International Journal of Nanoelectronics and Materials

In Press, Accepted Manuscript – Note to user

Development on mechanical properties of PMMA by blending it with natural rubber or silicone rubber and reinforced by Nanoparticle.

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Received 2 Jul 2019; Revised 30 Oct 2019; Accepted 24 Dec 2019

ABSTRACT

In this study, an attempt to develop the mechanical properties of PMMA that are used in dentures material, by using polymers blends of (PMMA: 2%NR) and (PMMA: 2%SR) as a matrix reinforced with natural nanoparticles of clove powder (CP) that were added with different weight fractions of (0.0, 0.1%, 0.3%, 0.5% and 0.7%). Two groups of composites samples were prepared according to the type of polymer blends matrix, by using hand lay-up method. The maximum values of tensile strength and modulus of elasticity where noted at 65 MPa and 1.033 GPa respectively for polymer blend nanocomposite [(PMMA: 2%NR): 0.5% CP], whereas, the Shore D hardness reach to a maximum value of (89) for polymer blend nanocomposite (PMMA: 2%NR): 0.7% CP). On the basis of these results, it can be concluded that the addition of clove powders in a nanometer size to polymer blends materials (PMMA: 2% NR), is one of the hopeful materials that can be utilized to develop the mechanical properties for dentures base applications.

Keywords: Nanocomposites, PMMA, NR, SR, Clove powder

1. INTRODUCTION

This work will concentrate on denture base without artificial teeth. Poly methyl methacrylate (PMMA) is the primary material used in dentistry for denture base constructions. There are various other polymeric materials, which have been utilized for denture base [1]. Acrylic resins came into utilization in dentistry between 1930 and 1940 and were employed as denture base [2]. Until now, PMMA stays the preferred material for removable prostheses, dental implants, and orthodontics instrument [3]. PMMA resin shows better mechanical, impact and physical properties compared with other polymer materials. Although PMMA material has poor mechanical and physical properties when used alone, where it is easily rupture during an accident, or when a patient applies a high mastication force on the denture base [1]. There are many trials to strengthen polymers using varied procedures, one of them tried to incorporate glass fiber to strengthen the fracture resistance and flexural strength of denture base resin. This study exhibited the possibility of improving the flexural strength of the heat curing polymerized PMMA after strengthening with glass fiber, and it may be possible to apply distal tension on partial and whole denture bases [4].

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Other study showed the influence of the reinforced denture base material with five aesthetic fibers (glass, rayon, polyester, nylon, and nylon 6.6) that were added individually. The results illustrated that the flexural strength, modulus of elasticity and max. load improved by nylon fibers [5].

The effect of adding siwak powder as a strengthening material on the mechanical properties of the heat cured PMMA acrylic resin. The results illustrated that the addition of (3% and 5%) of siwak powder to the acrylic resin had little influence on the tensile, impact and compressive properties according to the reference group [6]. The mechanical and physical properties of denture-based material fabricated by heat polymerized PMMA acrylic resin with incorporation of nanofillers of surface treated aluminum oxide as a strengthening material were investigated. The results showed that the transverse strength was developed by the addition of (1wt% and 2wt %) of Nano particles, while a considerable reduction took place in transverse strength at 3% ratio of nanoparticles [7]. The PMMA acrylic resin features were promoted by adding different kinds of nanoparticles, which were zirconia, fly dust, fly ash and aluminum as strengthening materials to self-polymerized (PMMA) resin, the results depicted that the values of the flexural strength, flexural modules, hardness and maximum shear stress were improved by adding these nanoparticles [8].

Some mechanical characterizations of PMMA resin strengthened with siwak fibers as a natural material were examined. The results revealed on improvement in hardness, Young's modulus, tensile strength, fracture toughness with the increasing of length and content ratios of siwak fiber, whereas the impact strength decreased with the increasing of fiber concentration in composite samples [9]. The effects of some co-monomers on the impact strength and flexural strength of acrylic resin, with glass fiber having 6 mm length at 3% weight ratio were studied. The results indicated that the highest flexural and impact strength were gained by the addition of 2% IBMA monomer strengthened with glass fiber [10]. The influences of adding zirconium oxide nanoparticles and glass fibers on the flexural and impact strengths of a polymethylmethacrylate denture base were considered. The results manifested the added 2.5% nano-ZrO_2 + 2.5% GF to the PMMA advanced flexural strength and impact strength of PMMA nanocomposite materials [11]. The effect of adding different concentrations of nano-diamonds on the flexural strength, impact strength, and surface roughness of heat curing polymerized acrylic resin was studied. The study showed that the addition of nano-diamonds improved the flexural strength and surface roughness at low concentrations (0.5%), while the impact strength decreased [12].

The aim of this study is an attempt to develop some mechanical properties for dentures base by a comparative study between two groups of nanocomposites based on the polymers' blends (PMMA: 2%NR) and (PMMA: 2%SR), which were reinforced by the natural nano powders of clove material.

2. MATERIAL AND METHODS

2.1 Materials

In this study, the complete dentures base samples include polymeric materials, which are poly methyl methacrylate (PMMA), natural rubber (NR), silicone rubber (SR) and reinforcement materials as natural powders in nanometer size. The matrix of materials contains polymeric blends (PMMA (heat curing): 2%NR), (PMMA: 2%SR) as control sample. PMMA material was utilized as fluid resin matrix, type (Spofa Dental Company, Czech Republic). The blending materials are the natural rubber (NR) and silicone rubbers (SR). A strengthening material as natural powder of clove powder (CP) shown in Figure (1) was chosen with a concentration of (0.0, 0.1, 0.3, 0.5 and 0.7% wt.) and with average diameter 75.18 nm. The atomic force microscope (AFM) was utilized to check the average diameter of the nanoparticle and the nanoparticles distribution, as shown in Figure (2).

