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INVESTIGATION SOME MECHANICAL PROPERTIES OF SELF CURED PMMA RESIN REINFORCED BY DIFFERENT TYPES OF NANO PARTICLES

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ABSTRACT :-

In the present research, study the effect of adding two different types of reinforcing particles, which included: nano-alumina (nano-Al_2O_3) and nano-silica (nano-SiO_2), that added with different volume fractions of (1%, 2% and 3%), on the some mechanical properties of composite prosthesis complete denture base materials by using self (cold) cure poly methyl methacrylate (PMMA) resin as new fluid resin matrix **. In** this research the composite prosthetic dentures specimens consist of from two groups were prepared by using casting methods, type (Hand Lay-Up) method according to the types of reinforced particles which includes: the first group consists of PMMA resin reinforced by nano-alumina particles, and the second group consists of PMMA resin reinforced by nano-silica particles. The mechanical tests were performed on these specimens include compression test, impact test, and hardness test . **The** result of this study showed the values of (compression strength and hardness) properties increased with increasing the volume fraction of both (nano-Al_2O_3 and nano-SiO_2) particles in PMMA complete denture base materials. While, the values of (impact strength) property decreased **. Also** the addition of (nano-Al_2O_3) particles has a noticeable effect on the most properties of composite material for prosthetic denture base specimens more than the (nano-SiO_2) particles, except the impact strength property .

KEY WORDS : PMMA, Nano-Al_2O_3 Particles, Nano-SiO_2 Particles, Compression Strength, Impact Strength, and Hardness.

الأستقصاء من بعض الخصائص الميكانيكية لراتنج البولي مثيل ميثا أكريليت المقوى بانواع مختلفة من الدقائق النانوية

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الخلاصة :

في هذا البحث تم دراسة تاثير اضافة نوعين مختلفين من دقائق التقوية تضمنت دقائق الالومينا النانوية ودقائق السيليكا النانوية والتي تمت اضافتهما بكسور حجمية مختلفة هي (1%,2%,3%)، على بعض الخواص الميكانيكية لمواد قاعدة طقم الاسنان الأصطناعية الكاملة المتراكبة، وذلك باستخدام راتنج البولي مثيل ميثا اكريليت المعالج ذاتياً كمادة أساس على شكل راتنج سائل جديد . في هذا البحث حضرت عينات أطقام الاسنان الأصطناعية المتراكبة باستخدام طرق السباكة، باستخدام طريقة (الصب اليدوي) على شكل مجموعتين هي: المجموعة الاولى تضم راتنج البولي مثيل ميثا اكريليت مقواة بدقائق الاولومينا النانوية، والمجموعة الثانية تضم راتنج البولي مثيل ميثا اكريليت مقواة بدقائق السيليكا النانوية.

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تضمنت الاختبارات الميكانيكية التي تم اجرائها على هذه العينات فحوصات الانضغاط، الصدمة، والصلادة للعينات. وقد أظهرت نتائج هذه الدراسة بان قيم خواص (مقاومة الانضغاط والصلادة) ازدادت مع زيادة الكسر الحجمي لكلا من دقائق الالومينا النانوية ودقائق السيليكا النانوية. بينما قلت قيم خواص (مقاومة الصدمة) . كذلك ان دقائق الالومينا النانوية تمتلك تاثير اكبر من دقائق السيليكا النانوية على أغلب خواص المواد المتراكبة لعينات قاعدة طقم الاسنان الأصطناعية، ماعدا خاصية مقاومة الصدمة .

INTRODUCTION :-

Dental Materials, it is the science which deals with the materials used in the dentistry, their physical, chemical, mechanical properties and with their manipulation as such properties are related to the proper selection and use by the dentist. The study of dental material enables the dentist to understand the behavior of these materials, proper selection for appliance in patient and how to use them to their best advantage [Anusavice K. J., 1996].
Prosthetic dentistry is meaning the replacement of missing teeth, which may have been lost for a variety of reasons, with either fixed or removable dentures, that using depending upon a many factors of these replacements [Wachter W., 2009]. Many biomaterials are available in medicine and dentistry fields. Biomaterials can be classified as metals, polymers, ceramic and composites materials. Polymeric materials have a many applications for implantation since they are available in a wide variety of compositions and properties [Park, J. et. al., 1992]. The poly (methyl methacrylate) is one type of thermoplastic polymer and it is more type of acrylic resin used as denture base materials, the first using self-curing of PMMA resin was introduced in Germany since (1938) [Wachter W., 2009]. The composite materials have many important properties which make them suitable for many industrial uses. This is approach the scientist to study the effected of different type of reinforced materials on the mechanical properties for the composite material, such as (compression strength, impact strength, hardness, surface roughness and tensile, etc.). The literature surveys include some researches which are accomplished in this field it's:

Schajpal S. B. et. al., [1989], this study investigates the effect of adding the (silver, copper and aluminum) powders to acrylic denture base resin in different volume fraction. They found when increased, all these types of metal fillers lead to increased compressive strength, but decrease in tensile strength, also the fillers increased thermal conductivity but did not proportionally as the filler concentration increased.

Panyayong W. et. al., [2002], this study investigates the effect of adding the mixture of titanium dioxide (TiO_2) powders and Zirconium dioxide (ZrO_2) powders, on the some mechanical and physical properties of PMMA resin with different volume fraction (1%, 2%, and 3%) in ratio of 1:1, 1:2, 2:1. The results showed that addition of (TiO_2) and (ZrO_2) particles in these ratio combinations, lead to decrease the water absorbed, and increased flexural strength as well as the impact strength of PMMA resin.

