



STUDY OF FLEXURAL AND IMPACT PROPERTIES OF NANO-HYBRID COMPOSITES MATERIALS BY USING POLY METHYL METHACRYLATE (PMMA) MATRIX

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Abstract: The present work studies the effect of adding nano-silica (nano-SiO₂) particles, that were added with different volume fractions of (0.5 %, 1 % and 1.5 %), and random woven fiber glass with fixed volume fraction of (3 %), on some mechanical properties of composite prosthesis complete denture base materials by using poly methyl methacrylate (PMMA) resin as matrix. In this work, the prosthetic dentures base specimens consist of two groups include particulate composites materials and nano-hybrid laminated composites materials, according to the types of reinforced materials that were prepared by using Hand Lay-Up method, the first group consists of PMMA resin reinforced by nano- silica particles, only, while the second group consists of PMMA resin reinforced by nano- silica particles and random woven fiber glass. The mechanical tests were performed on these specimens include (flexural and impact tests). The results showed that the addition of (nano-SiO₂) particles has a noticeable effect on the most properties of particulate composite material specimens and nano-hybrid laminated composites materials specimens for prosthetic denture base, the values of (flexural modulus, flexural strength, shear stress and impact strength) properties increased with the increase of the volume fraction of (nano-SiO₂) particles in the composite denture base material, whereas the values of (flexural strain) property decreased.

Keywords: PMMA, Nano-Silica, Flexural, Shear Stress and Impact.

دراسة خصائص الانحناء والصدمة لمواد متراكبة هجينية نانوية ذات أساس من البولي مثيل ميثا أكريليت

الخلاصة: في البحث الحالي تم دراسة تأثير اضافة دقائق السيليكا النانوية والتي تمت اضافتهما بكسور حجمية مختلفة هي (0.5 %، 1 % و 1.5 %)، مع حصيرة اليفاج العشوائية بكسر حجمي ثابت (3 %) على بعض الخواص الميكانيكية لمتراكبات مواد قاعدة طقم الاسنان الاصطناعية الكاملة، وذلك باستخدام راتنج البولي مثيل ميثا اكريليت كمادة اساس. في هذا البحث حضرت عينات اطقم الاسنان الاصطناعية على شكل مجموعتين تحتوي على مواد متراكبة دقائق هجينية نانوية بالاعتماد على نوع مواد التقوية. وقد تم تحضير هذه العينات باستخدام طريقة الصب اليدوي في قالب زجاجي، المجموعة الاولى تضم راتنج البولي مثيل ميثا اكريليت المقوى بدقائق السيليكا النانوية، بينما المجموعة الثانية تضم راتنج البولي مثيل ميثا اكريليت المقوى بدقائق السيليكا النانوية مع حصيرة اليفاج العشوائية. تضمنت الاختبارات الميكانيكية التي تم اجرائها على هذه العينات فحوصات (الانحناء والصدمة). وقد اظهرت نتائج هذه الدراسة ان دقائق السيليكا النانوية تمتلك تأثير اكبر على معظم خواص المواد المتراكبة الدقائقية والمواد المتراكبة الطباقية الهجينية النانوية لعينات قاعدة طقم الاسنان الاصطناعية، حيث ان قيم خواص (معامل مرونة الانحناء، مقاومة الانحناء، اجهاد القص الأقصى ومقاومة الصدمة) ازدادت مع زيادة الكسر الحجمي لدقائق السيليكا النانوية في متراكبات مادة قاعدة طقم الاسنان، بينما قلت قيم خاصية (الانحناء الانفعالي).

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1. Introduction

Prosthetic dentistry can be defined as replacement of missing teeth, which may have been lost for a variety of reasons, either fixed or removable dentures, are used depending upon many factors of these replacements. Synthetic polymeric materials have been used in dental materials, implant, dressing, encapsulate, polymeric drugs delivery, orthopedic devices and medical disposable supplies. Polymers can be classified as non resorbable and resorbable polymers. Some examples to non-resorbable polymers are polypropylene, polyethylene and poly methyl methacrylate. But the resorbable polymers include Poly glycolic acid and poly lactic acid, which means they degrade in body. Polymer composites materials have good elastic modulus, high strength and reduce the creep and high heat deflection compared to the pristine polymers and they are suitable for several orthopedic applications. Each type of materials has good properties which make them suitable for various biomedical applications [1].