Figure 1. Reinforcement material: (Clove powder) having nanoparticles size (75.18 nm)

Table 1. Particle size analysis of clove powder by AFM test with average diameter (75.18 nm)

Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)	Diameter(nm)<	Volume(%)	Cumulation(%)
40.00	4.55	4.55	75.00	4.55	20.45	105.00	7.58	73.48
45.00	1.52	6.06	80.00	9.85	30.30	110.00	8.33	81.82
50.00	1.52	7.58	85.00	8.33	38.64	115.00	12.12	93.94
55.00	2.27	9.85	90.00	6.06	44.70	120.00	6.06	100.00
60.00	1.52	11.36	95.00	10.61	55.30			
65.00	4.55	15.91	100.00	10.61	65.91			

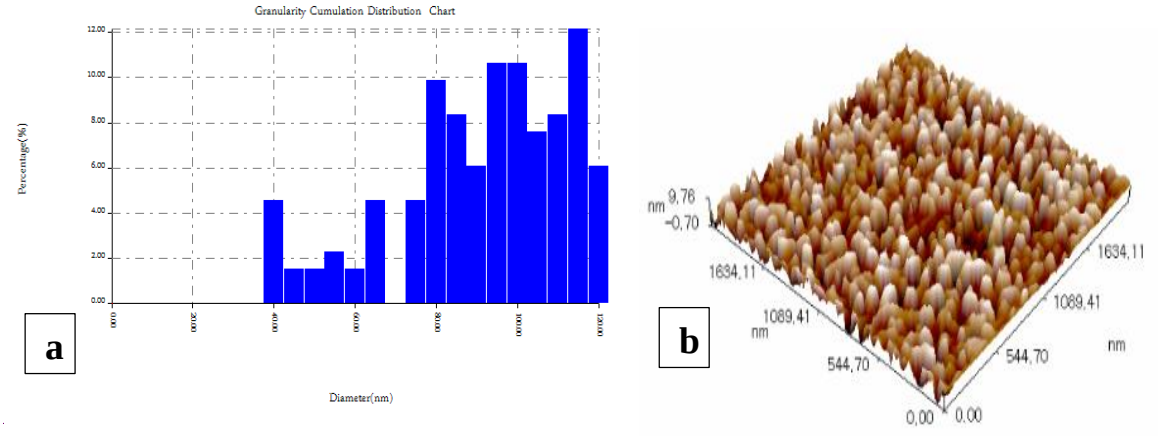


Figure 2. AFM test of clove powder nanoparticles with average diameter 75.18 nm, where a: Granularity accumulation distribution chart of clove powder and b: Three-dimensional (XYZ) AFM pictures for clove powder

2.2 Preparation of Specimens

In the present study, the bio nanocomposite materials consist of PMMA base material which include two parts, polymer powder and monomer liquid (methyl methacrylate, MMA). The standard percentage in mixing ratio for a heat curing acrylic resin is usually taken in the volumetric ratio about 3 parts of polymer powder (PMMA) and one part of monomer liquid (MMA) according to company instructions. In the current study to prepare a polymer blends samples and bio nanocomposites samples, the liquid (MMA) part of acrylic resin was initially mixed with the 2% ratio of NR or SR material, until the mixture was getting perfectly homogeneous. After that, a powder of PMMA and reinforced material (clove powder (CP)) were added to this mixture, with a continuous mixing process, and then the mixture was poured into metallic mould prepared for this purpose. The mould was pressed using a hydraulic compressor

with a pressure of about 2.5 bars to gain a smooth surface and to block the gases vapor to enter into PMMA during the curing. The curing process for acrylic was carried out under the conditions of 70 ºC and 2.5 bar for 30 min according to the company instructions. And then raise the temperature was raised to 100 ºC, and the acrylic was remained at this temperature for one hour. Then, cooling the mould started into the curing device to oust the residual monomer. The samples were extracted from the metallic mould, with very smooth surfaces. Then, final heat treatment at 55°C for 3 hrs. was done to remove the residual stresses found within samples.

3 Mechanical and Physical Test

3.1 Fourier transform infrared spectra (FTIR)

Fourier transformation Infrared (FTIR) spectrum was used to obtain specific information about the chemical bonds and molecular structure of polymer samples. The (FTIR) spectrum test was carried out according to (ASTM E1252) [13]. By using FTIR spectrometer, model (TENSOR 27) made in Germany, by (Bruker Optics Company), Infrared spectrum was used as within range of (400- 4000) cm^{-1}.

3.2 Tensile test

Tensile test specimen was prepared according to ASTM standard D638-87 [14]. The machine utilized for the testing is micro computer controlled electronic universal testing machine (model WDW 200 E) made in China. The test was conducted at a velocity of (5 mm/min) at ambient temperature, tensile stress was applied till the failure of the specimen and stress-strain curve was obtained.

3.3 Hardness test

Hardness test (Shore D) used in this test was done according to ASTM- D-2240 [15]. To estimate the hardness of the specimens, and the utilized ones should have plain surface, smooth with thickness at minimum more than (3 mm) and should not be subjected to mechanical vibrations, so that the sample made with dimensions (10×10×4 mm).

3.4 Compression Test

The compression test was carried out at room temperature by the universal tensile test machine produced by (Laryee Company in china), type (WDW-50), due to the (ASTM D695) [16]. The cross-head speed was (0.2 mm/min), and the load was applied step by step until the fracture of the samples took place. According to (ADA Specification No.12, 1999), all the test samples after preparation and polishing operations must be kept in distilled water at (37± 1℃) for 48 hr [17].

