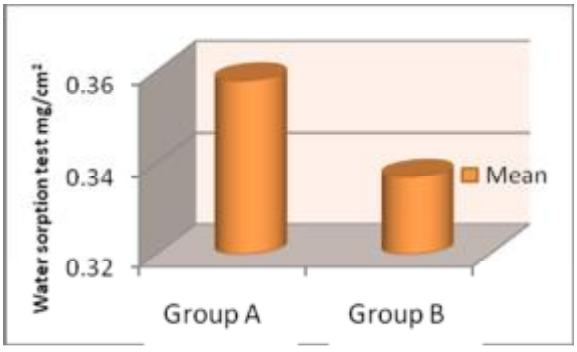

Figure 8: Bar chart of Water sorption means for studied groups

Mean values, standard deviation, standard error, maximums and minimums of water solubility test results with t-test parameters are presented in Table 12, 13, and figure 9.

Table 12: Descrptive data of water solubility

	Control	3%
Mean	0.017	0.016
SD	0.00104	0.00033
SE	0.00125	0.00039
Max	0.0192	0.018
Min	0.0163	0.014

Table 13: t-test analysis for water solubility

t-test	p- value	Significance
3.056	0.007	HS

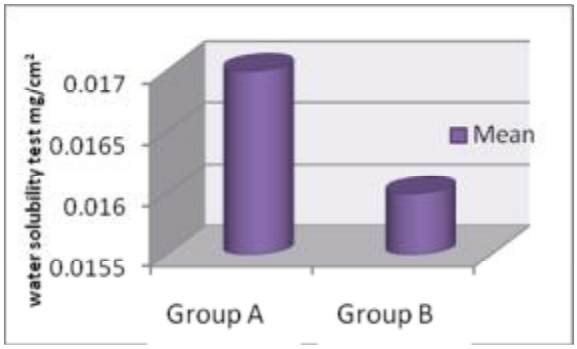

Figure 9: Bar chart of water solubility means of studied groups

DISSCUSION

The addition of silanized nano –TiO_2 to heat cured acrylic was done to evaluate the physical and mechanical properties. Titania (TiO_2) was used because it is biocompatible material (non toxic) and with pleasing color [11,12]. The two most useful properties of TiO_2 are corrosion resistance and the highest strength-to-weight ratio of any metal oxide [13]. The addition of silanized Nano-TiO_2 increased the value of impact strength, this may be due to high interfacial shear strength between nano fillers and matrix, also the crack propagation can be decreased by good bonding between nano filler and matrix [14], as particle size decrease, the total particle/matrix interfacial surface area available for energy dissipation increase and the critical stress for particles/matrix debonding also increase [15], also the addition of TiO_2 Nano fillers may lead to form efficient network (3Dimentional network) of PMMA and TiO_2 Nano particles thus lead to reduce the segmental motion [16]. There was increasing in the value of transverse strength when 3% of nano TiO_2 were added to PMMA compared with the control group, This may be due to a well dispersion of the <50 nm size of Nano particles

and fill the spaces between chains [17], segmental motion of the macromolecular chains were of the resin [18].

On the other hand, the increase of the transverse strength of the denture base with silanized TiO_2 Nano filler might be due to transfer of stresses from more the flexible polymer matrix to the higher modulus [19]. It was found that the hardness and surface roughness increased significantly, the increasing in surface hardness might be due to two factors: higher fillers content and use of silane coupling agent [17]. The increasing in surface roughness might be due to the presence of nano particles on the surface of each specimen. In this study there was a highly significant decrease in water sorption and solubility of acrylic resin, the decreasing in water sorption might be due to the formation of porosity and micro voids among polymer chains which facilitate fluid trasnsport into and out of polymer. TiO_2 nano particles used were insoluble in water and could reduce the overall volume of absorbing polymer [20], the use of silane coupling agent in silanization process of nano particles could lead to a reduction in the amount of water that reached to the inner layers of polymer matrix [21], Also the decrease in water sorption could be due to the fact that titania Nano particles replaced hydrophilic resin, result in a decrease in water uptake. The decreasing in water solubility could be attributed to the fact that titania nano fillers were insoluble in water which would lead to decrease the overall solubility of acrylic resin.

In conclusion; the addition of TiO_2 nano particles to heat cured acrylic resin improve the impact strength, transverse strength and surface hardness of heat cured acrylic resin, at the same time this addition decrease water sorption and solubility. On the other hand there was an increase in surface roughness with the addition of 3% silanized nano TiO_2.

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الخلاصة

بيان المشكلة: ميثاكريلات هو المادة الاكثر استخداماً في تصنيع طقم الاسنان. المواد بعيدة عن المثالية في الوفاء بالمتطلبات الميكانيكية. كان الغرض من هذه الدراسة هو لتقييم اضافة 3% بالوزن من حبيبات اوكسيدالتيتانيوم النانوية المعالجة سطحياً لطقم الاسنان الاكريللي الحراري الراتنجي.

المواد والطرق: تم تصنيع 100 عينة، 50 منها اعدت من الاكريلك دون اضافات (القياسي) و 50 عينة الاخرى محضرة من الاكريلك الحراري مع اضافة حبيبات اوكسيدالتيتانيوم النانوية تم تقسيم كل مجموعة الى 5 مجموعات فرعية وفقاً للاختبارات التي اجريت وقد تم الخلط من قبل المسبار الة صوتية فائقة.

النتائج: لوحظ وجود زيادة كبيرة للغاية في قوة الصدمة والقوة العرضية عند اضافة حبيبات اوكسيد التيتانيوم النانوية للاكريلك الحراري الراتنجي و زيادة قليلة في صلابة السطح وخشونته مع انخفاض بشكل ملحوظ في امتصاص وذوبانية الماء مقارنة مع المجموعة القياسية.
الاستنتاج: ان اضافة حبيبات اوكسيد التيتانيوم النانوية المعالجةسطحيالمادة الاكريلك الحراري تحسن من قوة الصدمةوالقوةالعرضية وصلبة السطح وكذلك تقلل من امتصاص وذوبانية الماء من ناحية اخرى هناك زيادة في خشونة السطح.