Bandugula U. C. et. al., [2005], this study investigates the effect of adding (nano-TiO_2) particles with different volume fraction on PMMA denture resin. The result indicated that stress intensity and impact strength values increase with increasing the concentration of titanium oxide up to (1% vol) and subsequently decrease at higher concentration.

Ali S. A., [2006], this study investigates the effect of adding the (Ti powder and ZrO_2 powder) on some properties of hot cure PMMA denture base material. The results showed that adding of Ti powder lead to slightly increase in the transverse strength, but slightly decrease both impact strength and hardness. While the addition of ZrO_2 powder lead to slightly increases in the hardness and surface roughness but highly decrease both impact strength and flexural strength.

Azlan A. et. al., [2008], this study investigate the effects of adding the hydroxyapatite (HA) with different volume fraction (5%, 10%, 15%, 20%), on the flexural properties, fracture toughness and thermal properties of heat cure PMMA resin to form PMMA/HA composites. The results showed the flexural modulus of PMMA was increased by increasing the volume fraction of HA particles this is attributed to the reinforcement effects of HA particles. Ayman E. et. al., [2008], this study investigates the effect of adding the aluminum (Al_2O_3) particles with different weight fraction (5%, 10%, 15%, and 20%), on the flexural strength and thermal diffusivity of heat-polymerized acrylic resin. It was found increased significantly the flexural strength and thermal diffusivity in proportion to the weight percentage of alumina (Al_2O_3) particles. Increasing the flexural strength and heat transfer characteristics of the acrylic resin base material could lead to more patient satisfaction. Ihab N. S., [2011], this study investigate the effects of adding the nano-zirconium oxide (nano-ZrO_2) particles with different weight fraction (2%, 3%, 5% and 7%) on some properties of heat cure PMMA denture base material. The results showed that addition of (nano-ZrO_2) particles lead to increase impact strength and flexural strength of heat cure PMMA resin and reaches to the maximum value at (5 %) of (nano-ZrO_2) particles, and then strength decreases with further increase of (nano-ZrO_2) particles. Also the addition of (nano-ZrO_2) particles lead to slightly increases in the hardness. Chow W. S. et. al., [2013], this study investigate the effects of adding the hydroxyapatite (HA) particles on the flexural properties of heat cure poly methyl methacrylate denture base materials, to prepared the PMMA/HA composites. The results showed that the flexural modulus of PMMA was increased with increase of HA particles, while the flexural strength and flexural strain of PMMA was decreased with increase of HA particles. In order to withstand against any denture fracture and avoid or reduce the incidence problems in denture base materials. In this research, using new type of denture base materials as new fluid resin matrix, and studying the effect of some reinforcing particles (nano-Al_2O_3 and nano-SiO_2) particles with selected volume fractions on the (compression, impact and hardness) tests of the composite materials for prosthetic complete denture base.

MATERIALS USED :-

Acrylic Resin Denture Base Material

In this research the composite prosthetic complete dentures specimens consist of polymer matrix and reinforced particles materials. Matrix material included cold curing PMMA that used in this research as new fluid resin matrix, type (Castavaria), made from (Vertex – Dental Company), to preparation specimens of composite prosthetic denture base. Vertex™ Castavaria is a multifunctional self-polymerizing acrylic which is perfectly useable as a pouring, relining, rebasing and as a repair acrylic.

This type of materials distinguishes by many properties compared with other type of PMMA polymer such as: softer feel, low molecular weight, color stable in the long run, minimized shrinkage, stable polymerization cycle with a perfect end result, the acrylic is long pourable and modelable for a long period of time. But have some disadvantage properties such as: low strength, low hardness and more difficult using during fabrication [William. J & O' Brain, 2002]. Table (1) shows some physical and mechanical properties of self-cure PMMA resin, type (Castavaria) that used in this study, according to (Vertex – Dental Company).

Particles Reinforcement Materials

Two types of ceramic particles were used in this study as reinforces materials with selected volume fraction of (1%, 2% and 3%), the additions include the following:

Nano Aluminum Oxide (Al_2O_3 99.9%) Particles

Aluminum oxide is represented one of the most cost effective and widely used of ceramic materials. Aluminum oxide is supplied as (nano-particles), also it is possesses excellent size, shape and high purity about (99.9%). The structure of aluminum oxides have strong ionic bonding which gives its high strength, stiffness, hardness, wear resistant, and good thermal conductivity of alumina [Power. J. M. et. al., 2006].
The result of particle size and particle size distribution of (nano-Al_2O_3) particles is obtained by using atomic force microscopy (AFM) was carried out in baghdad university laboratories, which shows the average diameter was (57.48 nm). The result of particle size distribution of (nano-Al_2O_3) particles is shown in the Figure (1). Table (2) shows the mechanical and physical properties of (Al_2O_3) particles.

Nano Silicon Oxide (SiO_2) Particles

Silicon oxide is represented one of the most complex and most abundant families of ceramic materials. Silicon oxide is supplied as (nano-particles), each unit of silicon oxide includes one atom of silicon and two atoms of oxygen. Silicon oxide has a high chemical resistance, good thermal shock resistance, excellent strength, good transparent and good electrical insulation [Power. J. M. et. al., 2006]. The result of particle size distribution of (nano-SiO_2) particles is obtained by using atomic force microscopy (AFM) was carried out in baghdad university laboratories, which shows the average diameter was (24.29 nm). The result of particle size distribution of (nano-SiO_2) particles is shown in the Figure (2). Table (3) shows the mechanical and physical properties of (SiO_2) particles.

