# The Fracture Toughness of Heat Cured Acrylic- Natural Rubber/Silicone Rubber Blend Reinforced with Pomegranate Peels Powder

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## Abstract

The development of PMMA resin in denture base fabrication by using the strengthen method represent important factor in the last years, so this study aims to development the mechanical strengthens of PMMA denture base by manufacturing samples based on two types of polymers blends are (poly methyl methacrylate (PMMA) resin: 2% (silicone rubber (SR)) and (PMMA resin: 2% natural rubber (NR)), each one of them used as a matrix material, reinforced by natural nanoparticles of pomegranate peel (PPP), with selected weight fractions ratios (0.0, 0.1, 0.3, 0.5 & 0.7% wt.). Some mechanical properties and analytical of (FTIR) were investigated. The results showed an appreciably improvement in the values of flexural strength, max. Shear stress, impact strength and fracture toughness for both groups of hybrids nanocomposites specimens comparing with matrix materials of (PMMA: 2%SR) and (PMMA: 2%NR). Hybrid nanocomposites specimens that based on polymer blend (PMMA: 2%NR) as matrix material reinforced with PPP nanoparticle , showed the highest values in these properties (144MPa, 12MPa, 10KJ/m² and 5.74MPa.m½) respectively, as compared with specimens of hybrid nanocomposites (PMMA: 2%SR): X% PPP) . On the basis of these results, it can be conclusion that the addition of 2% natural rubber with pomegranate peels powder nanoparticles to the poly methyl methacrylate material is one of the hopeful materials in use to improve the fracture strength for the complete or partial dentures base.

**Key words:** Nano-Composites, PMMA, SR, Natural powder, Mechanical properties.

## Introduction

Poly methyl methacrylate (PMMA) is the most widely used materials in contemporary prosthodontics. Its popularity are good color low water sorption, stability, adequate strength and low solubility strength. However, the mechanical strength of acrylic resin is not enough to maintain the longevity of dentures (Uzun et al, 1999). The basic problem of PMMA are its poor strength characteristics, low fatigue resistance impact strength [1]. Thus, there is a require to advance the performance of PMMA in denture base application. Several methods used to adjust the properties of denture base materials. Between these methods is strengthening the acrylic resin by modifying with fiber and used graft co-polymerization with high-impact resins [2, 3]. There were many attempts to strengthen polymers using different procedures. The introduction of nano dentistry in the last years , led to great advantages in reinforcing the mechanical properties of denture base materials [4] Also, clinical application of denture base not only requires excellent mechanical performance, but also expects good biocompatibility and biosafety, while some inorganic materials may cause irritation or even damage to gingival tissue and mouth mucosa [5]. There were many attempts to strengthen polymers using different procedures. The influence of glass fiber as a strengthening material, on fracture resistance and flexural strength of acrylic denture base resin was investigated, the result was giving an indication about the possibility for beneficent the flexural strength of heat cured PMMA [6]. It was reported that hydroxyapatite Nano powder had been used in dental material as an effective biocompatible filler to reinforce self-cured polymer matrix [7]. By the addition of HA, the flexural modulus of PMMA was increased, in addition, that the fracture toughness, young modulus and glass transition temperature of PMMA were affected by the addition of HA particles [8].The influence of the particles size and content ratio of silica (SiO2) ceramic particles on the mechanical characteristics of PMMA polymer, the results showed that the, bending modulus, tensile strength and modulus elasticity of PMMA composites are increased with increasing the addition of SiO2.

