The effect of Silicon di oxide Nano -Fillers reinforcement on some properties of heat cure polymethyl methacrylate denture base material

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ABSTRACT

Background: The purpose of this study was to evaluate the effect of addition of surface treated silicon dioxide Nano filler (SiO_2) on some properties of heat cured acrylic resin denture base material (PMMA). The properties were impact strength, transvers strength, and surface hardness.

Materials and methods: In addition to controlled group SiO_2 powder was added to PMMA powder by weight in three different percentages 3%, 5% and 7%, mixed by probe ultra-sonication machine.120 specimens were constructed and divided into 3 groups according to the test (each group consist of 40 specimens) and each group was subdivided into 4 sub-groups according to the percentage of added SiO_2 (finally each subgroup consist of 10 specimens). The tests conducted were impact strength (charpy test), transverse strength and indentation hardness (shore D).

Results: A highly significant increase in impact strength and transverse strength was observed with the addition of SiO_2 powder to (PMMA) at the percentage of 3% and 5%; while a significant reduction occurred in both impact and transverse strength specimen's tests at the percentage of 7% A Highly significant increase in surface hardness was observed at the percentage of 3%, 5%and7.

Conclusion: The addition of Nano SiO_2 powder to acrylic resin improves the impact strength and transverse strength of acrylic resin at the same time this addition increase surface hardness with the increase in the concentration of Nano SiO_2 particles.

Keywords: PMMA (heat cure acrylic), Silicon di oxide Nano-filler (SiO_2), Nano technology. **(J Bagh Coll Dentistry 2014; 26(1):32-36).**

INTRODUCTION

Despite the widespread use of (PMMA) in prosthetic dentistry the fracture of dentures is a common clinical occurrence in prosthodontic service and still remains as an unsolved problem ⁽¹⁾.To overcome their physical and mechanical limitations, polymers had been reinforced by adding materials such as metal strengtheners, carbon-graphite fiber, aramid fiber, and glass fiber ⁽²⁾. Compared with glass-fiber reinforcement, Silanated Nano filler may be the material of choice for reinforcing denture base polymers because of their well-documented improvement in flexural properties and fatigue resistance, as well as good esthetic quality ⁽³,⁴⁾. In the chemical industry, during the past 10 years, research has been devoted to the development of a new industrial process that incorporates nanoparticles into (PMMA) providing a new class of (PMMA) that offers the strength of the Nano-oxides, with the flexibility of polymer matrix⁽⁵⁾.Nano oxide as SiO_2, TiO_2, and ZrO_2 are characterized by their small size, large specific surface area, active function, and strong interfacial interaction with the organic polymer ⁽⁶⁾. Therefore, they can improve the physical, thermal and optical properties of the organic polymer, as well as provide resistance to environmental stress-caused cracking and aging of denture base ⁽⁷⁾. Amorphous particles usually adopt a spherical shape (due to their microstructural isotropy), whereas the shape of anisotropic microcrystalline whiskers corresponds to their particular crystal habit ⁽⁷⁾. At the small end of the size range, nanoparticles were often referred to as clusters. Spheres, rods, fibers, and cups are just a few of the shapes that have been grown. The study of fine particles is called micromeritics ⁽⁸⁾·Increased bond strength by utilization of silanes is a result of a complex set of factors: wet out, surface energy, boundary layer absorbtion, polar adsorption, acid-base interaction, interpenetrating network formation and covalent reaction ⁽⁹⁾.The general formula of an organosilane (***RnSiX (4-n)***) shows two classes of functionality. The X functional group was involved in the reaction with the inorganic substrate SiO_2. The bond between X and the silicon atom in coupling agents were replaced bond between the inorganic substrate and the silicon atom. **X** is a hydrolyzable group, typically, alkoxy, acyloxy, amine, or chlorine. The most common alkoxy groups are methoxy and ethoxy, which give methanol and ethanol as byproducts during coupling reactions ⁽¹⁰⁾.

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MATERIALS AND METHODS

Silicon Di Oxide (SiO_2) Nano filler With Epoxy coupling agent, Heat–curing acrylic (powder and liquid), Hard dental stone type III and Separating medium (Tin foil substitutes) were used. One hundred and twenty specimens were prepared for this study. The specimens were divided into 3 groups according to the tests selected. Each group consisted of 40 specimens and these were subdivided according to the concentration of SiO_2 (Silicon dioxide Nano filler) into four sub groups as follow:

1- Group (A) control group (50 specimens of acrylic resin without SiO_2).
2- Group (B) 3% modified group (50 specimens of acrylic resin + 3% by weight (SiO_2).
3- Group (C) 5%modified group of (50 specimens of acrylic resin+5% by weight (SiO_2).
4- Group (D) 7% modified group of (50 specimens of acrylic resin + 7% by weight (SiO_2).