PROCEDURE TO PREPARATION OF TEST SPECIMENS :-

Proportioning and Mixing of Acrylic

The PMMA denture base materials consist of polymer powder and monomer liquid (methyl methacrylate, MMA). The cold cure acrylic resin type (Castavaria), was used to prepare the PMMA composite specimens. The standard proportion in mixing ratio for cold cure (self-cure) acrylic resin is usually about (15 g) polymer powder (PMMA) and (9.5 g) monomer liquid (MMA) (1.5 g / 0.95g) by weight according to the manufacturer's instructions of manufacturer company. When mixing powder and liquid many changes will take place due to solution of polymer in the monomer. This ratio was effect on the workability of the mixture, dimensional changes and toxicity of acrylic resin specimens [Jorge. J. H. et. Al., 2003]. This type of cold cure acrylic resin is mouldable for a long period time, where the mixture was mixed of liquid (MMA) in the clean and dry glass beaker, then slow addition of dry powder (PMMA) to liquid (MMA), the mixture was stirred at room temperature continuously by using mechanical mixing (brabender plastograph mixer) at speed (20 r.p.m.) until reached to the dough stage and poured mixture in the center of opening mould with maximum time about (4.5 min) according to manufacture company. During mixture pouring inside the glass mould, the mould must be slow rocked and vibrated from side to side to remove any gas bubbles from the specimens, and reminder of the mixture was poured into mould hole until the glass mould filling and left mould to stand on the bench top at room temperature for (8-13 min) from beginning of mixing process as working time to increase the viscosity of mixture and surface of casting become hard.

Curing Cycle Process

According to the manufacture's instruction polymerization curing all specimens after casting process was placed inside the oven at (60 ºC) and let them for (30 min) according to manufacturer's instructions of manufacturer company, because of the specimens' complete polymerization under this condition. The advantage of this technique is polymerization may be accomplished in short time, post cured of specimens and give minimum level of residual monomer. The process of place the casting specimens into oven and the castings composite prosthetic dentures specimens outside the oven at room temperature to complete the cooling and complete hardening of specimen as shown in Figure (3).

MECHANICAL TESTS :-

The mechanical tests were performed in this study to evaluate some mechanical properties of the PMMA composite materials for the prosthetic denture base which include:

Compression Test

The compression test is performed according to (ASTM D695) by using the same tensile machine (universal testing machine), type (Instron) at across head (strain rate) of (5mm/min) and applied compressive load was (25 kN) on the specimen's top and bottom at the same time until the break of the specimen occur, five times were tested for each specimen and take the averaged for the final result of five specimens it was tested [Annual Book of ASTM Standard, 2002].

Impact Test

Izod impact is defined as the kinetic energy needed to initiate fracture and continue the fracture until the specimen is broken. The impact test is performed according to (ISO-180) and (ADA Specification No.12, 1999), by using Izod Impact test machine type is (XJU series pendulum Izod/Charpy impact testing machine). For Izod test: the specimen clamped at one end by fixing it on the base of the device and held vertically cantilevered beam, which it has broken at impact energy of (5.5 J) of pendulum and impact velocity about (3.5 m/s). Then release the pendulum to impact the specimen, then record amount of energy that required to broken the specimen, five times were tested for each specimen and take the averaged for the final result of five specimens it was tested [Annual Book of ISO Standard, 2006].

Hardness Test

The hardness test is performed according to (ASTM D2240) by used Dorumeter hardness test, type (Shore D), when place the specimen under the indenter area at load applied equal to (50 N) and depressing time of measuring equal to (15sec). The surface of specimens must be smooth in zone testing. The hardness value is very sensitive to the (specimen thickness, specimen diameter and distance from the edge more than 12 mm). Therefore, the minimum thickness of the specimen is (3 mm) with diameter more than (30 mm). Each specimen was tested seven times on different areas of each specimen at same time and make sure it's in the middle not on the edge of the specimen, finally the average value was taken [Annual Book of ASTM Standard, 2003].

All these tests carried out in air at room temperature (23 ± 2) ℃ after complete finishing and polishing processes, and immersion the specimens in distilled water at

(37 ± 1) ºC for (48 hr), in order to remove any residual monomer and release residual stress, also to ensure that the denture base materials remains in semi oral environment [Annual Book of ANSI/ADA Standard, 1999] .

RESULTS AND DISCUSSIONS OF MECHANICAL TESTS :-

Results and Discussion of Compression Test for Modified Composites

Chart (1) and Figure (4), shows the effect of adding both types of the ceramic particles which include (nano-alumina or nano-silica) with different volume fractions on the compressive strength of PMMA matrix. From this Figure can be noticed how the compressive strength values increased with the increasing of the volume fraction for both types of these particles in both groups of PMMA composite materials. This is due to the strengthening mechanism which meaning those particles reduced the slippage of the PMMA resin chains by filling the any spaces in the PMMA resin and it can also be related to the higher compressive strength of (alumina and silica) particles as compared with PMMA resin . **It** can also be noticed that the addition of nano-alumina particles has a noticeable effect on the compressive strength of PMMA composite specimens more than the nano-silica particles, therefore, compressive strength for first group specimens (PMMA - nano-Al_2O_3) composite is higher than the values of compressive strength for second group specimens (PMMA - nano-SiO_2) composite. This is due to the improvement of the mechanical properties that are associated with the addition of Al_2O_3 particles, that related to the nature of Al_2O_3 particles which has high compressive strength comparing with SiO_2 particles as shown from tables (2) and (3). Thus, the compressive strength values increased from (87.5 MPa) for PMMA specimen (as referenced) to reach to the higher value of compressive strength (535 MPa) for (PMMA - 3% nano-Al_2O_3) composite materials [WWW. SP System.Com., 2004] .