Also, the values showed that the impact energy and fracture toughness of PMMA composites decrease with rising the content ratio of SiO2 particles. Reinforcement of acrylic denture base with zirconia significantly increases its transverse strength of denture base resin [9, 10]. Additions of Nano zirconia to PMMA denture base have been reported to increase the transverse strength due to its small size and homogenous distribution [11]. Nanotechnology invaded the prosthodontics field for medical and material enhancement purposes. The properties of the reinforced resin by nanoparticles depend on the size, shape, type, and concentration of the added particles [12]. An improved in PMMA acrylic resin characteristics by adding four types of nanoparticles, which were zirconia, fly dust, fly ash and aluminum as a reinforcing material to self-polymerized PMMA resin, the results exhibited that the values of the flexural strength, flexural modules, hardness and maximum shear stress improved by adding these Nano powders [13]. Investigated the properties of PMMA when reinforcing with siwak fiber which cut into three lengths and used various concentrations, the results indicated that the impact properties improved with the increasing the fiber length [14]. Investigation the effect of addition TiO2 Nano particles on impact strength, thermal conductivity and color stability of acrylic resin cured by microwave was done and the result of this study showed that the curing of high-impact acrylic by microwave had not changed the color stability and thermal conductivity in comparison to the water bath, but it decreased the impact strength [15]. The mechanical properties of PMMA composites reinforced with different types of natural powders (pomegranate peels and seed powder of dates Ajwa) on the flexural properties, impact strength and fracture toughness values was study, the result showed a considerably improvement in these properties for both groups of bio composites, moreover, all bio composite specimens reinforced with pomegranate peels Nano powder showed the highest properties [16]. Another study show that, the flexural and impact properties for PMMA composites reinforced with bamboo powder and rice husk in individually form, was increased with increasing the fillers content of Rice Husk and Bamboo powder in composite materials [17].

### The aim of this study

An attempt to improve the fracture toughness for heat cured acrylic (PMMA) which used for a denture base application. By blending the PMMA with natural rubber (NR) or silicone rubber (SR) in individually form, and prepare hybrid polymer blend composites reinforced by the natural pomegranate peels powder in nanometer size.

## 2. Materials and Methods

### 2.1 Materials used

In this research, the composite prosthetic complete dentures specimens consist of a polymer matrix material which are PMMA, natural rubber (NR) or silicone rubber (SR) and reinforcement material as natural powders. Matrix materials included of two groups of polymeric blends (Heat curing PMMA: 2%NR) or (Heat curing PMMA: 2%SR). PMMA material used as fluid resin matrix, type (Spofa Dental Company, Czech Republic). A blending material are the natural rubber (NR) and silicone rubber (G-815, Dongguan guochuang Silicone Co.LTD, Chine). A reinforcement material as natural nanoparticles (pomegranate peels powder (PPP)), with average diameter of 53.38 nm.

### 2.2 Preparation specimens

The PMMA denture base materials consist of polymer powder and monomer liquid (methyl methacrylate, MMA). The standard proportion in mixing ratio for a heat curing acrylic resin is usually volumetric ratio about (3) polymer powder (PMMA) and (1) monomer liquid (MMA) according to company instructions. In this work to prepared a polymer blends, as well as, the polymer blend Nano composites samples. Initially it was mixed the liquid (MMA) part of acrylic resin with 2% NR solution or with 2%wt of SR, until the mixture was becoming perfectly homogeneous, after that, a powder of PMMA it was added to this mixture, with continuous mixing process, then the mixture was poured into metallic mould prepared for this purpose. The mould pressed by using a hydraulic compressor with pressure of about 2.5 bars to obtain a smooth surface and to prevent gases vapor to entry into PMMA during the curing. The curing process for acrylic was performed at conditions of (70 ºC, 2.5 bar and 30 min) according to company instructions. And then raise temperature to the (100 ºC) and stay at this temperature for one hour. Then the process of cooling the mould begins inside the curing device in order to remove the residual monomer. The composites samples were removed them from the metallic mould, with very smooth of surfaces. Then, final heat treatment at 55°C for 3 hr was done to remove residual stresses as a result of de-molding of the specimens from the metallic mold cavity.

## 3. Mechanical and Physical Tests

### 3.1 Fourier transform infrared spectra (FTIR)

Fourier transformation Infrared (FTIR) spectrum is used to obtain specific information about the chemical bonds and molecular structure of polymer samples. The (FTIR) spectrum test is performed according to (ASTM E1252) [18]. By using Fourier transform infrared spectrometer, model (TENSOR 27) made in Germany, by (Bruker Optics Company). Infrared spectrum was used within range of (400-4000) cm-1.

### 3.2 Mechanical tests

Flexural strength measured from three-point test, instrument, (model WDW 200 E) made in China, this test is preformed according to ASTM D 790-78 [19] at room temperature with velocity (5mm/min) until the failure of the specimen occurred. Impact strength test was conducted by the ISO 179 [20] with Charpy type impact testing instrument (N 43-1, New York, USA). Ten specimens were constructed for control and experimental groups, is performed at room temperature.