The most commonly polymers used in biomedical and dentistry application are acrylic resin. In the 20th century, acrylic resin and other plastic materials were used as denture base materials due to their attractive properties. Acrylic resins are the most widely used in dentistry application and accepted among all denture base materials, and it was estimated that they represent (95%) of the polymer in prosthodontics applications [2].

The poly (methyl methacrylate) is one type of thermoplastic polymer and it is the most type of acrylic resin used as denture base materials, the first using self-curing of PMMA resin was introduced in Germany in (1938) [3]. The literature surveys include some researches which are accomplished in this field it's:

Kanie et. al., (2002), showed the effect of the position and number of woven glass fibers on the flexural strength, flexural modulus, impact strength, and fracture toughness of cold cure denture base resin. It was found that the flexural strength and fracture toughness of the denture base resin reinforced with woven glass fiber was enhanced by increasing the number of woven glass fibers, and increase the flexural strength and impact strength of denture base resin with glass fiber are higher than denture base resin only [4].

Ali S., (2006), this study investigates the effect of (Ti and ZrO₂) powders on some properties of heat cure PMMA denture base material. The results showed that adding of Ti powder lead to slightly increase in the flextural strength, but slightly decrease both impact strength and hardness. While the addition of ZrO₂ powder lead to slightly increases in the hardness and surface roughness but highly decrease impact strength and flexural strength [5].

Ihab N. (2011), studied the effect of addition of modified nano-zirconium oxide (ZrO₂) particles by tri methoxy sily prople methacrylate (TMSPM) on some properties of heat cure denture base in different percentages (2%, 3%, 5% and 7%) by weight. It was found that the addition of modified nano-ZrO₂ highly increase the impact and

flexural strength in nano-composite containing (5wt %) of nano-ZrO₂ then strength decreases with further increase [6].

Chow W. S. et. al., (2013), investigated the effect of hydroxyapatite (HA) particles on the flexural properties of a heat polymerizing PMMA denture base resin. The flexural modulus of PMMA/HA composites was increased by the addition of HA particles. But the flexural strength and flexural strain were decreased in the presence of HA [7].

2. Aim of This Work

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 different types of reinforcing shape (random woven fiber glass and nano-SiO₂ particles) with selected volume fractions on the (flexural and impact) tests of the composite materials for prosthetic complete and partial denture base.

3. Materials Used

3.1 Acrylic Resin Denture Base Material

The composite prosthetic complete dentures specimens consist of polymer matrix and reinforced particles materials. Most dentist using heat cure PMMA resin, while in this work using matrix material as cold curing PMMA that used as new fluid resin matrix as the first time, type (Castavaria), made from (Vertex – Dental Company), to preparation specimens of composite prosthetic denture base.

This type of materials is distinguished by having many properties compared with other type of PMMA polymer. 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).

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

Young's Modulus (GPa)	Impact Strength (KJ/m ²)	Flexural Strength (MPa)	Flexural Modulus (GPa)	Water Absorption (%)	Density (g/cm ³)
1.63-3	8.3	79	2.3	2.5	1.19

3.2 Particles Reinforcement Materials

The ceramic particle was used in this work as reinforced materials with selected volume fraction of (0.5 %, 1 % and 1.5 %). It was added to the acrylic powder including:

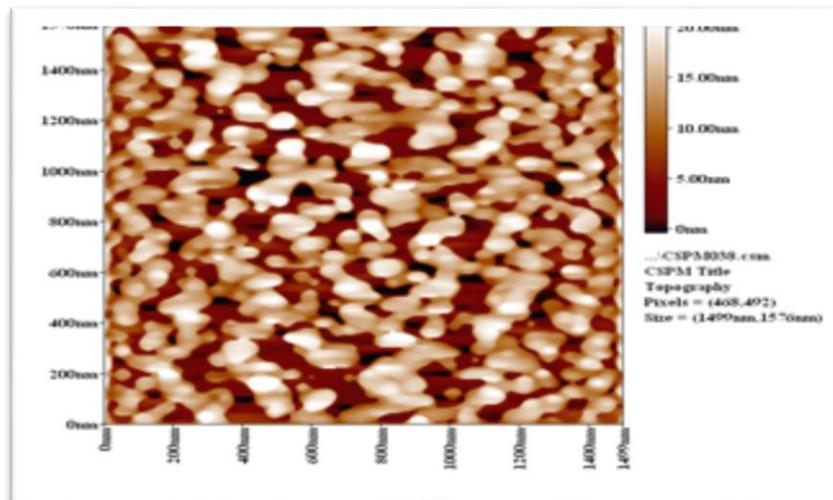
3.2.1 Nano Silicon dioxide (SiO_2) Particles