Ratio of mixing ratio of $SiO_2$with PMMA

SiO_2		Amount of polymer	Amount of monomer
0%	0	100g	40ml
3%	3g	97g	40ml
5%	5g	95g	40ml
7%	7g	93g	40ml

Plastic model preparation:

Three plastic models were constructed by cutting plastic plate of different gauge (0.5, 2.5 and 4 mm) into desired shape and dimension using high accurate laser cutting machine (figure 1).

Figure 1: laser cutting machine

1. Surface roughness test and Shore D. hardness test bar shaped specimen with dimension of (65 mm x 10 mm x 2.5 ± 0.1 mm) length, width, thickness respectively (11).

2. Transverse strength test bar shaped specimen with dimension of (65mm x 10 mm x 2.5 ± 0.1 mm) length, width, thickness respectively (11).

3. Impact strength test: bar shaped specimen with dimension of (80 mm x10 mm x 4 mm) length, width and thickness respectively (12).

Mould preparation

Conventional flasking technique for complete dentures was followed during the Mould preparation. Separating medium (cold mold seal) was used for coating the plastic model and allowed to dry, before investing the lower portion of the metal flask which was filled with dental stone and mixed according to the manufacturer's instructions with vibration to get rid of the trapped air, then left to set. The plastic model was inserted to approximately one half of their depth as to be easily removed after setting of the stone fig (2).

Figure 2: Mould preparation

Addition of Silicon di oxide Nano fillers

The addition of modified SiO_2 Nano filler powder was done by weight in four groups; the addition includes 3%, 5%, and 7% to monomer. An electronic balance with accuracy of (0.001g) was used, the filler well dispersed in the monomer by ultra-sonication type of mixing using probe sonication apparatus (120W, 60KHz) fig (3) for three min. to brake them into individual Nano crystals *(13)*.The suspension of monomer with SiO_2 Nano filler was immediately mixed with acrylic powder to reduce the possibility of particle aggregation and phase separation.

Figure 3: Probe sonication apparatus

Packing of acrylic resin

Packing was started when the acrylic reached to dough stage, the resin removed from the Jar and rolled, then packed into the mold previously coated with separating medium with the aid of polyethylene sheets. The two portions of the flask were closed together and placed under the hydraulic press, and the pressure was slowly applied on the flask so that the dough evenly flows throughout the mould space. The pressure was released, the flask was opened and the excess material was removed by sharp scalpel. A second trail closure was performed. The stone surface was again coated with the separating medium, allowed to dry and the polyethylene sheet was removed. Finally the two portion of the flask were closed until an intimate contact obtained, and left under press (20 bars) for 5 minutes before clamping, and then transferred to the water bath

Curing

This was done by placing the clamped flask in a water bath and processed by heating at 74°C for about one hour and half. The temperature then increased to boiling point for 30 minutes (14).

Finishing and polishing

All the specimens were finished and .polished with a lathe- polishing machine, flashes of acrylic were removed with W&H laboratory engine and an acrylic bur to get smooth surface stone bur was used followed by (120) grain sand paper with continuous cooling (immersed in cold water in rubber bawl, polishing was accomplished by using bristle brush and ruge wheel with pumice in lathe polishing machine, a gloss surface was obtained by using chamois baff and polishing swap. All specimens were measured by using digital vernier to make sure about their dimension. The speed of polishing machine was low speed (1500) rpm. With continuous cooling In order to avoid excessive heat, this may lead to distortion of the specimens

Impact strength test

A- Test specimens

The specimens were prepared with dimension (80mm x 10 mm x 4mm) (12)for unnotched charpy specimens Impact strength test.Ten Specimens of each concentration were prepared make a total of (40) specimens for impact strength measurements. Acrylic Specimens were stored in distilled water at 37°C for 48 hour on the incubator before the test (11).

B-Testing equipment and procedure

The impact strength test was evaluated following the procedure recommended by the use of impact testing device (12). The specimens were supported horizontally at each end and struck by free swinging pendulum of two Joules and digital display to show the impact energy fig (4). The scale reading gives the impact energy in Joules. The Charpy impact strength of unnotched specimens was calculated in kilo Joules per square meter (KJ/M2)

Figure 4: Impact testing device

Transverse strength test

A- Specimen design:

The specimens used were prepared with dimensions of (65mm x 10mm x 2.5mm) (11).Ten specimens for each concentration plus the control will make a total of (40) specimen for the measurement of transverse strength. All the specimens were immersed in distilled water on the incubator at 37°C for (48) hours before testing (11).