Results and Discussion of Impact Test for Modified Composites

The impact strength values results that obtained from impact test that carried out on PMMA composite specimens for prosthetic denture base materials that prepared in this research are shown in the Chart (2) and Figure (5), shows the effect of adding both types of ceramic particles (nano-alumina or nano-silica) with different volume fractions on the impact strength of PMMA matrix.From this Figure can be noticed how the impact strength values decreased with increasing the volume fraction for both types of these particles in both groups of PMMA composite materials, because of any increasing in these particles numbers, it will be act as points for localized stress concentration regions from which the failure will begin. Furthermore, the natural of these particles is brittleness and weakness in the ability of resistance to impact loads comparing with PMMA resin. Also may be because aggregated of nano-particles which have high surface energy [Ivor H., 1982].

It can also be noticed that the addition of nano-silica particles has a noticeable effect on the impact strength of PMMA composite specimens more than the nano-alumina particles, therefore, impact strength for first group specimens (PMMA - nano-Al_2O_3) composite are less decreasing than the values of impact strength for second group specimens (PMMA - nano-SiO_2) composite, this is due to the higher mechanical strength of Al_2O_3 particles comparing with SiO_2 particles. Thus, the impact strength value decreased from (7.45

KJ/m^2) for PMMA specimen (as referenced) to reach to the lower value of impact strength (2.73 KJ/m^2) for (PMMA - 3% nano-SiO_2) composite materials.

Results and Discussion of Hardness Test for Modified Composites

Chart (3) and Figure (6), shows the effect of adding both types of the ceramic particles which include (nano-alumina or nano-silica) with different volume fractions on the hardness of PMMA matrix. From this Figure can be noticed how the hardness values increased with the increasing of the volume fraction for both types of these particles in both groups of PMMA composite materials. This is due to the high hardness and brittleness of the (alumina and silica) particles as compared with PMMA resin alone, furthermore, related to the wettability and the bonding strength between the matrix and these particles, which lead to make the harder surface by restricting the movement of the matrix towards the direction of stress applied [W. Bolten, 1998].

It can also be noticed that the addition of nano-alumina particles has a noticeable effect on the hardness of PMMA composite specimens more than the nano-silica particles, therefore, hardness for first group specimens (PMMA - nano-Al_2O_3) composite is higher than the values of hardness for second group specimens (PMMA - nano-SiO_2) composite. This is due to the improvement of the mechanical properties that are associated with the addition of Al_2O_3 particles. Thus, the hardness values increased from (79.5 MPa) for PMMA specimen (as referenced) to reach to the higher value of hardness (83.5 MPa) for (PMMA - 3% nano-Al_2O_3) composite materials.

CONCLUSIONS :-

According to the experimental results of composite prosthetic complete denture base materials, which prepared in this research, can be the conclusions the following sentences:

1- Some mechanical properties such as (compressive strength and hardness) of PMMA composites prosthetic denture (PMMA - nano-Al_2O_3), (PMMA - nano-SiO_2), were increased with increasing of the volume fractions of (nano-Al_2O_3 and nano-SiO_2) particles.
2- Other mechanical property values such as (impact strength) of PMMA composites prosthetic denture (PMMA - nano-Al_2O_3), (PMMA - nano-SiO_2), were decreased with increasing of the volume fraction of (nano-Al_2O_3 and nano-SiO_2) particles.
3- The addition of (nano- alumina) particles has a noticeable effect on the most properties of composite prosthetic complete denture base specimens more than the nano-silica particles, except the impact strength property.
4- The maximum values for properties (compressive strength and hardness) were obtained in PMMA composites prosthetic denture (PMMA - nano-Al_2O_3), while the minimum values for impact strength property was obtained in PMMA composite prosthetic denture (PMMA - nano-SiO_2).

(Table 1): Some Mechanical and Physical Properties of Self Cure PMMA Resin.

Young's Modulus (GPa)	Impact Resistance (KJ/m^2)	Flexural Strength (MPa)	Water Absorption (%)	Density (g/cm^3)
1.63 - 3	8.3	79	2.5	1.19

(Table 2): Some Mechanical and Physical Properties of (Al_2O_3) Particles.

Compressive Strength (MPa)	Thermal Conductivity (W/m.K)	Water Absorption (%)	Density (g/cm^3)
2600	35	0	3.9

(Table 3): Some Mechanical and Physical Properties of (SiO_2) Particles.