Silicon dioxide (SiO_2) is commonly referred to silica, which is represented one of the most complex and most abundant families of ceramic materials. Silicon dioxide is supplied as (nano-particles) which made by (Zhoushan Mingri Nanomaterials Co. Ltd., Zhejiang, China), each unit of silicon dioxide formed by strong covalent bonds and one atom of silicon and two atoms of oxygen. Silicon dioxide has a high purity (99.9 %), high chemical resistance, good thermal shock resistance, excellent strength, good transparent and good electrical insulation [8]. Table (2) shows the mechanical and physical properties of (SiO_2) particles.

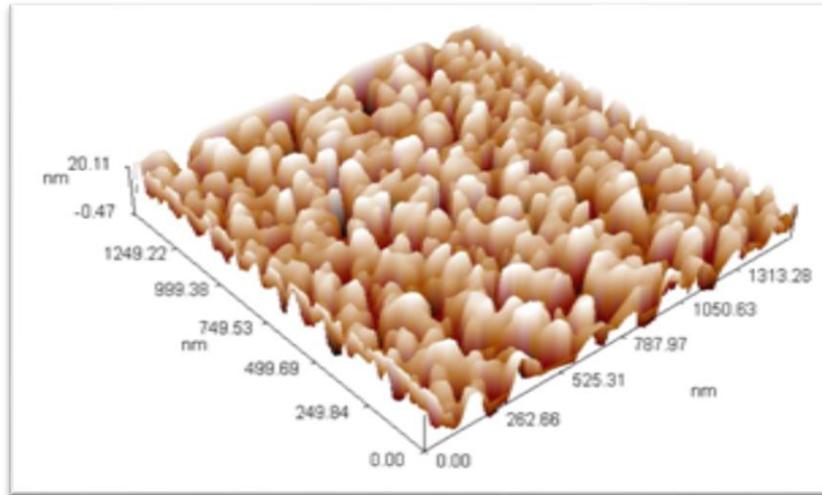
Table (2): Some Mechanical and Physical Properties of (SiO_2) Particles.

Compression Strength (MPa)	Water Absorption (%)	Thermal Conductivity (W/m.K)	Density (g/cm^3)
1108	0	1.38	2.3-2.65

The results of particle size and particle size distribution of (nano- SiO_2) particles is obtained by using atomic force microscopy (AFM), which was carried out in Baghdad university laboratories. The result shows that the average diameter was (24.29 nm). The result of particle size distribution for (nano- SiO_2) particles structures in (2-D) and (3-D) image is shown in Figure (1).



-a-



-b-

Figure (1): (a) 2-Dimensions and (b) 3- Dimensions of (Nano-SiO₂) Particles by AFM Test.

3.3 Fiber Reinforcement Materials

The fiber was used in this work as reinforced materials to the acrylic resin with constant volume fraction of (3 %) including:

3.3.1 Random Glass Fiber

The E-glass fiber used in this study is made by (Mowding Ltd. UK Company), as woven mat fibers, the fibers distribution in it as randomly fashion. This type of fiber is the most commonly used for commercial fiber reinforced materials which were immersed in matrix (PMMA) and in a polymer composite as a laminate arrangement.

4. Experimental Part

4.1 Mould Preparation

The mould was used is made of glass with dimensions (20cm × 20cm × 0.4cm) and covered with a glass plate to provide smooth surface of prepared specimens. The particulate composites materials and nano-hybrid laminated composites specimens were prepared by using casting methods, type (Hand Lay-Up) method. Also the thermal silicon was used to close any spaces that may be found in the glass mould and prevents any flow of acrylic resin out of mould.

4.2 Proportions and Mixing of Acrylic

The PMMA denture base materials consist of polymer powder and monomer liquid (methyl methacrylate, MMA). The standard proportion in mixing ratio is usually for this type of PMMA resin is (15 g) polymer powder (PMMA) and (10 ml) liquid monomer (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 is important as it effected on the workability of the mixture, dimensional changes and toxicity of acrylic resin specimens.

