

Development on Mechanical Properties of PMMA by Blending it with Natural Rubber or Silicone Rubber and Reinforced by Nanoparticle

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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 the hand lay-up method. The maximum values of tensile strength and modulus of elasticity were noted at 65 MPa and 1.033 GPa respectively for polymer blend nanocomposite [(PMMA: 2%NR): 0.5% CP], whereas, the Shore D hardness reaches 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 CP 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 concentrates on the denture base without artificial teeth. Polymethyl 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 as the preferred material for removable prostheses, dental implants, and orthodontics instrument [3]. PMMA resin shows a better mechanical, impact and physical properties compared to other polymer materials. Although PMMA material has poor mechanical and physical properties when used alone, where it is easily ruptured 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 where one of the procedure is to incorporate glass fibre to strengthen the fracture resistance and flexural strength of denture base resin. This study highlight the possibility of improving the flexural strength of the heat curing polymerized PMMA after strengthening with glass fibre, and it may be possible to apply distal tension on partial and whole denture bases [4].

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

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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%) 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 the incorporation of nanofillers of surface-treated aluminium oxide as a strengthening material were investigated. The results showed that the transverse strength was developed by the addition of (1 wt% and 2 wt%) nanoparticles, 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 aluminium as strengthening materials to self-polymerized (PMMA) resin. The results depicted that the values of the flexural strength, flexural modulus, hardness and maximum shear stress were improved by adding these nanoparticles [8].

Some mechanical characterizations of PMMA resin strengthened with siwak fibres as a natural material were examined. The results revealed improvement in hardness, Young's modulus, tensile strength, fracture toughness with the increase of length, and content ratios of siwak fibre, whereas the impact strength decreased with the increase of fibre concentration in composite samples [9]. The effects of some comonomers on the impact strength and flexural strength of acrylic resin, with glass fibre having 6 mm length at 3% weight ratio were studied. The results indicated that the highest flexural and impact strength was gained by the addition of 2% IBMA monomer strengthened with glass fibre [10]. The influences of adding zirconium oxide nanoparticles and glass fibres on the flexural and impact strengths of a polymethylmethacrylate denture base were considered. The results manifested the added 2.5% nano-ZrO₂ + 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 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 nanopowders of clove material.

2. MATERIAL AND METHODS

2.1 Materials

In this study, the complete dentures base samples include polymeric materials, which are Polymethyl Methacrylate (PMMA), natural rubber (NR), silicone rubber (SR) and reinforcement materials as natural powders in nanometre size. The matrix of materials contains polymeric blends (PMMA (heat curing): 2%NR), (PMMA: 2%SR) as a control sample. PMMA material was utilized as a 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.

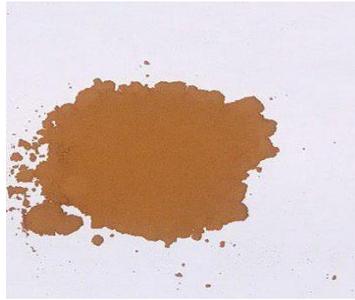


Figure 1. Reinforcement material: CP with nanoparticles size (75.18 nm).