Compressive Strength (MPa)	Thermal Conductivity (W/m.K)	Water Absorption (%)	Density (g/cm^3)
1108	1.38	0	2.3

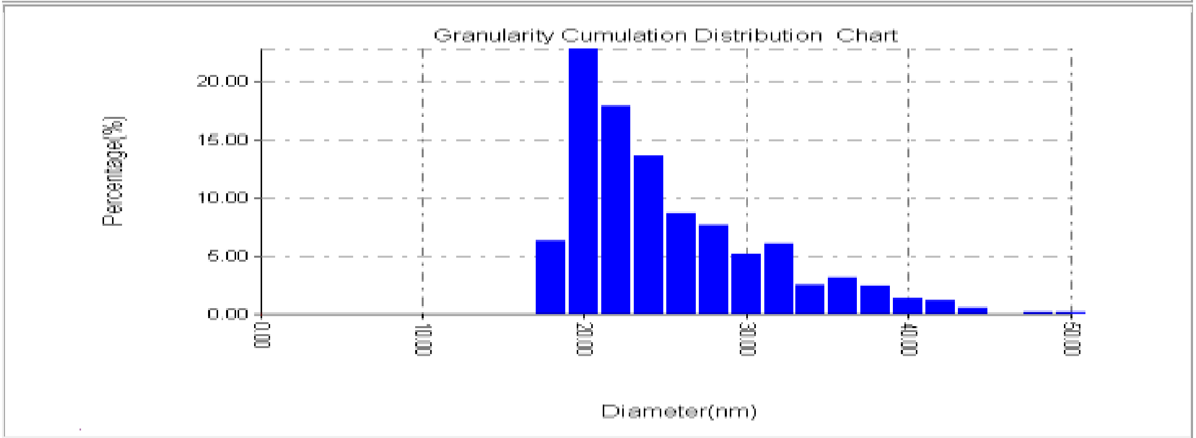
SPM Scanning Probe Microscope

Granularity Cumulation Distribution Report

Sample:SiO_2	**Code:**Sample Code
Line No.:lineno	**Grain No.:**822
Instrument:CSPM	**Date:**2016-01-04

Avg. Diameter:24.29 nm

Diameter (nm) <	Volume (%)	Cumulation (%)	Diameter (nm) <	Volume (%)	Cumulation (%)	Diameter (nm) <	Volume (%)	Cumulation (%)
18.00	6.33	6.33	30.00	5.23	82.12	42.00	1.22	98.91
20.00	22.75	29.08	32.00	6.08	88.20	44.00	0.61	99.51
22.00	17.88	46.96	34.00	2.55	90.75	48.00	0.24	99.76
24.00	13.63	60.58	36.00	3.16	93.92	50.00	0.24	100.00
26.00	8.64	69.22	38.00	2.43	96.35			
28.00	7.66	76.89	40.00	1.34	97.69			



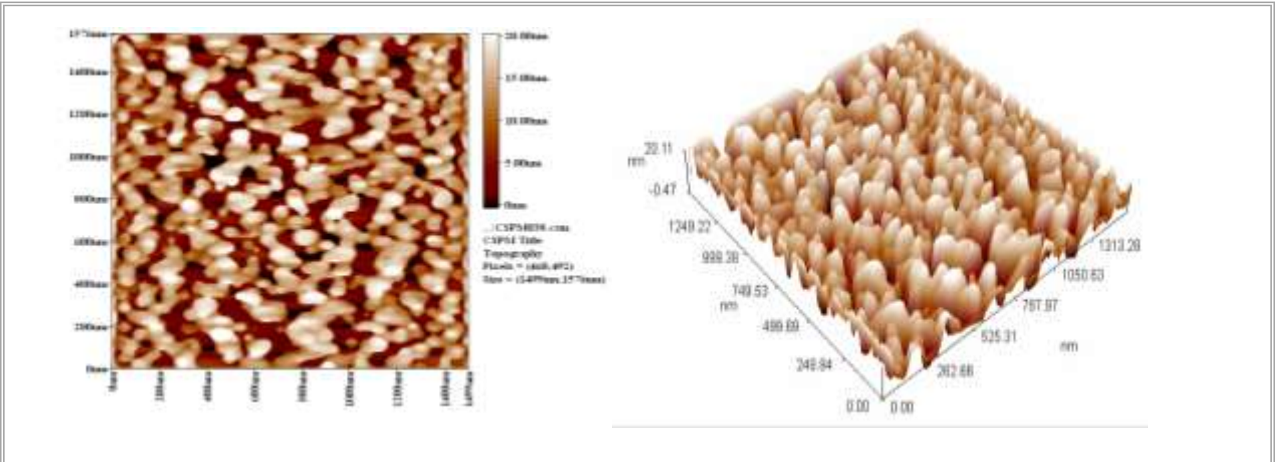


Fig. 1 AFM Test of Nano-SiO_2 Particles.

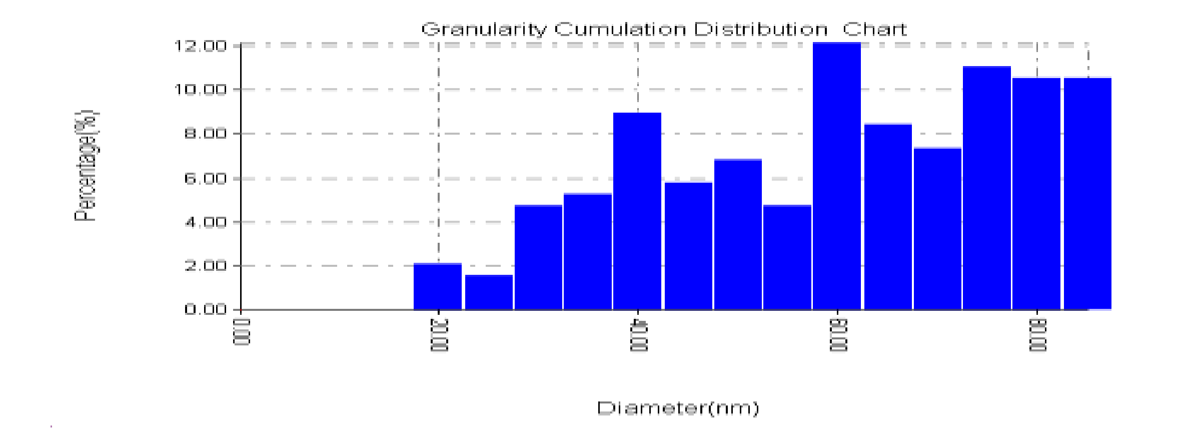
SPM Scanning Probe Microscope

Granularity Cumulation Distribution Report

Sample:Al_2O_3	**Code:**Sample Code
Line No.:lineno	**Grain No.:**190
Instrument:CSPM	**Date:**2016-01-04