### 3.3 Atomic force microscope AFM

This test was used to determine the average diameter of nanoparticle and its distribution shown in Fig.1.

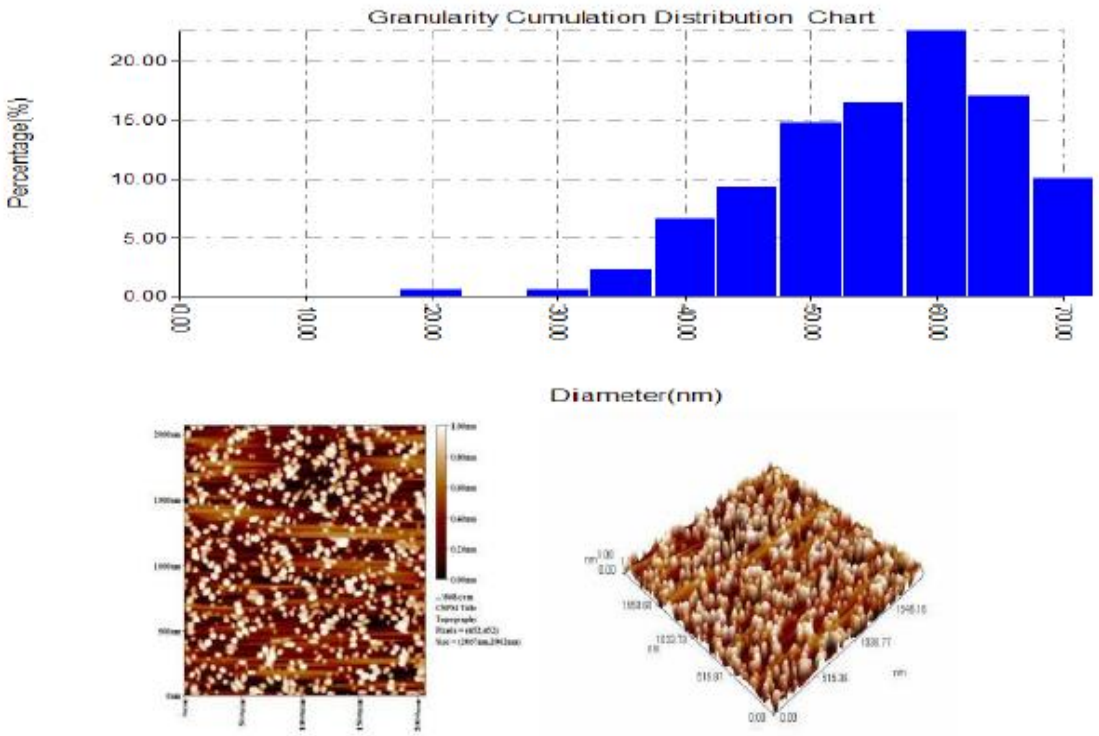


**Figure 1. AFM test of pomegranate peels nanoparticles (Average diameter 53.38 nm).**

## 4. Results and Discussions

### 4.1 Fourier Transform Infrared Spectrometers (FTIR) Test Result

This test is used for fully characterization of PMMA the heat curing, binary polymer blends (PMMA: 2NR) and (PMMA: 2SR) and Nano-composites specimens as a function of addition nature powders of (pomegranate peels powder (PPP)) with base of binary polymer blend. The FTIR spectrum in the frequency range (400-4000 cm-1) was used in this study. The infrared spectrum for neat PMMA shown in Fig.2. Is quite similar to that reported in literature [21 and 22]. The absorption peaks around (2991.51 cm-1 and 2950.40 cm-1) correspond to C-H asymmetric stretching in CH3 and CH2, respectively. The vibrational band at (2849.97 cm-1) is due to the C-H symmetric stretching in CH3. The characteristic band for the neat PMMA is observed at 1722.54 cm-1, which corresponds to

C=O stretching band. The vibrations mode due to deformation modes of CH3 groups appear at 1434.50 cm-1and at 1386.33 cm-1. Medium bands at 1239.49 cm-1correspond to C-O stretching modes. The band at 1189.65cm-1 corresponds to CH3 wagging, and two bands at 1142.75 cm-1 are due to the CH3 twisting. The vibration modes due to C-C stretching appear at (985.98 cm-1 and 964.96 cm-1). The peaks at (911.30 cm-1 and 840.40 cm-1) are assigned to CH2 rocking, and the peaks at (808.09 cm-1 and 749.44 cm-1) are due to the CH2 rocking in plane and out of plane bending, respectively. These results are in a good agreement with other workers results [22 and 23].