This type of acrylic resin is mouldable for a long period, where the mixture was mixed by weigh the required amount of PMMA powder and the nano-silica particle according to the extracted numbers by using rule of mixture by using the sensitive balance. Then mix the dry ingredients all together in one clean and dry glass beaker and try to make them softer by stirring it against the wall of the beaker in order to become smoother, follow weigh the liquid monomer (MMA) and add these powder mixture gradually into the liquid (MMA) material, the mixture was stirred at room temperature continuously by using mechanical mixing (brabender plastograph mixer) at medium speed (20 r.p.m.) until reached to the dough stage and poured mixture in the center of mould with maximum time about (6 min) before it starts to solidify as it's because it solidifies quickly. During mixture pouring inside the glass mould, the mould must be slow rocked to remove any gas bubbles from the specimens, and the reminder of the mixture was poured into mould hole until the glass mould filling and left mould at room temperature for (15 min) from beginning of mixing process as working time to increase the viscosity of mixture and casting surface became hard.

If using woven mat glass fibers to prepare the nano-hybrid laminated composites materials specimens must be cutting the woven mat fibers in rectangular shape of dimensions (19×19) cm which slightly less than those of the test mould (Glass mold) to avoid the exposure of glass fiber to the environment. The part of acrylic dough mixture was poured into the mould which nearly filled half of the mould then the pre-prepared woven mat glass fiber was placed over the lower half of the mixture by using tweezers then the other half of the mixture was poured into the mould over the woven mat fibers in this stage using glass rod during pouring of mixture in order to ensure the fiber well wetted with acrylic dough mixture. Then the mould was vibrated very gently and vibrated from side to remove any entrap gas bubbles that may be found in this mixture, which may cause creating the voids that may be affecting on the properties of the final nano-hybrid laminated composite materials.

4.3 Curing Cycle Employed

According to the manufacture's instruction, polymerization curing was carried out for the closed mould by placed it in autoclave. Therefore all specimens were then placed inside autoclave at (65°C) and pressure equal to (2.5 bar) and let for (60 min) according to manufacturer's instructions, to complete polymerization under this condition. The advantage of this technique is polymerization may be accomplished in short time, post cured of specimens and given minimum level of residual monomer. After polymerization completion, it was follow by curing process, about (30 min) at room temperature to

complete the cooling and complete hardening of specimen. After cooling, the specimens were de-mould to remove from the mould and cleaning.

All tests carried out were done at room temperature (23 ± 2) °C after complete finishing and polishing processes, and immersion the specimens in distilled water at (37 ± 1) °C 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 ^[9].

4.4 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 include:

4.4.1 Flexural Test

The flexural test is performed according to (ASTM D790) and (ADA Specification No.12, 1999), all data measured from three point bending test machine by using the same tensile machine at across head speed (strain rate) of (5 mm/min) and load was applied equal (5 kN) until break the specimen occur [10].

4.4.2 Maximum Shear Stress Test

The shear test is performed according to (ASTM D2344) and (ADA Specification No.12, 1999), all data measured from three points bending test machine by using (Hydraulic press) type (Leybold Harris No. 36110) with used short beam and gradually load applied [11].

4.4.3 Impact Test

Izod impact is defined as the kinetic energy needed to initiate fracture and continue the fracture until the specimen is broken. Impact test specimens might be with or without notch. 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). Unnotched Izod impact is a single point test that measures a material resistance to impact from a swinging pendulum, 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 [12].

5. Results and Discussion

5.1 Flexural Test

The flexural strength values of PMMA composite and hybrid composite specimens for prosthetic denture base materials that prepared in this research are shown in the Figure (2), this Figure show the effect of adding two different types of reinforcing shape (random woven fiber glass and nano-silica particles) on the flexural strength values of PMMA matrix.

From this Figure it can be noticed how the flexural strength values increased with the increase of the volume fraction of nano-silica particles in both of PMMA particulate composites and nano-hybrid laminated composites materials. This is due to ability of silica particles to obstacle the crack propagation inside PMMA matrix according to strengthening mechanism and the strong bonding between the PMMA matrix and these particles [13].