Avg. Diameter:57.48 nm

Diameter (nm) <	Volume(%)	Cumulation (%)	Diameter (nm) <	Volume (%)	Cumulation (%)	Diameter (nm) <	Volume (%)	Cumulation (%)
20.00	2.11	2.11	45.00	5.79	28.42	70.00	7.37	67.89
25.00	1.58	3.68	50.00	6.84	35.26	75.00	11.05	78.95
30.00	4.74	8.42	55.00	4.74	40.00	80.00	10.53	89.47
35.00	5.26	13.68	60.00	12.11	52.11	85.00	10.53	100.00
40.00	8.95	22.63	65.00	8.42	60.53			



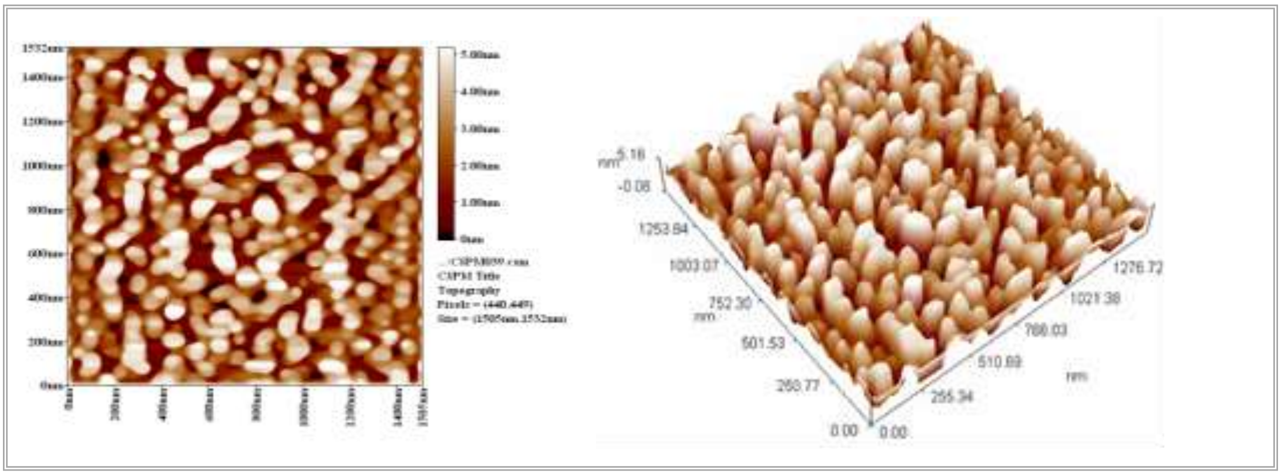


Fig. 2 AFM Test of Nano-Al_2O_3 Particles.

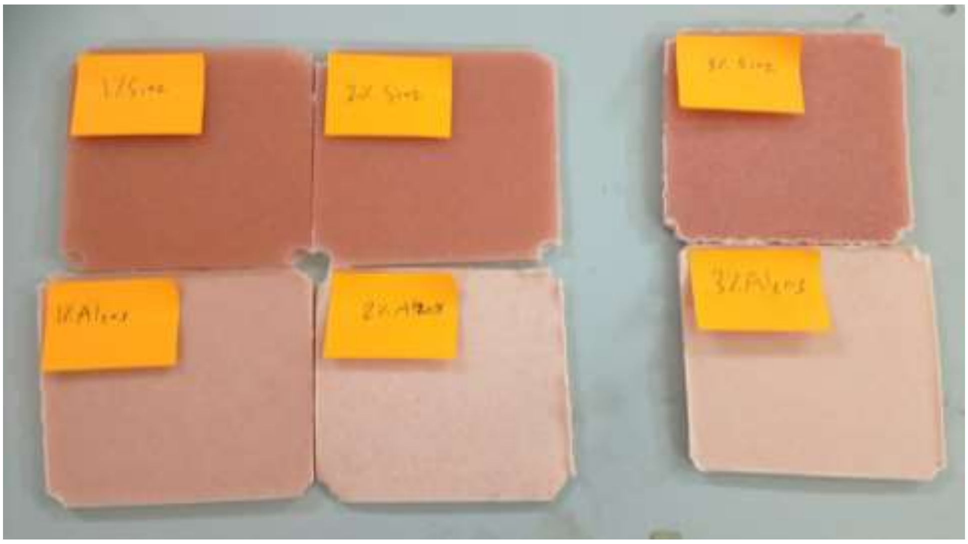
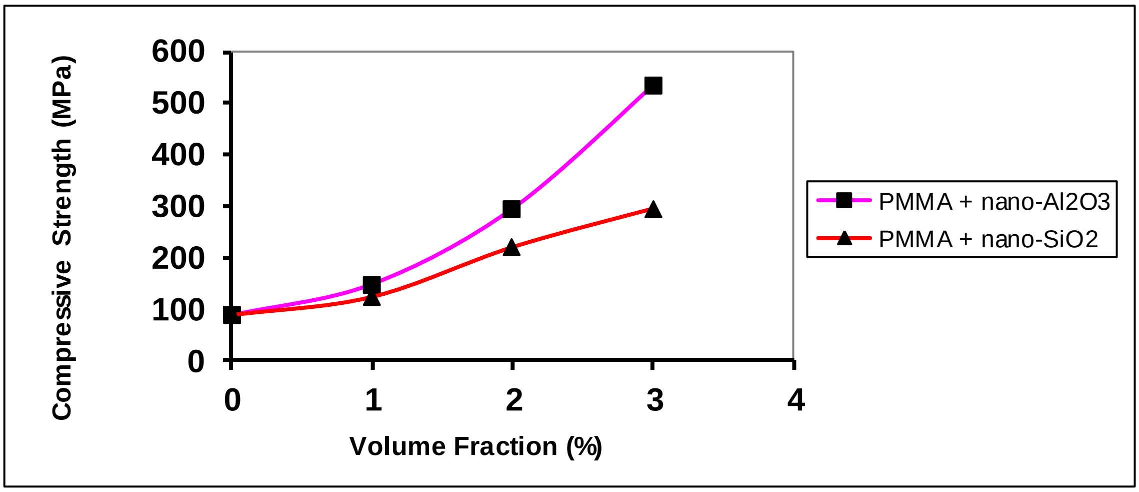