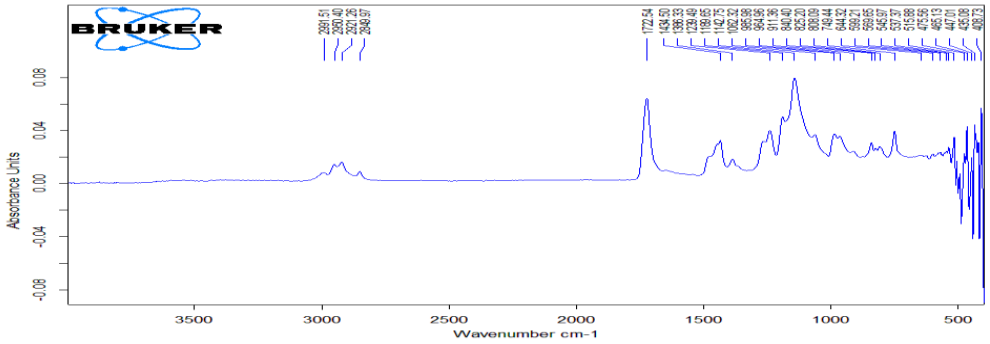


**Figure 2. The Fourier Transform Infrared Spectrum for Neat PMMA (heat curing),**

Fig.3 and (4) show the FTIR spectra for two groups of polymeric blends Nano composites, which are (PMMA: 2%SR): X%PPP) and (PMMA: 2%NR): X%PPP) as a function of PPP content (0.0, 0.1, 0.3, 0.5 and 0.7%). It can be seen from Fig.3 that all the characteristics vibration bands of neat PMMA (Fig.2) were presented in FTIR spectrum of polymeric blend specimen (PMMA: 2% SR). The characteristic band for the PMMA in the blend is observed at 1722.31 cm-1, which corresponds to C=O stretching band. The vibrations mode due to deformation modes of CH3 groups appear at (1474.23 and at 1434.78 cm-1 and at 1386.86 cm-1). Medium bands at 1240.40 cm-1 correspond to C-O stretching modes. The band at 1189.89 cm-1 corresponds to CH3 wagging, and two bands at 1143.29 cm-1 are due to the CH3 twisting. Moreover, it can be seen from the infrared spectra of the first group of polymeric blend composites specimens (fig.3); these spectra are quite similar to the FTIR spectrum of neat PMMA (fig.2) and polymer blend (PMMA: 2%SR), no other new peak or peaks shifts were observed for the polymeric blend composites of (PMMA: 2%SR): X%PPP) specimens with the addition of PPP nanoparticle.

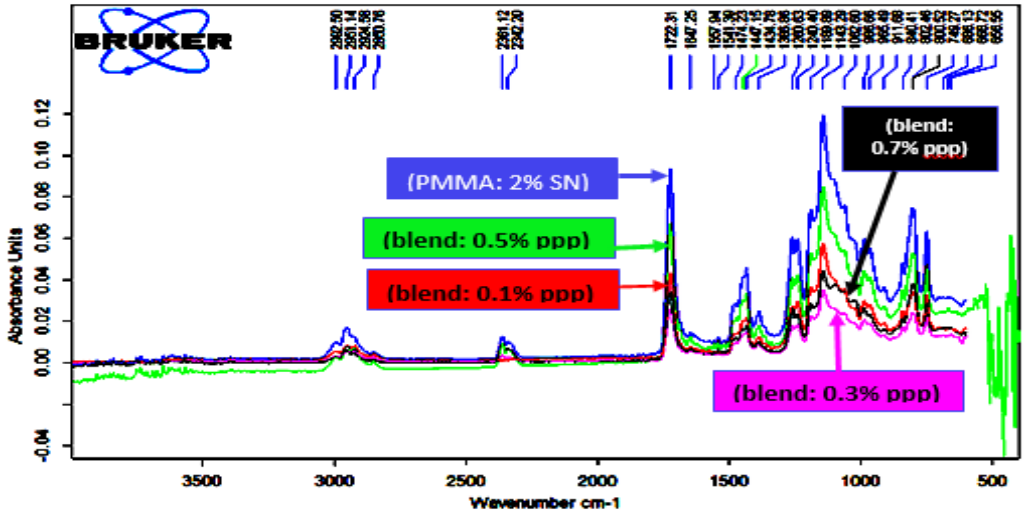


**Figure 3. FTIR spectra for polymer blend (PMMA: 2%SR) and polymer blend composites ((PMMA: 2%SR): X%PPP) as a function of pomegranate peels powder content in composite.**