It can also be noticed that when adding random woven fiber glass in PMMA composite to form hybrid laminated composite material, the flexural strength increased as compared with PMMA particulate composite. This could be attributed to the fact that glass fibers and (nano-silica) particles are characterized by higher resistance toward the flexural strength than PMMA matrix and contribution of each (random woven fiber glass and nano-silica particles) to be carried out of the load applied on the hybrid laminated composite specimens. Furthermore, this increase may be due to higher bond strength that may occur between the PMMA and reinforcing material, all these reasons led to the improving of the flexural strength values of the hybrid laminated composite specimens [14]. Thus, flexural strength values increased from (82 MPa), for PMMA specimen (alone) to reach the higher values about (187.5 MPa) for hybrid laminated composite materials (PMMA - 3% Glass fiber - 1.5% nano-SiO₂).

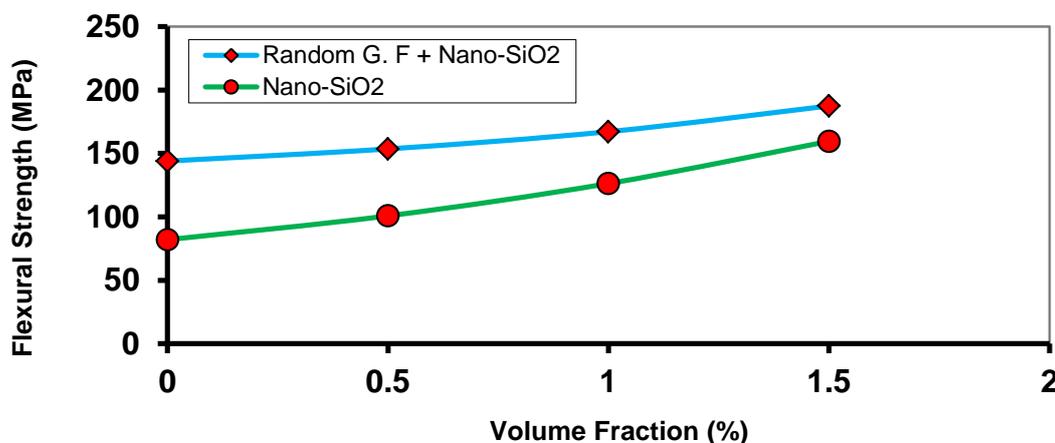


Figure (2): Flexural Strength of PMMA Particulate Composites and Nano-Hybrid Laminated Composites as Function of (Nano-SiO₂) Particles (Vol %).

The flexural modulus values results that were obtained from flexural test that was carried out on PMMA particulate composites and nano-hybrid laminated composites specimens for prosthetic denture base materials that prepared in this research are shown in the Figure (3), which shows the effect of adding two different types of reinforcing (random woven mat fiber glass and nano-silica particles) on the flexural modulus of PMMA matrix.

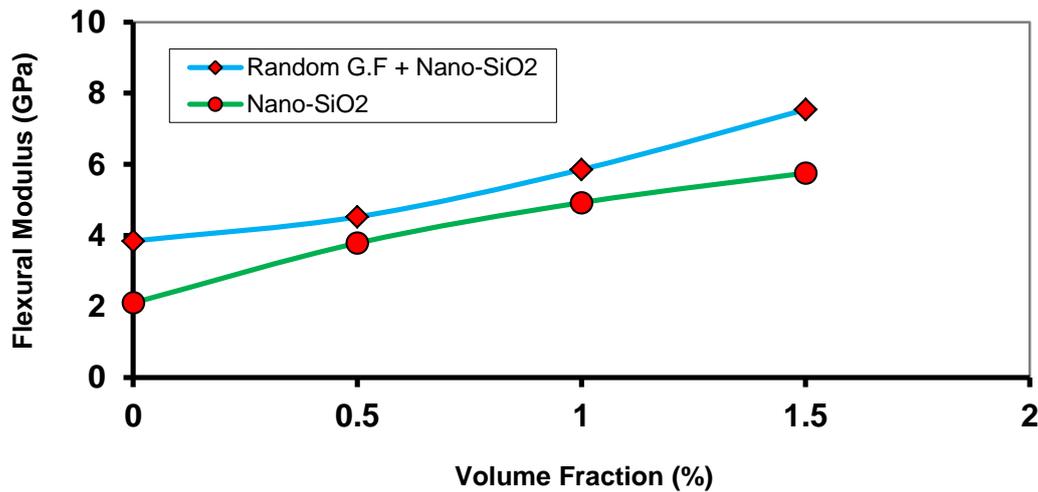


Figure (3): Flexural Modulus of PMMA Particulate Composites and Nano-Hybrid Laminated Composites as Function of (Nano-SiO₂) Particles (Vol %).