Fig. 3 Composite Prosthetic Dentures Specimens after Curing Process.



(Fig. 4): Compressive Strength of PMMA Composite Materials as Function of (Nano-SiO_2 and Nano-Al_2O_3) Particles (Vol %) in Composite.

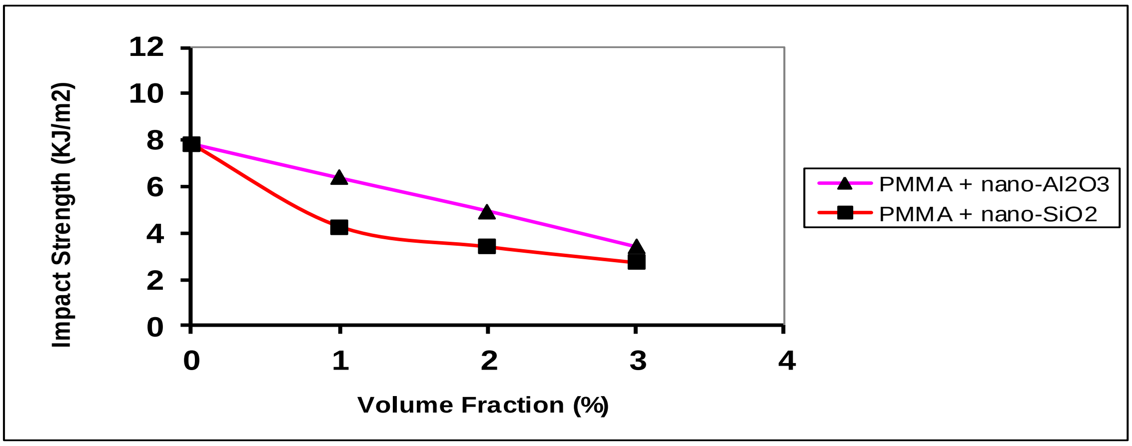


Fig. 5 Impact Strength of PMMA Composite Materials as Function of (Nano-SiO2 and Nano-Al2O3) Particles (Vol %) in Composite.

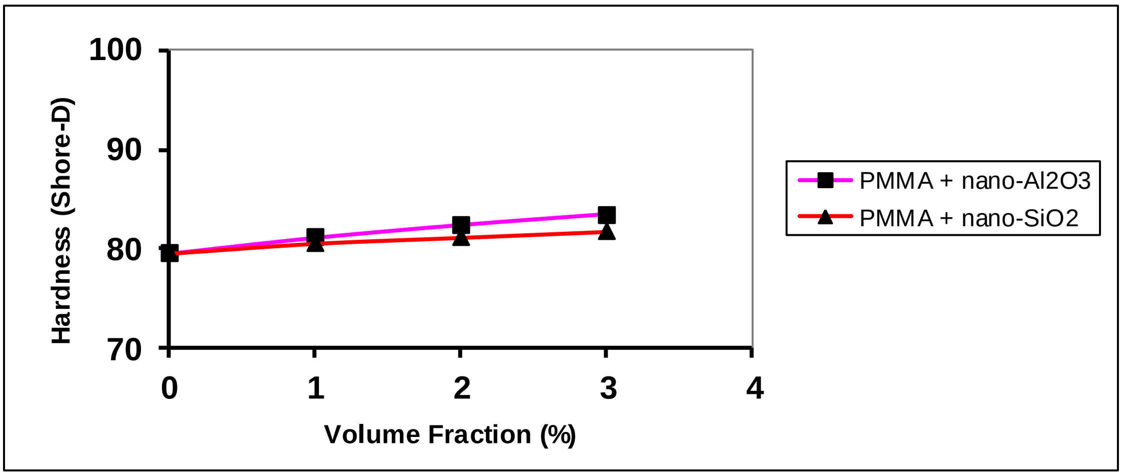


Fig. 6 Hardness (Shore-D) of PMMA Composite Materials as Function of (Nano-SiO2 or Nano-Al2O3) Particles (Vol %) in Composite.