As well as, from the infrared spectrum of the second group of polymeric blend composites specimens (fig.4); these spectra are quite similar to the FTIR spectrum of neat PMMA (fig.2) and polymer blend (PMMA: 2%SR), The characteristic band for the PMMA in second blend is observed at 1721.54 cm-1, which corresponds to C=O stretching band. The vibrations mode due to deformation modes of CH3 groups appear at (1434.77 cm-1 and at 1387.04 cm-1). Medium bands at 1239.93 cm-1 correspond to C-O stretching modes. The band at 1189.06 cm-1 corresponds to CH3 wagging, and two bands at 1140.69 cm-1 are due to the CH3 twisting. no other new peak or peaks shifts were observed for the polymeric blend composites of (PMMA: 2%NR): X%PPP) specimens with the addition of PPP nanoparticle. These results due to the find physical bond and absence of any cross-linking and chemical reaction between constituents of polymeric blends, as well as a there is no any new interaction in these specimens of polymeric blend composite.

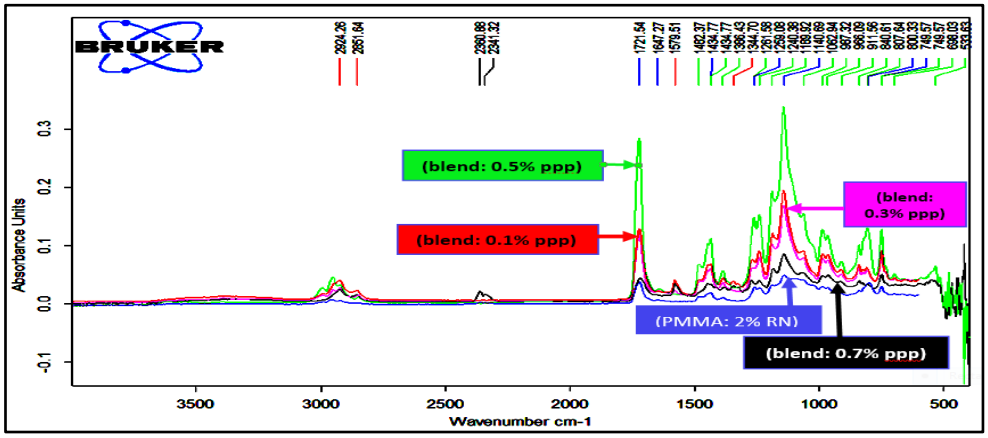


**Figure 4. FTIR spectra for polymer blend (PMMA: 2%NR) and polymer blend composites ((PMMA: 2%NR): X%PPP) as a function of Pomegranate peels powder content in composite.**

## 4.2 Results and Discussion of Flexural Test

The effect of the addition natural filler (pomegranate peels powder (PPP)) as a reinforcement particle to binary polymer blend (PMMA: 2NR) and (PMMA: 2SR) on the flexural strength, flexural modulus and maximum shear stress of PMMA hybrid nanocomposites, is shown in Fig (5 ,6 and 7) respectively. From these figures it was observed, there is a gradual increase of flexural strength, flexural modulus and maximum shear stress with a further increase of the weight fraction of PPP content for both types of PMMA hybrid Nano-composites; this is due to good compatibility between the constituents of matrix materials and PPP natural reinforcing fillers [27]. Moreover, that the maximum value of flexural strength was reached at (0.5%) ratio of the weight content of PPP in composite of (PMMA: 2% NR), and the maximum value of Max.shear stress was reached at ratio (0.5%) of the weight content of PPP in same composite, and the higher value of flexural modulus was reached at (0.7%) ratio of the weight content of PPP in hybrid nanocomposites with a bases material of (PMMA: 2NR) in same composite. This result related to the ability of PPP natural particles to prevent the propagation of cracks inside matrix according to reinforcing mechanism in addition to the strong bonding between the constituents of matrix materials and PPP natural particles. The presence of the compatible between natural reinforcing particle of PPP and the constituents of matrix material (PMMA: 2% NR or SR) had a very considerable effect on a decrease in the molecular motion of polymer chains and reduces free volume in the prepared composites samples, which that increasing from the flexural strength, flexural modulus and Max.shear stress properties for hybrid prepared samples [28]. The highest value of flexural strength, flexural modulus and max. Shear stress were (110MPa), (3.19GPa) and (11.86MPa) respectively, for hybrid composite specimens (PMMA: 2%SR: x% PPP), whereas these values for hybrid composite specimens (PMMA: 2%NR: x% PPP) reached to ((144MPa), (3.3GPa) and (12MPa) respectively.