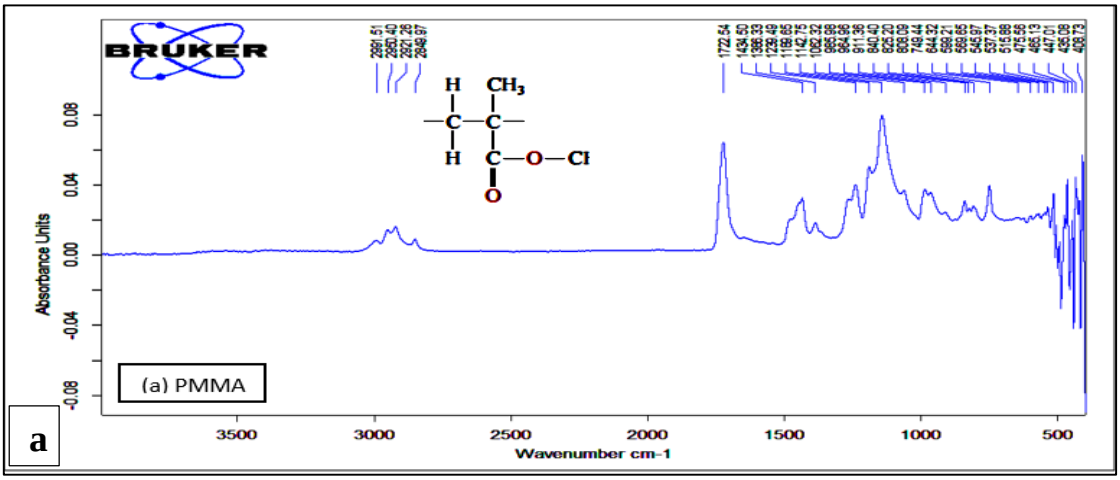
4 Results and Discussion

4.1 Fourier Transform Infrared Spectrometers (FTIR) test result

This test was utilized for the fully characterization of PMMA, the heat curing, binary polymer blends (PMMA: 2% natural rubber (NR)), (PMMA: 2% Silicone rubber (SR)) and nanocomposites specimens as a function of nature powders (Clove powder (CP)) which were added individually to a base of binary polymer blend. The frequency ranges used in this test are (400-4000 cm^{-1}).

The infrared spectrum for neat PMMA is shown in Figure 3 (a), it is quite similar to that reported in literature [18 and 19]. The absorption peaks are 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 according to the C-H symmetric stretching in CH3. The characteristic band for the neat PMMA was observed at (1722.54 cm^{-1}), which corresponds to C=O stretching band. The vibrations mode according to deformation modes of CH3 groups appeared at (1434.50 cm^{-1}) and at (1386.33 cm^{-1}). Medium bands at (1239.49 cm^{-1}) correspond 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 appeared 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 an excellent agreement with other workers results [19 and 20]. Figure 3 (b) shows the FTIR spectrum for binary polymer blends (PMMA: 2% NR), and all the vibration bands of polymeric blend specimen (PMMA: 2% NR) are matching with that appeared in the FTIR spectrum of neat PMMA in Figure 3 (a).

The band for the PMMA was noted at (1721.54 cm^{-1}), which match with C=O stretching band. The vibrations mode according to deformation modes of CH3 groups seem 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}) matches to CH3 wagging, and two bands at (1140.69 cm^{-1}) are due to the CH3 twisting. As well as, the FTIR test for (PMMA: 2% SR) in Figure 3(c) revealed results very similar to that appeared into neat PMMA and (PMMA: 2% NR). Where, the vibration bands of C=O stretching band appeared at (1722.31 cm-1) and the vibration bands for CH3 groups at (1434.78 cm^{-1} and at 1386.86 cm^{-1}), the peak at (1240.40 cm^{-1}) corresponds to C-O stretching modes, the peak at (1189.89 cm^{-1}) matches to CH3 wagging and two peaks at (1143.29 cm^{-1}) are due to the CH3 twisting bands.