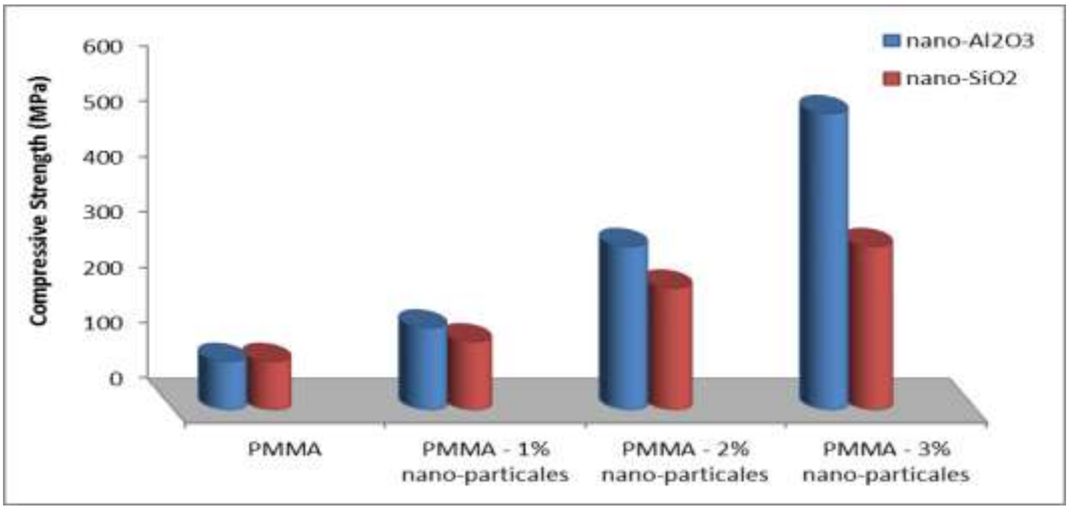


Chart 1 Compressive Strength Data of PMMA Composite Denture Base Materials.

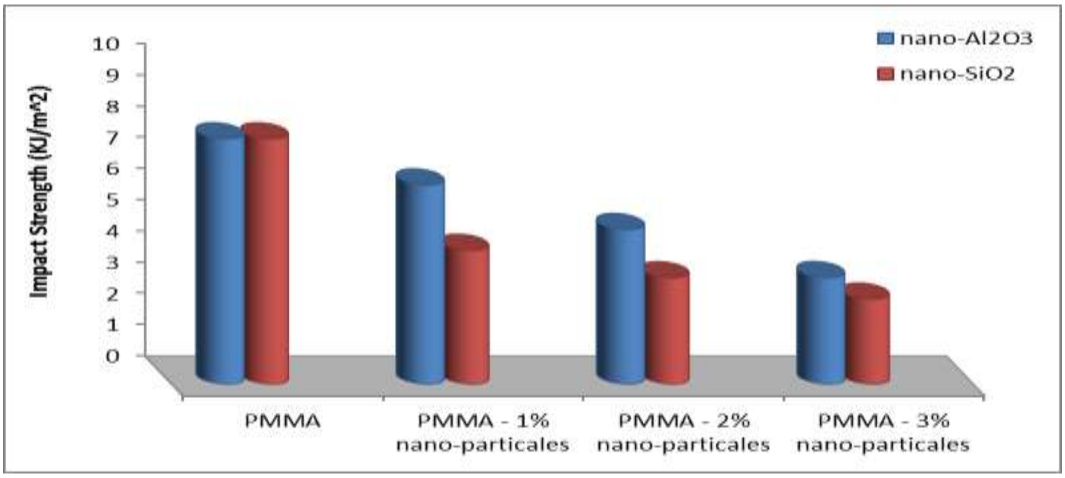


Chart 2 Impact Strength Data of PMMA Composite Denture Base Materials.

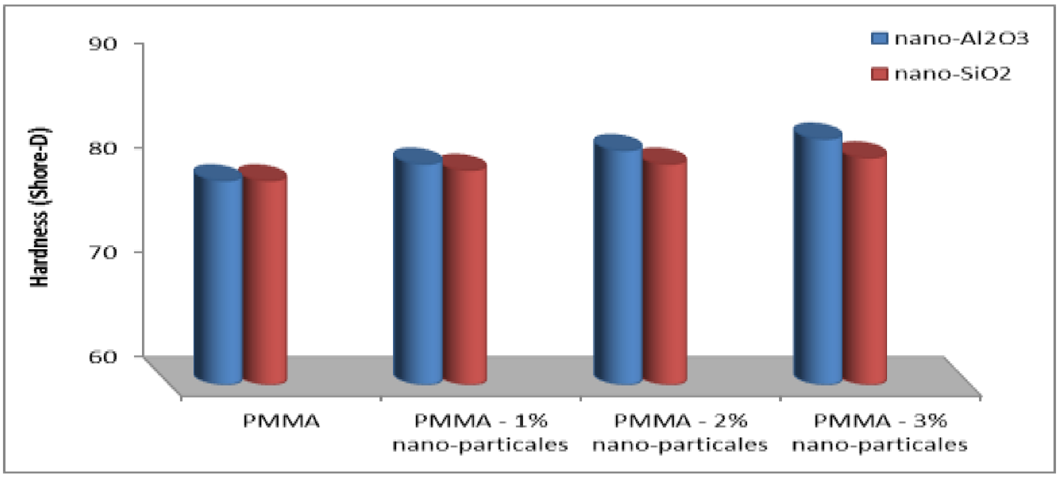


Chart 3 Hardness Data of PMMA Composite Denture Base Materials.

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