B-Testing procedure:

Test were performed using a universal Instron testing machine, each specimen was positioned on the bending fixture which consists of two parallel supports (50)mm apart, the full scale was 50 Kg, and 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 fig (5).

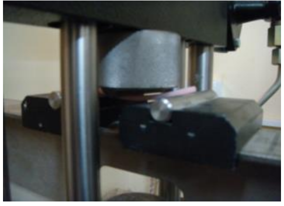

Figure 5: Universal Instron testing machine

Surface hardness testing

A- Specimen design:

The specimens were prepared with dimensions of (65mm x 10mm x 2.5mm) according to (11). Ten specimens for each concentration plus the control will make a total of (40) specimen for the testing of surface hardness. All specimens were immersed in distilled water for (48) hours before testing (11).

B-Testing procedure**:** Test was performed using durometer hardness tester (shore D hardness) that was fabricated by (HARTIP 3000compant) according to (11) which is suitable for acrylic design material.The instrument consists of a blunt pointed indenter (0.8 mm in diameter) that present in a cylinder (1, 6 mm in diameter) .The indenter was attached to a digital scale that is graduated from 0 to 100 unit the usual method was to press down firmly and quickly on the indenter and recorded the maximum reading as the shore D hardness, measurements were taken directly from the digital scale reading. Five measurements were recorded on different areas of each specimen (the same selected area of each specimen) and an average of five reading was calculated fig (8).

Figure 8: Durometer hardness tester

RESULTS AND DISCUSSION

Impact strength

Table 2 shows the means, standard deviations, standard error of the means, minimum and maximum values of experimental specimens measuring impact strength in different concentrations of SiO_2 Nano filler.

Table 2: distractive data of Impact strength parameters analysis (Kj/m^2)

		Group A	Group B	Group C	Group D
N		10	10	10	10
Mean		8.61	9.18	9.617	7.77
SD		0.264	0.113	0.168	0.365
SE		0.835	0.359	0.532	0.115
Range	**Min.**	8.1	9	9.42	7.2
	Max.	8.9	9.4	9.9	8.1

The highest mean value appeared in group C with a mean of 9.617 Kj/m^2 and the lowest mean was in group D which was 7.77 Kj/m^2.The increase of impact strength due to interfacial shear strength between Nano filler and matrix was high due to formation of cross-links or supra molecular bonding which cover the Nano filler and prevent cracks. Also the crack propagation can be changed by good bonding between Nano filler and matrix (16)

Transverse Strength

Means, Standard deviations, standard error of mean value, minimum and maximum values of the transverse strength of the acrylic resin are listed in Table 3 for different groups of added concentrations of the Nano filler

Table 3: Descriptive data of Transverse strength parameters (N/mm^2)

		Group A	Group B	Group C	Group D
N		10	10	10	10
Mean		114.937	120.145	124.562	111.6
SD		1.455	0.864	1.56	0.506
SE		0.835	0.359	0.532	0.115
Range	**Min.**	112.43	118.99	121.99	110.99
	Max.	116.68	122.1	127.01	112.08

From table 3 which plots the different means of transverse strengths across different concentrations of the added SiO_2 Nano filler show that the highest mean appeared in group C which was124.562 contrast to group D with mean of111.6 which showed the lowest mean. The increase in transverse strength that occur with addition of 3%SiO_2 Nano particles due to good distribution of the <100nm size of Nano particles enable them to inter between liner macromolecules chains. Segmental motion of the macromolecular chains were restricted and lead to increase strength and rigidity of the risen so this improved the fractural resistance and lead to improve transverse strength (17).

Surface hardness: Table 4 shows the means, standard deviations, standard error of the means, minimum and maximum values of experimental specimen measuring surface hardness in different concentrations of SiO_2 Nano filler.

Table 4: descriptive data of Surface hardness parameters analysis

		Group A	Group B	Group C	Group D
N		10	10	10	10
Mean		83.611	84.81	85.149	85.327
SD		0.767	0.919	0.558	0.332
SE		0.242	0.290	0.176	0.105
Range	**Min.**	82.44	83.01	84.24	84.4
	Max.	84.48	86.011	86.23	85.55

The highest mean value appeared in group D with a mean of 85.327 and the lowest mean was in group A (control) which was 83.611. This increase may be attributed to the randomly distributed particles of a hard material (Silicon di oxide) into acrylic matrix. The slightly increase of the hardness of the Nano composite at low Nano particles concentration (3%) would be dominated by network density, while increased hardness of the Nano composite at%5and7% attributed to the accumulation of the (SiO_2) particles in to the acrylic matrix specially on the surface (18).

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