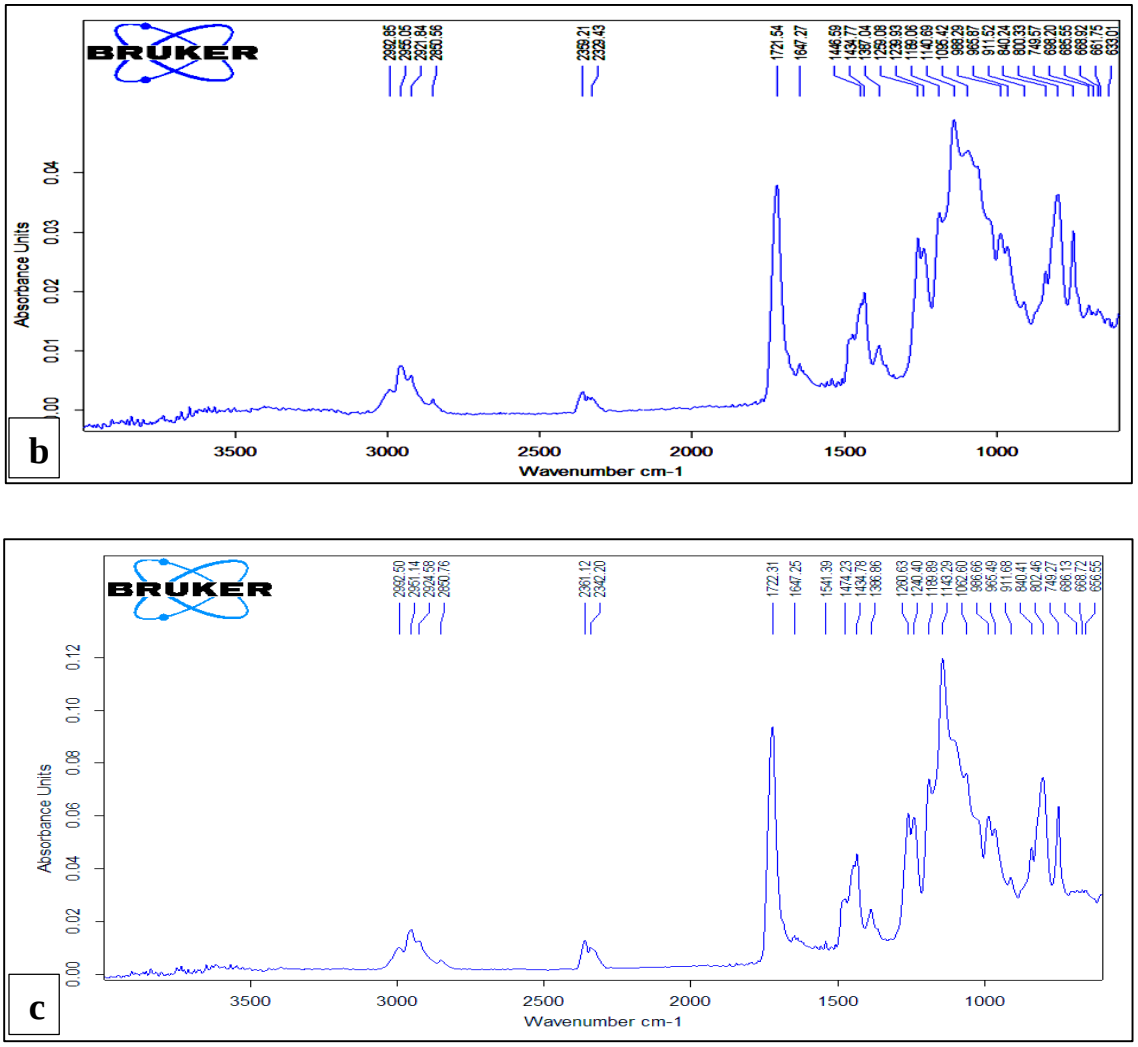


Figure 3. The Infrared Spectrum for a: Neat PMMA (heat curing), b: Polymer blend (PMMA: 2% NR) and c: Polymer blend (PMMA: 2% SR).

Figure 4 evinces the FTIR spectrum of Clove powder (CP). From this spectrum, it was observed that the peak at (3442.94 cm^{-1}) for O-H stretching band which reinforced the existence of alcohols compounds and carboxylic acids. The stretching band of C═C alkyne group was noticed at bandwidth (2951.09 cm^{-1}). The sharp peak at (1732.08 cm^{-1}) characteristic to carbonyl group C=O which leads to presence of aldehydes, ketones and carboxylic acids, and the sharp peak at 1616.35 cm^{-1} shows the existence of unsaturated compounds (alkenes). The band at 1516.05 cm^{-1} for CH2 bending verifies the presence of cellulose.

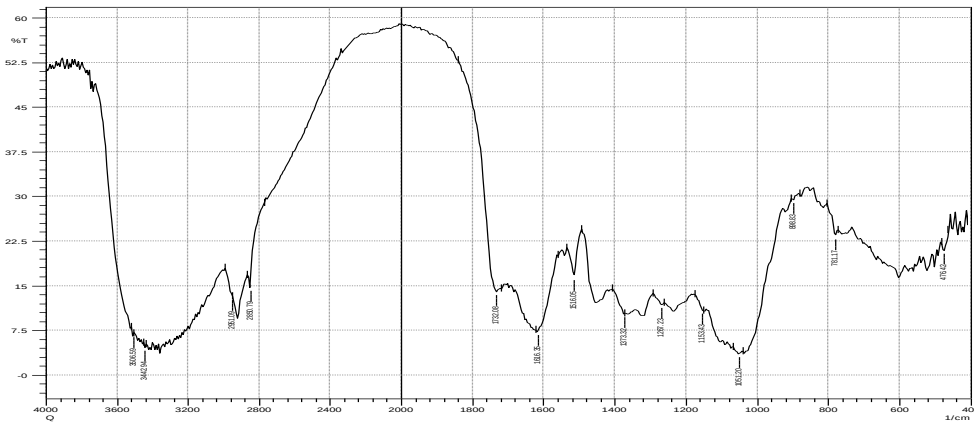


Figure 4. The FTIR spectrum for clove powder used.

Figures (5) and (6) elucidate that the FTIR spectra for two groups of bio nanocomposites are (PMMA:2%NR): X%CP) and (PMMA:2%SR): X%CP) as a function of CP content (0.0, 0.1, 0.3, 0.5 and 0.7%) in composite, these spectra are quite similar to the FTIR spectra of neat PMMA in figure 3 (a) and polymer blend (PMMA: 2%NR) in figure 3 (b). As well, FTIR spectra for the composite's specimens (PMMA: 2%SR): X%CP) are similar to that appeared in the polymer blend (PMMA: 2%SR) in Figure 3 (b). Moreover, from these spectra, no any other new peaks were appeared, or any aberration in the positions of peaks was noted for all samples of nanocomposites having the bases of polymer blends. These results are according to the physical bond, and no any cross-linking and chemical reaction are between the components of nanocomposites, and also no any chemical interaction in these samples of nanocomposites based on the polymer blends.