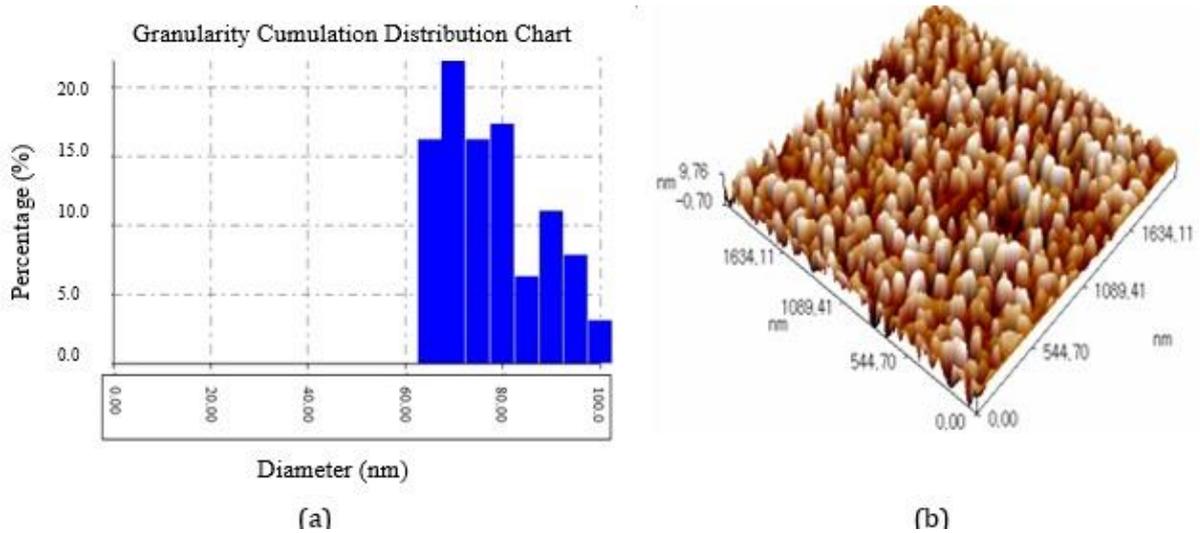


Figure 2. AFM test of CP nanoparticles with average diameter 75.18 nm, where (a) Granularity accumulation distribution chart of CP, and (b) Three-dimensional (XYZ) AFM pictures for CP.

Table 1 Particle size analysis of CP by AFM test with the 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			

2.2 Preparation of Specimens

In the present study, the bio-nanocomposite materials consist of PMMA base material which includes two parts: polymer powder and monomer liquid (methyl methacrylate, MMA). The standard percentage in mixing ratio for a heat curing acrylic resin was 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 almost homogeneous. After that, a powder of PMMA and reinforced material (e.g. clove powder) was 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 vapour from entering into PMMA during the curing. The curing process for acrylic was carried out under the conditions of 70°C and 2.5 bar for 30 minutes according to the company instructions. And then raise the temperature was raised to 100°C, and the acrylic 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, a final heat treatment at 55°C for 3 hours 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), the infrared spectrum was used as within a range of (400 - 4000) cm^{-1} .

3.2 Tensile Test

The tensile test specimen was prepared according to ASTM standard D638-87 [14]. The machine utilized for the testing is a microcomputer-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 until the failure of the specimen and the 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 a plain surface, smooth with thickness at a 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