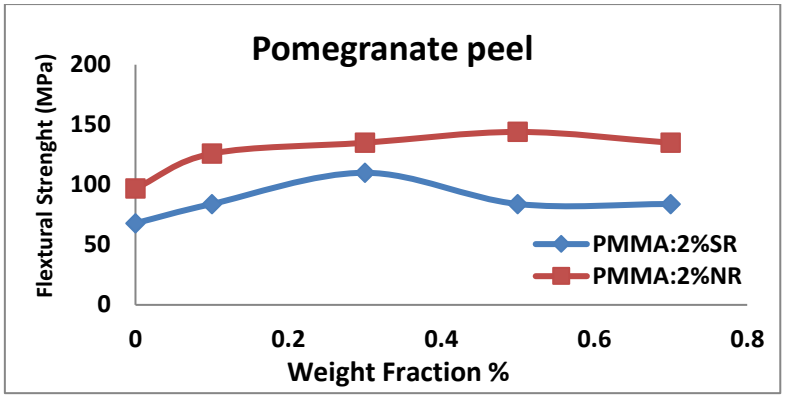


**Figure 5. Flexural strength for PMMA hybrid composite specimens as a function of weight fraction content for PPP in composites**

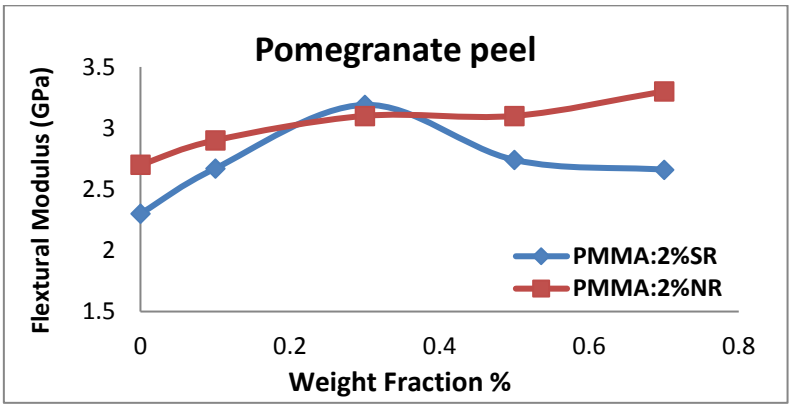


**Figure 6. Flexural modulus for PMMA hybrid bio composite specimens as a function of weight fraction content for PPP in composites**

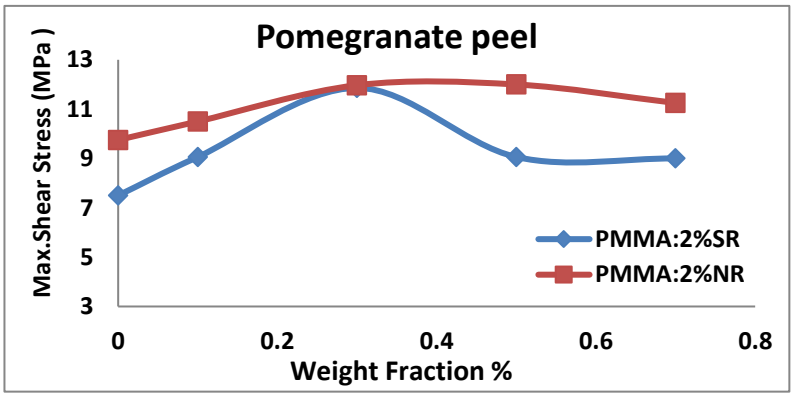


**Figure 7. Max. Shear stress for PMMA hybrid bio composite specimens as a function of weight fraction content for PPP in composites.**