From this Figure it can be noticed how the flexural modulus values increased with the increase of the volume fraction of nano-silica particles in both of PMMA composite materials and hybrid laminated composite materials. This is due to the strengthening mechanism which mentioned the amount of these particles that played an important role by obstacle the motion of PMMA chains. And good wettability of these particles by (MMA) liquid that may cause an increase of the bonding force between the matrix and reinforced material, so the resultant composite will require high stress to break their physical bonding [15].

It can also be noticed that when adding random woven fiber glass in PMMA composite, the flexural modulus increased as compared with PMMA particulate composite. This could be attributed to the contribution of each (random woven fiber glass and nano-silica particles) to be carried out of the load applied on the hybrid laminated composite specimens that suit their mechanical properties, nature and their geometry. Furthermore, the regular and randomly distribution of nano-silica particles inside the PMMA matrix and ease penetration of the matrix material through the particles and woven mat fibers to give strong bonding between the matrix and reinforcing material, all these reasons lead to improve the flexural modulus values of the hybrid laminated composite specimens.

Thus, the flexural modulus value increased from (2.1 GPa) for PMMA specimen (alone) to reach the higher value of flexural modulus (7.54 GPa) for hybrid laminated composite materials (PMMA- 3% Glass fiber - 1.5% nano-SiO₂).

Also the results of the flexural strain that were obtained from flexural test that carried out on PMMA composite and hybrid composite specimens for prosthetic denture base materials that prepared in this research are shown in Figure (4), which shows the effect of adding two different types of reinforcing shape (random woven fiber glass and nano- silica particles) on the flexural strain of PMMA matrix.

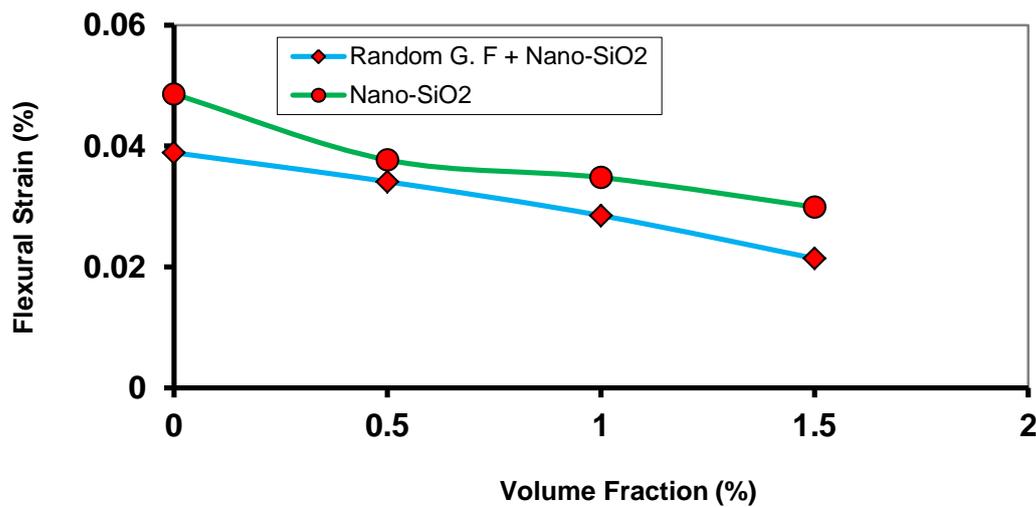


Figure (4): Flexural Strain of PMMA Particulate Composites and Nano-Hybrid Laminated Composites as Function of (Nano-SiO₂) Particles (Vol %).

It can be noticed from this Figure how the flexural strain values decreased with increasing the volume fraction of nano- silica particles in both PMMA composite materials and hybrid laminated composite materials, because of the decreasing in the flexural strain depends upon the interfaces bonding between the PMMA matrix and reinforcing material (nano-silica particles and random woven fiber glass) therefore the decreasing in the flexural strain may be attributed to the formation of a strong interfaces bonding between the PMMA matrix and reinforcing materials.