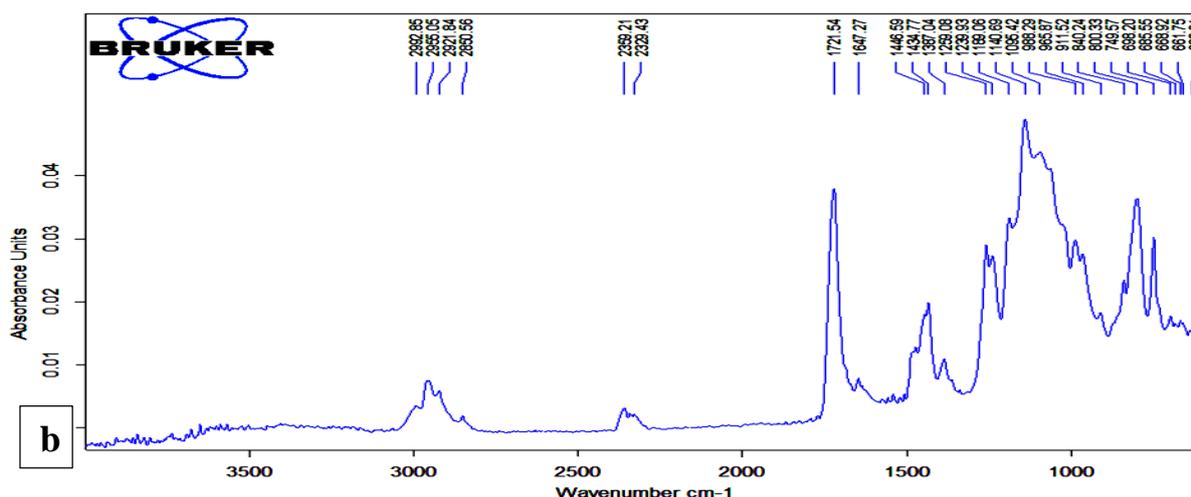
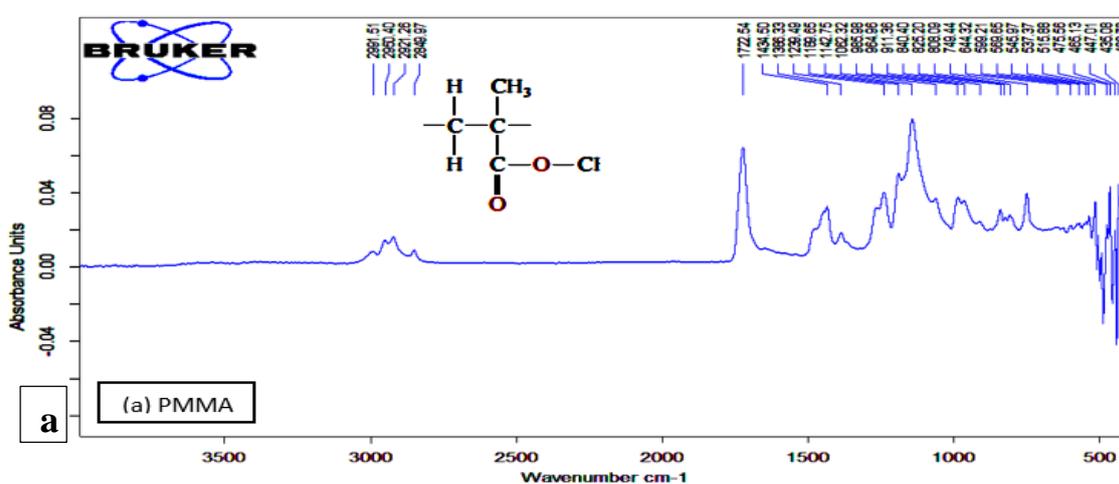
The compression test was carried out at room temperature by the universal tensile test machine produced by (Laryee Company in china), type (WDW-50), due to the (ASTM D695) [16]. The cross-head speed was (0.2 mm/min), and the load was applied step by step until the fracture of the samples took place. 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°C) for 48 hours [17].

4. RESULTS AND DISCUSSION

4.1 Fourier Transform Infrared Spectrometers (FTIR) Test Result

This test was utilized for the full 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) 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 the 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 1189.65 cm⁻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 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) match with that appeared in the FTIR spectrum of neat PMMA in Figure 3 (a).



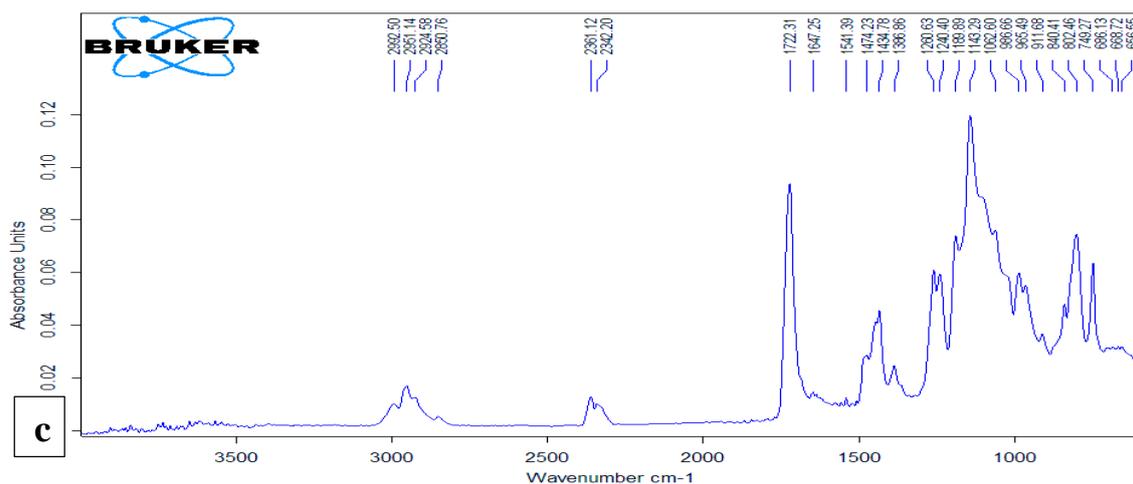


Figure 3. The Infrared Spectrum for (a) Neat PMMA (heat curing), (b) Polymer blend (PMMA: 2% NR) and (c) Polymer blend (PMMA: 2% SR).