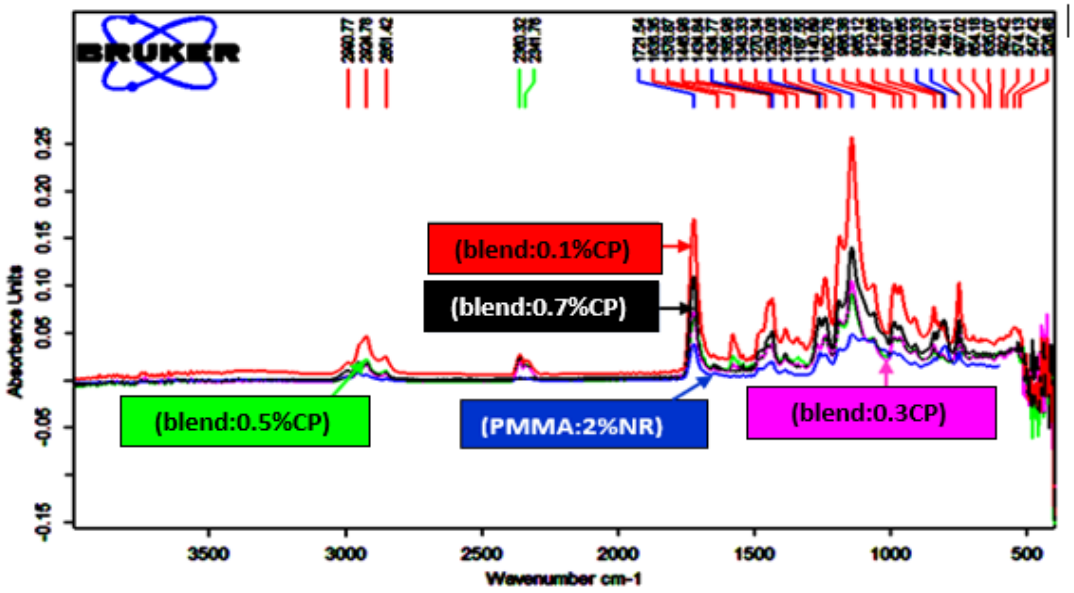


Figure 5. FTIR spectra for polymer blend (PMMA: 2%NR) and polymer blend composites ((PMMA: 2%NR): X% CP) as a function of Nano Clove powder content in composite.

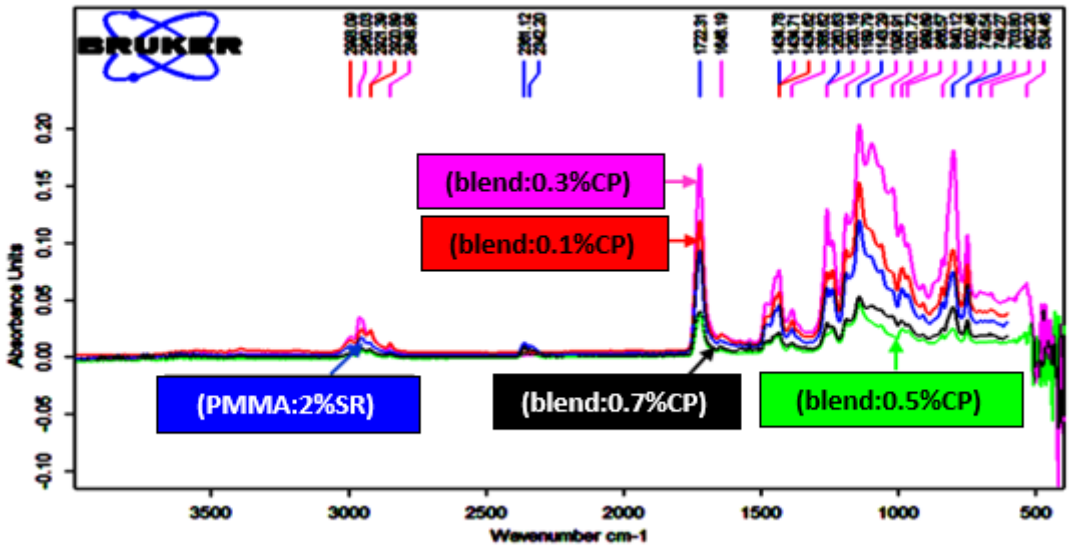


Figure 6. FTIR spectra for polymer blend (PMMA: 2%SR) and polymer blend composites ((PMMA: 2%SR): X% CP) as a function of Nano Clove powder content in composite.

4.2 Tensile Test Result

The influence of adding the natural particles of clove powder (CP) as strengthening particles to polymer blend (PMMA: 2% NR) and (PMMA: 2%SR) as a matrix on the stress-strain curve, tensile strength, elastic modules and elongation for the nanocomposite's samples is shown in figures (7, 8, 9, 10 and 11), respectively. From the figures (7) and (8), it was seen that the behavior of (stress-strain) curves changed with the percentage ratios of clove particles content in composite. (stress-strain) curve was linear, where the specimen behaved in an elastic region and finally developed into non-linear due to the deformation of samples in plastic region. The maximum stress at fracture for nanocomposites' samples was appeared for the sample (PMMA: 2%NR:0.3%CP) that strengthened with 0.3% CP nanoparticles compared with the other kinds of bio composite specimens.