It can also be noticed that when adding random woven fiber glass in PMMA composite to form hybrid laminated composite material, the flexural strain decreasing as compared with PMMA matrix composite. This is due to share the random woven mat fiber glass with nano-silica particles to increase the bond strength between them, that led to increasing the flexural strength and decrease the flexural strain of nano-hybrid laminated composite [16].

Thus, the flexural strain value decreased from (0.0486 %) for PMMA specimen (alone) to reach the lower value of flexural strain (0.0214 %) for hybrid laminated composite materials (PMMA - 3% Glass fiber - 1.5% nano-SiO₂).

5.2 Maximum Shear Stress Test

The Maximum shear stress values of PMMA composite and hybrid composite specimens for prosthetic denture base materials that prepared in this research are shown in Figure (5), this Figure shows the effect of adding two different types of reinforcing (random woven fiber glass and nano-silica particles) on the Maximum shear stress values of PMMA matrix.

From this Figure it can be noticed how the Maximum shear stress values increased with the increase of the volume fraction of nano-silica particles in both of PMMA composite materials and hybrid laminated composite materials. This is due to ability of silica particles to obstacle the crack propagation inside PMMA matrix according to strengthening mechanism, in addition to the strong bonding between the PMMA matrix and reinforcing particles ^[13].

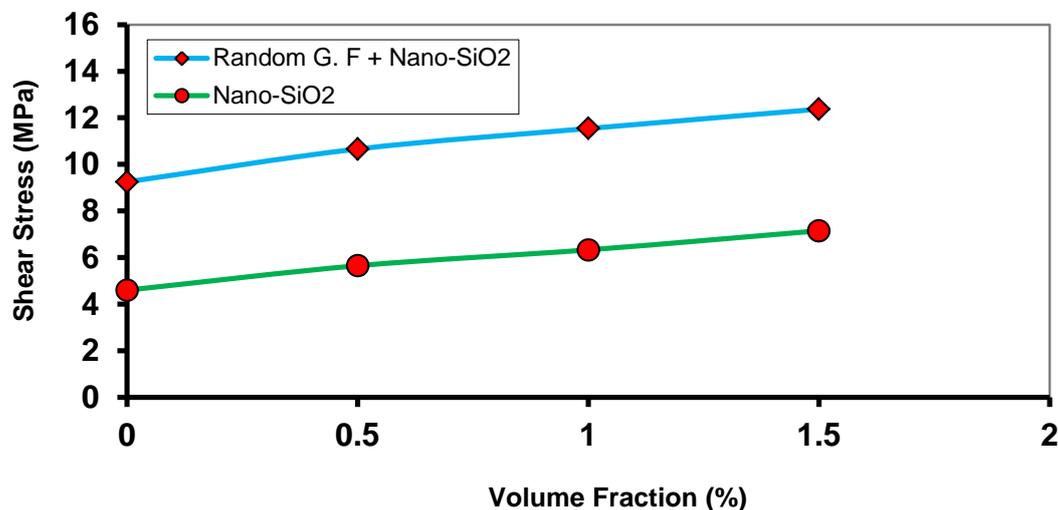


Figure (5): Maximum Shear Stress of PMMA Particulate Composites and Nano-Hybrid Laminated Composites as Function of (Nano-SiO₂) Particles (Vol %).

It can also be noticed that when adding random woven fiber glass in PMMA composite to form hybrid laminated composite material, the Maximum shear stress increased as compared with PMMA particulate composite. This could be attributed to the fact that glass fibers and (nano-silica) particles are characterized by higher resistance toward the Maximum shear stress than PMMA matrix and contribution of each (random woven fiber glass and nano-silica particles) to be carried out of the load applied on the hybrid laminated composite specimens. All these reasons led to the improving of the shear stress values of the hybrid laminated composite specimens ^[14].

Thus, the Maximum shear stress values increased from (4.6 MPa), for PMMA specimen (alone) to reach the higher values about (12.37 MPa) for hybrid laminated composite materials (PMMA - 3% Glass fiber - 1.5% nano-SiO₂).

5.3 Impact Test

The impact strength values results that obtained from impact test that carried out on PMMA composite and hybrid composite specimens for prosthetic denture base materials are shown in the Figure (6), it shows the effect of adding two different types of reinforcing (random woven fiber glass and nano-silica particles) on the impact strength of PMMA matrix.