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 seems 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 similar results to that appeared in 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 4 shows 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 the bandwidth of 2951.09 cm^{-1} . The sharp peak at 1732.08 cm^{-1} indicates the characteristic of carbonyl group C=O which leads to the presence of aldehydes, ketones and carboxylic acids, whereas 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.

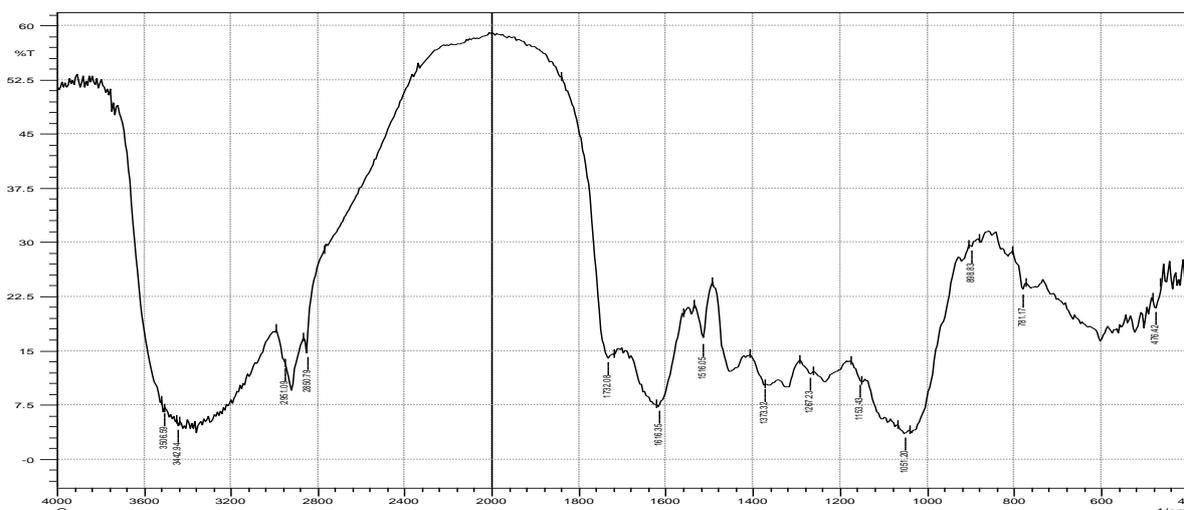


Figure 4. The FTIR spectrum for clove powder used.

Figures 5 and 6 elucidate the FTIR spectra for two groups of bio-nanocomposites which 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). In addition, 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 other new peaks have 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 in accordance with the physical bond, and no cross-linking and chemical reaction between the components of nanocomposites, and also no chemical interaction in these samples of nanocomposites based on the polymer blends.