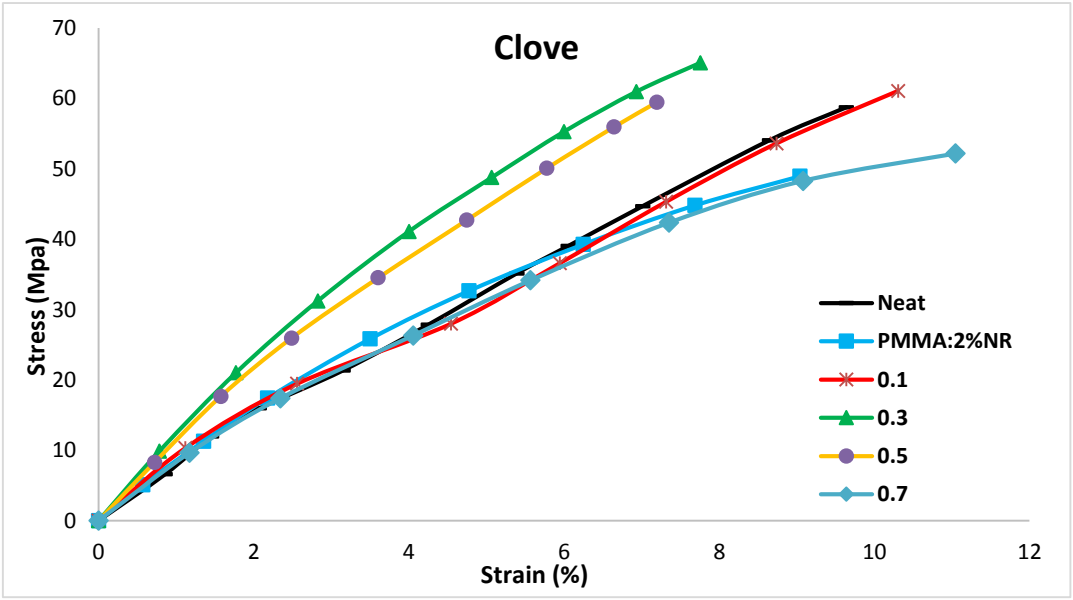


Figure 7. Stress-strain curve for PMMA bio composite specimens (PMMA:2%NR:X%CP) as a function of weight fraction content for (CP powder) in composites.

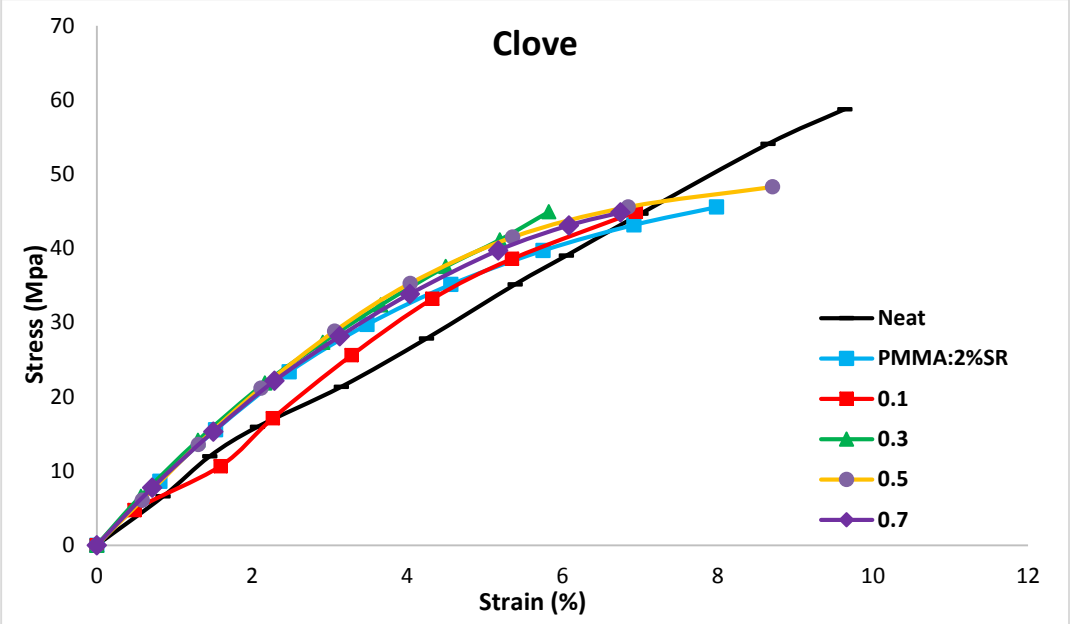


Figure 8. Stress-strain curve for PMMA bio composite specimens (PMMA:2%SR:X%CP) as a function of weight fraction content for (CP powder) in composites.

From the figures (9, 10 and 11), it can be noted that there is an increase in tensile strength, elastic modules and the percentage of elongation at fracture with increase of the weight fraction of CP nanofiller content for both kinds of PMMA nanocomposites as compared to types of polymer blend matrix (PMMA: 2% NR); (PMMA: 2% SR) and reached to maximum values at 3% ratio of CP for composite group samples ((PMMA: 2%NR): x %CP). The increase in tensile strength and elastic modules is due to the nature of bonding force between the polymer blends and nanoparticles of CP which is a strong bonding that does not let cracks or any defects formation in quick manner and in turn, the composite material will have a high tensile strength [21]. Moreover, this may be associated with the nature of stiffness between the elements of bio composites materials, this referred to a good compatibility between the polymer blends and natural powders [22]. The maximum values of stress at fracture reached to 66 MPa for bio composite consisting of ((PMMA: 2%NR):0.3%CP) compared with the other types of specimens. The higher values of tensile strength and elastic modules of nanocomposite having primary polymer blend (PMMA:2%NR) reinforced with (0.3 wt.%) of CP reached to (65 MPa, 1.033 GPa), respectively.
Moreover, from figures (9) and (10), it was found that the tensile strength and elastic modules respectively for nanocomposites samples ((PMMA:2%NR):x% CP) have higher values as compared with their counterparts of other group samples ((PMMA:2%SR): x% CP). This result may be related to the nature each of both NR and SR materials, as well as, it is attributed to a good compatibility between NR material and the components of nanocomposites samples.