From this Figure it can be noticed how the impact strength values increased with the increase of the volume fraction of nano-silica particles in both of PMMA composite materials and hybrid laminated composite materials, because of good distribution of nano-particles and when the increase of the concentration of these fine particles lead to filled and diminished of any spaces and porosities which were inside the PMMA resin and random woven fiber glass. Furthermore, the nature of these particles is high hardness and strong the ability of resistance to impact loads compared with PMMA ^[15].

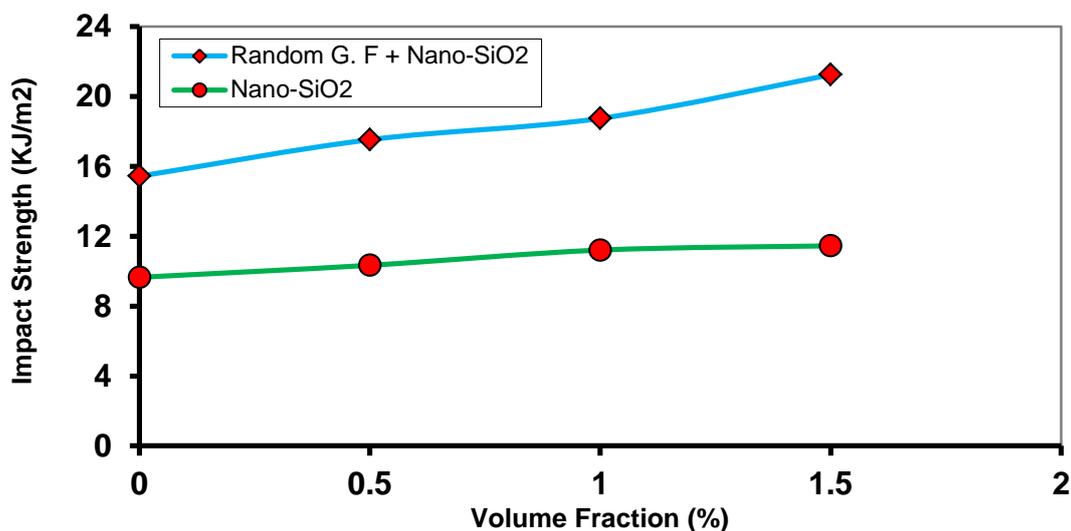


Figure (6): Impact Strength of PMMA Particulate Composites and Nano-Hybrid Laminated Composites as Function of (Nano-SiO₂) Particles (Vol %).

It can also be noticed that when adding random woven fiber glass in PMMA composite to form hybrid laminated composite material, the impact strength increased as compared with PMMA particulate composite. This could be attributed to the fact that glass fibers are characterized with high toughness and high impact strength than PMMA matrix and share with nano-silica particles to resist the impact loads that were applied on PMMA composite, therefore it led to improving the impact strength of the hybrid laminated composite specimens ^[15]. Thus, the impact strength value increased from (9.65 KJ/m²) for PMMA specimen (as referenced) to reach to the higher value of impact strength (21.25 KJ/m²) for hybrid laminated composite materials (PMMA - 3% Glass fiber - 1.5% nano-SiO₂).

6. Conclusions

According to the experimental results of composite prosthetic complete denture base materials, which were prepared in this research, it can be concluded that:

- 1- All mechanical properties such as (flexural modulus, flexural strength, maximum shear stress and impact strength) of PMMA composite and hybrid laminated composite for prosthetic denture base materials, were increased with the increase of the volume fractions of (nano-SiO₂) particles.
- 2- The flexural strain property of PMMA composite and hybrid laminated composite for prosthetic denture base materials, was decreased with the increase of the volume fraction of (nano-SiO₂) particles.
- 3- The addition of (random woven fiber glass and nano-SiO₂ particles) has a noticeable effect on all properties of composite prosthetic complete denture base specimens more than using (nano-SiO₂) particles only.
- 4- The maximum values for properties (flexural modulus, flexural strength, maximum shear stress and impact strength) were obtained in the hybrid laminated composite materials (PMMA - 3% Glass fiber - 1.5% nano-SiO₂).
- 5- The minimum values for flexural strain were obtained in the hybrid laminated composite materials (PMMA - 3% Glass fiber - 1.5% nano-SiO₂).

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