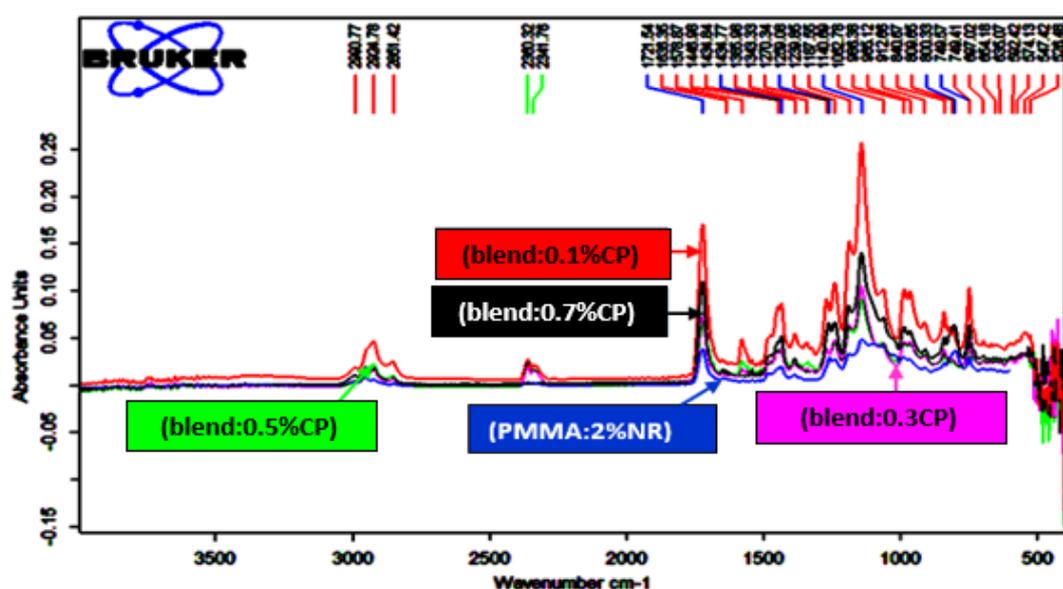


Figure 5. FTIR spectra for polymer blend (PMMA: 2%NR) and polymer blend composites ((PMMA: 2%NR): X% CP) as a function of Nano Clove powder content in composite.

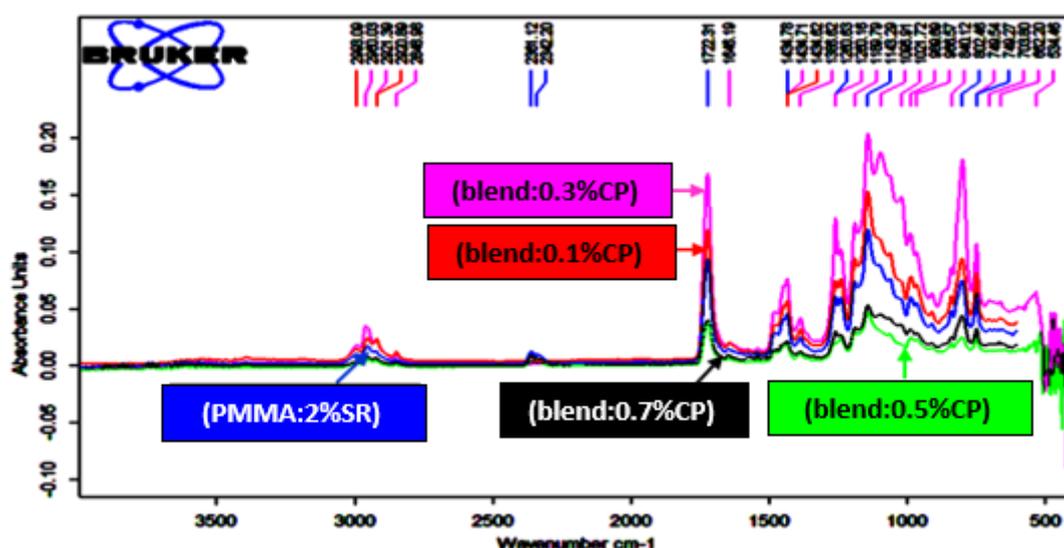


Figure 6. FTIR spectra for polymer blend (PMMA: 2%SR) and polymer blend composites ((PMMA: 2%SR): X% CP) as a function of nano clove powder content in composite.

4.2 Tensile Test Result

The influence of adding the natural particles of 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 modulus 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 behaviour of (stress-strain) curves changed with the percentage ratios of clove particles content in composite. The (stress-strain) curve was linear, where the specimen behaved in an elastic region and developed into non-linear due to the deformation of samples in the plastic region. The maximum stress at fracture for nanocomposites' samples appeared for the sample (PMMA: 2%NR:0.3%CP) that strengthened with 0.3% CP nanoparticles compared with the other kinds of biocomposite specimens.