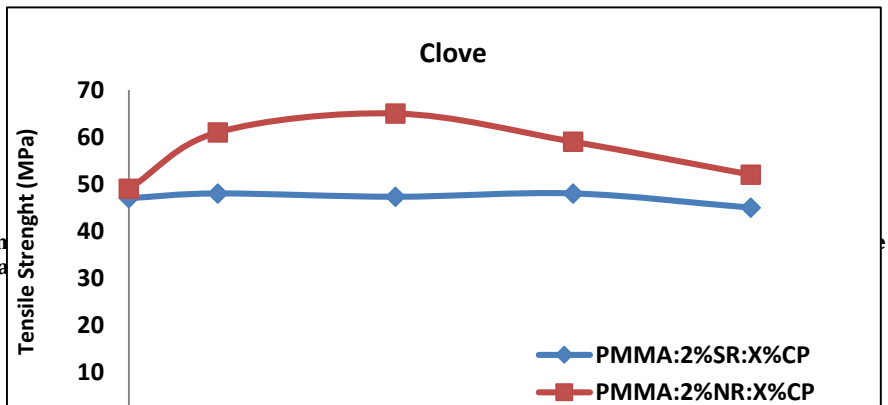


Figure 9. Tensile strength for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

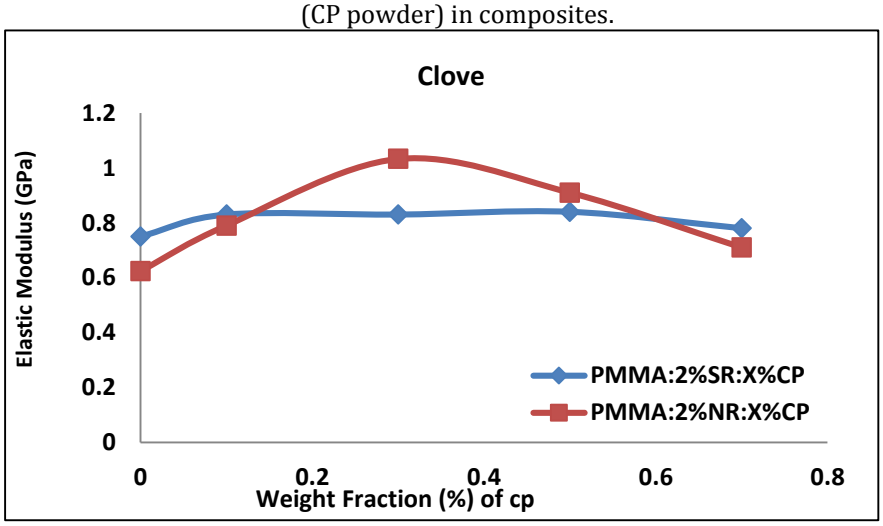


Figure 10. Elastic modules for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

Form the Figure (11), it can be observed that the percentage of elongation at fracture of bio composites specimens increased with increase the weight fraction of CP powder content in composites samples. The maximum value of percentage of elongation at break was reached to 5.88% for bio composite consist of (PMMA:2%NR:0.3%CP) compared with the other types of specimens. On the other hand, it was noticed that the percentage of elongation at break for nanocomposites samples ((PMMA:2%NR): x% CP) has higher values as compared with their counterparts of other group samples ((PMMA:2%SR): x% CP). This result may be related to the nature each of both NR and SR materials, where the chains structure of natural rubber material is more flexible as compared to the chains structure of silicone rubber, where the backbone chain of silicone rubber is made of alternating silicon and oxygen atoms and with two groups of atoms (CH3) that are side-bonded to the backbone chain [23].

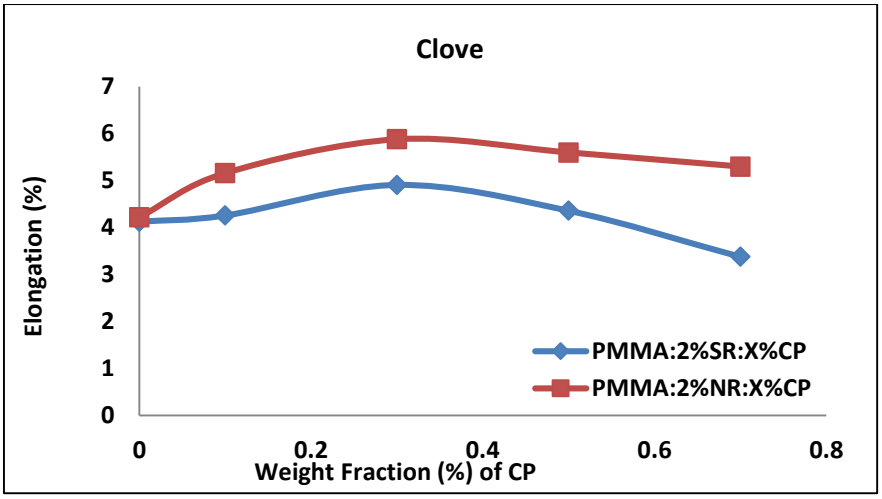


Figure 11. The percentage of elongation at break for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

4.3 Hardness Test Result

The relationship between hardness and weight fraction of CP nanoparticles is shown in the Figure (12). It can be seen that the hardens of the bio composite specimens increased with increase the weight fraction of CP for both types of nanocomposites polymer. This behavior is due to the high strength of compatibility between the constituents of nanocomposites and the nature of chains structure for both of natural or silicone rubber as a second material. Also, the high hardness of clove powder, as compared to polymer blend materials that used in this work, led to increase the hardness of bio composite specimen. The higher hardness values for nanocomposite for the samples that contain blend (PMMA: 2%NR) reinforced with (0.7 wt.%) of CP nanofiller reached to (89) compared with the (86.6) for the blend material of (PMMA:2%NR).