### 4.3 Results and Discussion of Impact Strength and fracture toughness

The impact test is various from other mechanical tests because it is so fast. Where the sample is subjected to the fast stress leading to changes in the behavior of composite material. The impact strength value is the most important property because it is giving an indication about the measure of a given material's toughness. Impact strength is the absorb energy by composite materials through a cross vassal area for composite samples. Impact strength of the composite specimen is controlled by two elements: first, the capability of the reinforcing material to stop crack propagation by absorbing energy and the second one, poor bonding between reinforcing fiber and matrix, which cause micro-spaces and result in crack propagation [29]. The effect of the addition natural filler (pomegranate peels

powder (PPP)) as a reinforcement particle to binary polymer blend (PMMA: 2NR) and (PMMA: 2SR) on the impact strength and fracture toughness of PMMA hybrid nanocomposites, is shown in Fig (8 and 9) respectively. From these figures it was noted, the impact strength and fracture toughness increased with a further increase of the weight fraction of PPP content for both types of PMMA hybrid nanocomposites. That the maximum values of the impact strength and was reached at (0.7%) ratio of the weight content of PPP in composite of (PMMA: 2% NR), and the maximum value of fracture toughness was reached at ratio (0.7%) of the weight content of PPP in same composite. The reason behind this behavior may be depend on that the impact test is a measure of a given material's toughness. So, the obtained results may be concerned with typical distribution of natural particles of (PPP) within binary polymer blend (PMMA: 2NR) and (PMMA: 2SR) and interfacial bonding between them leads to considerable increase in the energy absorbing capacity of the bio composite specimens [30,31]. The highest value of impact strength, fracture toughness was (7.21 KJ/m²), (4.72 MPa.m1/2) respectively, for hybrid composite specimens (PMMA: 2%SR: x% PPP), whereas these values for hybrid composite specimens (PMMA: 2%NR: x% PPP) reached to (10 KJ/m²), (5.74 MPa.m1/2) respectively.

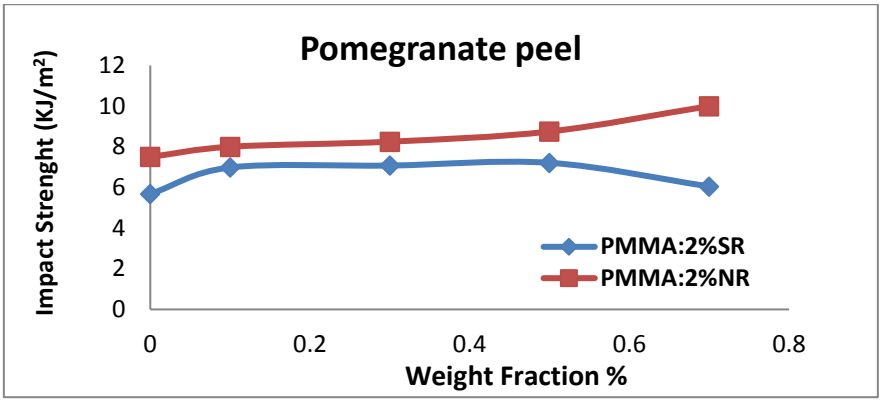


**Figure 8. Impact strength for PMMA hybrid bio composite specimens as a function of weight fraction content for PPP in composites.**