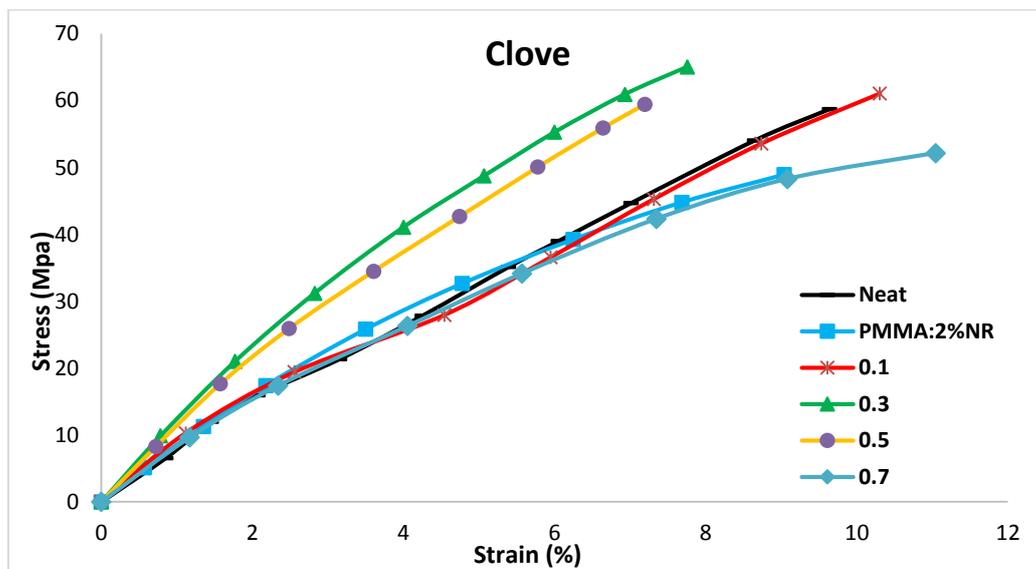


Figure 7. Stress-strain curve for PMMA biocomposite specimens (PMMA:2%NR:X%CP) as a function of weight fraction content for (CP powder) in composites.

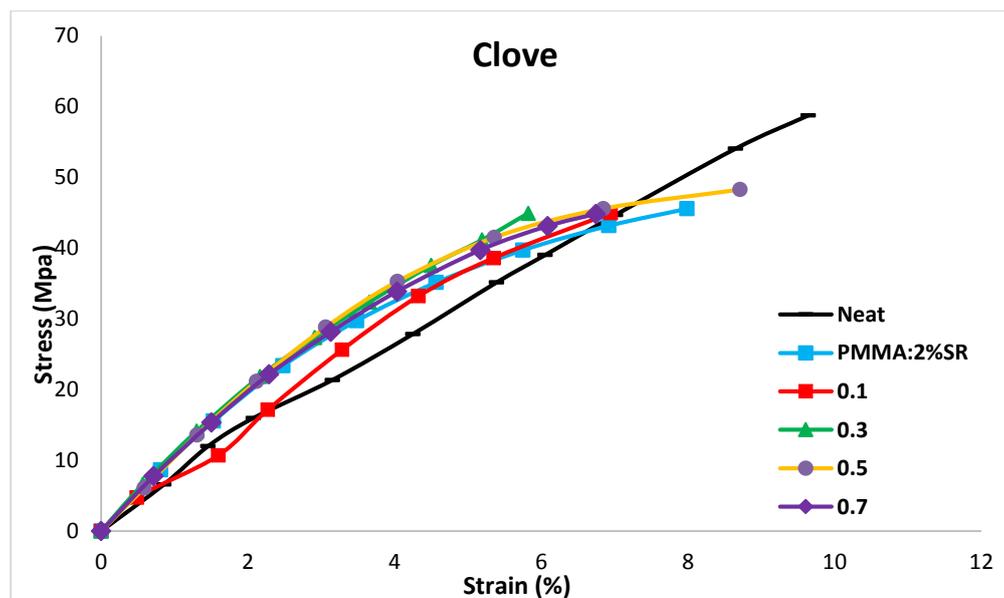


Figure 8. Stress-strain curve for PMMA biocomposite specimens (PMMA:2%SR:X%CP) as a function of weight fraction content for (CP powder) in composites.

From the Figures 9, 10 and 11, it can be noted that there is an increase in tensile strength, elastic modulus 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 modulus are 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 a 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 biocomposites materials, this referred to good compatibility between the polymer blends and natural powders [22]. The maximum values of stress at fracture reached 66 MPa for biocomposite consisting of ((PMMA: 2%NR):0.3%CP) compared with the other types of specimens. The higher values of tensile strength and elastic modulus of nanocomposite having primary polymer blend (PMMA:2%NR) reinforced with (0.3 wt.%) of CP reached to (65 MPa, 1.033 GPa), respectively.