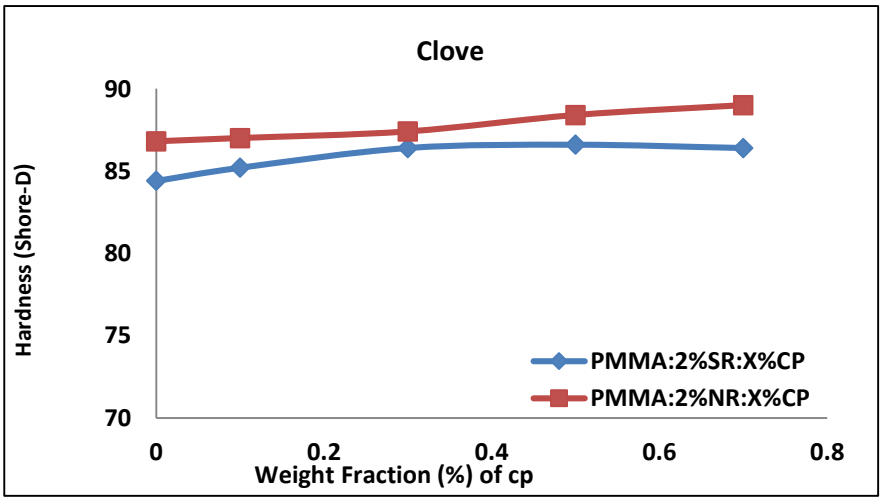


Figure 12. Hardness for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

4.4 Compression Test Result

Figure (13) illustrates the compression strength of bio nanocomposite specimens reinforced with (0.0%,0.1%,0.3%,0.5%,0.7%) of CP Nano powder. From this figure, it was observed that the compression strength increased with increase weight fraction of CP content for all bio composite

specimens. This is due to the high adhesion between the polymer blend and the high strengthening mechanism of CP powder that leads to decrease the free volume in the nanocomposites structure, and this reduced the motion of molecular polymer chains, so the result increased the stiffness of PMMA chains and this leads to prevent the crack propagation and exhibit a resistance to compression under applied strain [24]. The maximum value of compression strength found in the bio composite specimens containing ((PMMA: 2%NR): 0.5%CP) reached to (340 MPa) compared with the blend sample (PMMA:2%NR) that has equal to (230 MPa). As well, from this figure, it was noticed that the compression strength of bio nanocomposites specimens ((PMMA:2%SR): X%CP) was higher than the bio nanocomposite specimens ((PMMA:2%NR): X%CP). This result related to the nature of both NR and SR materials, as mentioned earlier, which associated to the chains structure of silicone rubber, where the backbone chain of silicone rubber is made of alternating silicon and oxygen atoms and with two groups of atoms (CH3) that are side-bonded to the backbone chain [23].

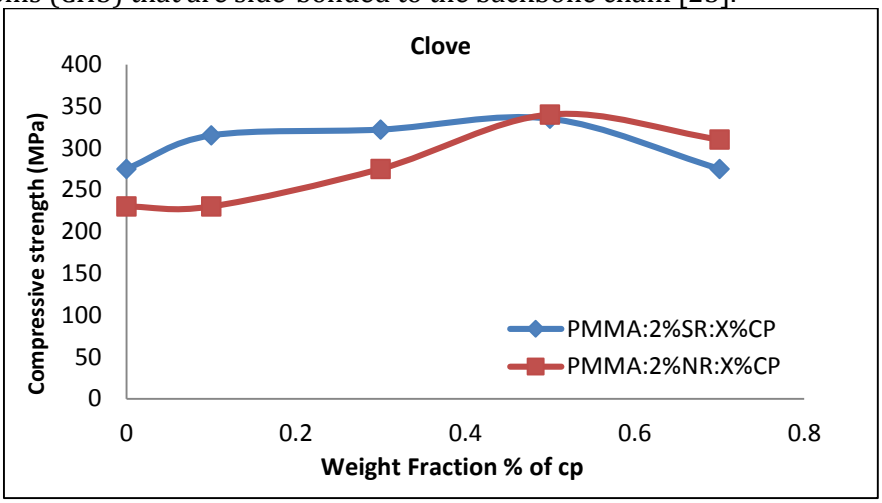


Figure 13. Compression strength for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

5. Conclusions

From the results of this work it was concluded the following items: -

1. The FTIR spectra showed that no any new peaks were appeared, or any aberration in the positions of peaks were noted for all samples of nanocomposites. This indicates that there is no any chemical interaction in these samples of nanocomposites based on the polymer blend.
2. Mechanical properties of polymer blend improved with adding natural powder of clove material to it.
3. The bio nanocomposites material ((PMMA: 2%NR): x% CP) got higher values in the mechanical properties (tensile strength, elastic modules, the percentage of elongation at break and hardness), as compared with ((PMMA: 2%SR): x% CP). Whereas, in contrast, the compression strength of nanocomposites samples ((PMMA: 2%SR): x% CP) gained the higher values.
4. The maximum value of tensile strength, elastic modules and elongation were reached at 0.3% ratio of CP content in composite based on (PMMA: 2% NR), and the maximum value of hardness reached at 0.7% ratio of CP content in the same group of nanocomposites. While, the higher value of compression strength of bio composite samples reinforced was found with 0.5% of CP powder for nanocomposites group samples ((PMMA: 2%SR): x% CP).

5. The highest values of tensile strength, elastic modules, elongation and hardness and compression strength for bio composite specimens with matrix (PMMA: 2% NR) are 65MPa, 1.033 GPa, 5.88%, 89 and 340 MPa, respectively.
6. Based on these results, it can be concluded that the addition of 2% natural or silicone rubber with the natural nanoparticles of clove powder to poly methyl methacrylate material are favorable materials in their use in improving the mechanical properties of the total or partial dentures base.

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