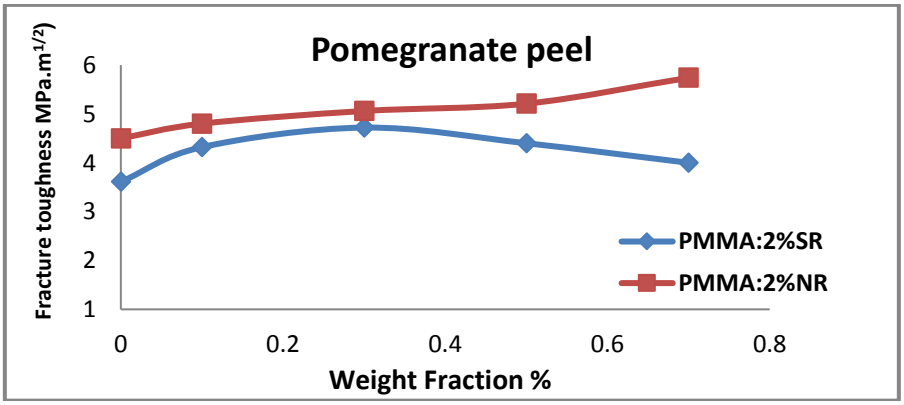


**Figure 9. Fracture toughness for PMMA hybrid bio composite specimens as a function of weight fraction content for PPP in composites.**

## 5. Conclusions

From discussion results of the prepared nanocomposites based on polymer blend reinforced with natural powder, it was concluded the following: -

1. The polymer blend (PMMA: 2%NR) get higher values in the mechanical properties, as compared the polymer blend (PMMA: 2%SR).
2. Mechanical properties for both types of polymer blends improved with adding natural powder of pomegranate peels to them.

3. The maximum value of flexural strength and max.shear stress were reached at ratio of 0.5% OF PPP content in composite based on (PMMA: 2% NR), and the maximum values of impact strength, fracture toughness and flexural modulus were reached at ratio of (0.7%) of PPP content in nanocomposites based on (PMMA: 2NR) also.
4. The highest value of flexural strength, flexural modulus, max. shear stress, impact strength and fracture toughness for bio composite specimens based on (PMMA: 2NR) are 144MPa, 3.3GPa, 12MPa, 10KJ/m² and 5.74MPam½ respectively.
5. Based on the what mentioned earlier of conclusions, it can be conclusion that the addition of 2% natural rubber and natural fillings nanoparticles (pomegranate peels powder) to the poly methyl methacrylate material is one of the hopeful materials in use to improve the fracture strength for the complete or partial dentures base.

**CONFLICT OF INTERESTS.**

- There are no conflicts of interest.

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**الخلاصة**

يمثل تطوير راتنج PMMA في تصنيع قاعدة الأسنان باستخدام طريقة التقوية عاملًا مهمًا في السنوات الأخيرة، لذلك يتعامل هذا العمل مع تطوير المقاومة الميكانيكية لقاعدة أسنان PMMA عن طريق تصنيع عينات تتكون من مزيج البوليمر كمادة اساس ومدعومة بجزيئات نانوية طبيعية من قشر الرمان (PPP). تتكون هذه المجموعات من نوعين من مزيج البوليمرات ( PMMA:2%مطاط السيليكون SR أو المطاط الطبيعي NR)، كل واحدة منها تستخدم كمواد اساس ، تم تعزيزها بواسطة مسحوق طبيعي من قشور الرمان (PPP) بحجم النانومتر ،بنسب وزنية محددة (0.0 ، 0.1 ، 0.3 ، 0.5 و 0.7٪ وزناً). تم التحقق من بعض الخواص الميكانيكية (مقاومة الانحناء، أقصى إجهاد القص، مقاومة الصدمة، متانة الكسر والصلابة) ومن معطيات الـــ (FTIR، وأظهرت النتائج تحسناً ملحوظًا في جميع الخواص الميكانيكية التي درست في هذا العمل لكلتا المجموعتين من المتراكبات مقارنة بمواد الاساس (PMMA: 2% SR) أو (PMMA: 2% NR)

أظهرت المتراكبات الحياتية المكونة من (PMMA: 2%NR): X% PPP) أعلى القيم لهذه الخصائص وهي 144MPa, $12MPa, 10KJ/m^2$ and $5.74MPa.m^{½}$على التوالي، بالمقارنة مع العينات المتراكبة المكونة من (PMMA: 2%SR): X% PPP).

بناءً على هذه النتائج، يمكن أن نلخص إلى أن إضافة 2٪ من المطاط الطبيعي مع حبيبات القشور النانوية إلى مادة بولي ميثيل ميثاكريليت هي واحدة من الاساليب الناجعة لتحسين متانة الكسر لقواعد أطقم الأسنان.

**الكلمات الدالة:** المتراكبات النانوية، بولي ميثيل ميثاكريليت، مطاط السليكون، مسحوق طبيعي، الخواص الميكانيكية.