Figures 9 and 10 shows the plot for tensile strength and elastic modulus, respectively. The tensile strength and elastic modulus in Figures 9 and 10 indicates that the 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 probably related to the nature of NR and SR materials, as well as, it is attributed to good compatibility between NR material and the components of nanocomposites samples.

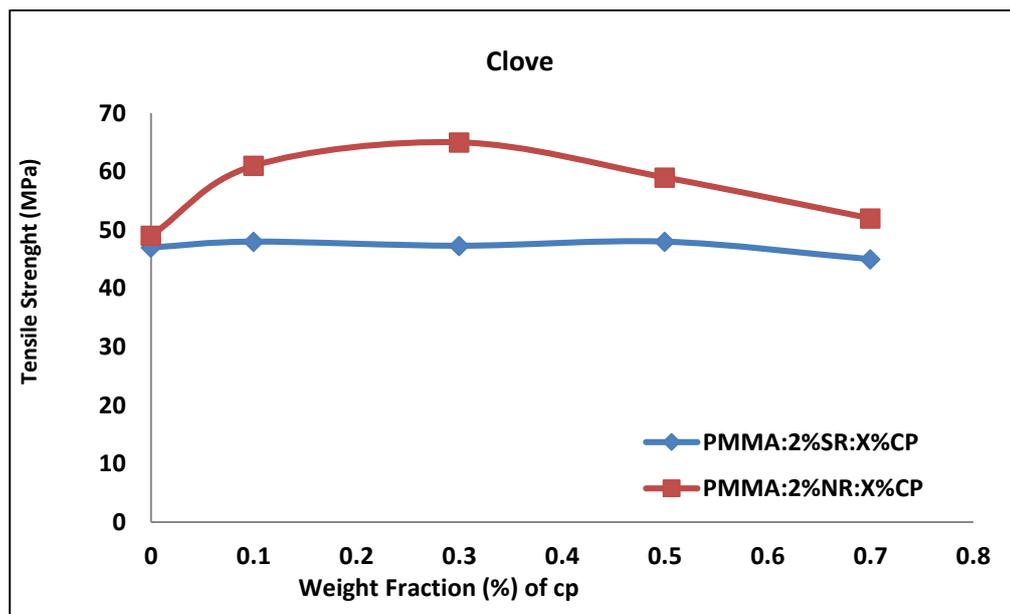


Figure 9. Tensile strength for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

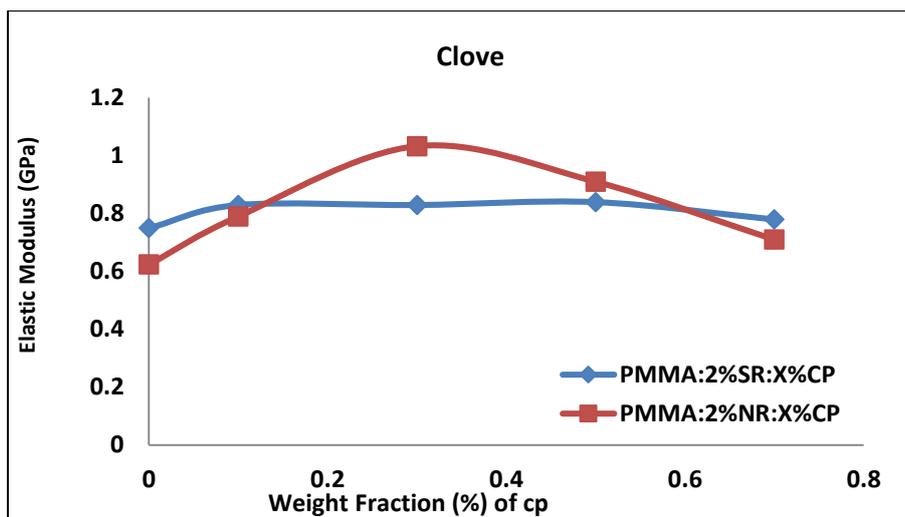


Figure 10. The elastic modulus for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

In Figure 11, it can be observed that the percentage of elongation at fracture of biocomposites specimens increased with increasing weight fraction of CP powder content in composites samples. The maximum value of the percentage of elongation at break reached to 5.88% for biocomposite that 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 to the counterparts of other group samples ((PMMA:2%SR):x% CP). This result may be related to the nature of the NR and SR materials, where the chains' structure of natural rubber material is more flexible compared to the chains' structure of silicone rubber, where the backbone chain of silicone rubber comprises of alternating silicon and oxygen atoms and with two groups of atoms (CH₃) that are side-bonded to the backbone chain [23].

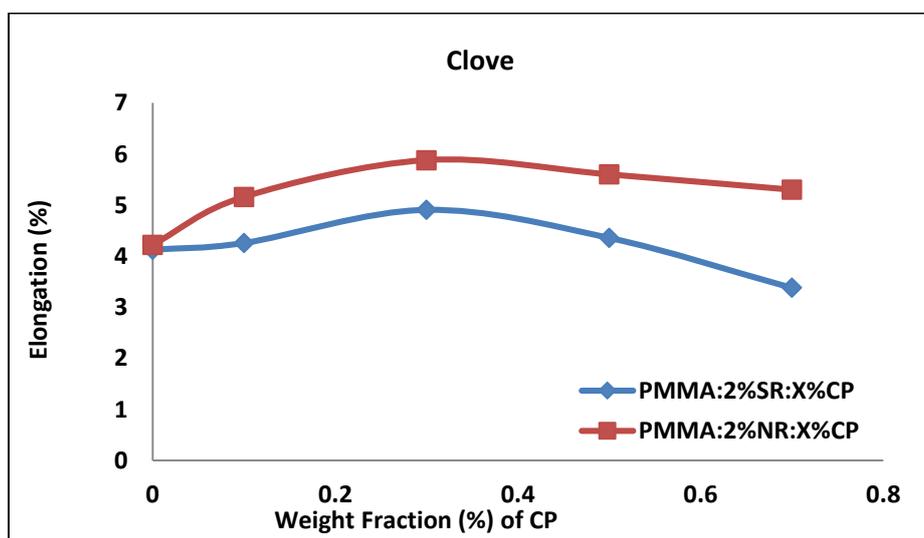


Figure 11. The percentage of elongation at break for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

4.3 Hardness Test Result

The relationship between hardness and weight fraction of CP nanoparticles is shown in Figure 12. It can be seen that the hardness of the biocomposite specimens increased with increasing the weight fraction of CP for both types of nanocomposites polymer. This behaviour is due to the high strength of compatibility between the constituents of nanocomposites and the nature of chains structure for both natural or silicone rubber as a second material. Also, the high hardness of clove powder, as compared to polymer blend materials used in this work, led to increasing the hardness of biocomposite 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).

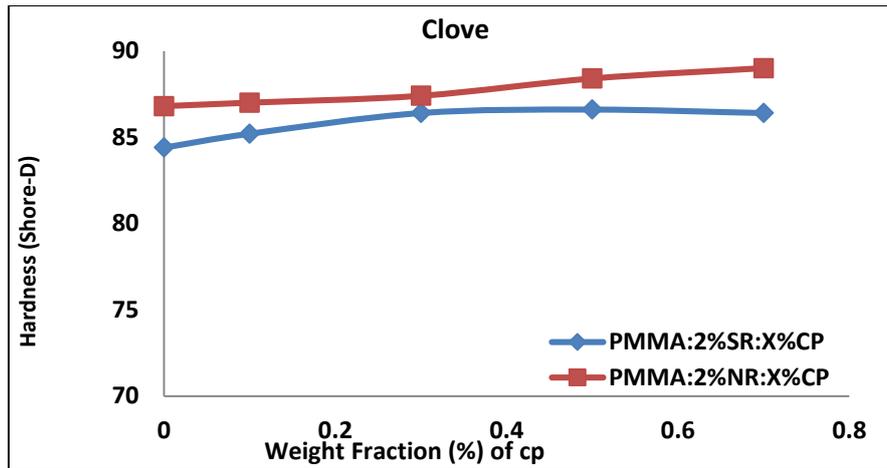


Figure 12. Hardness for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

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 nanopowder. The compression strength increased with the increased 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. Hence, result in increased the stiffness of PMMA chains that leads to preventing the crack propagation and exhibit resistance to compression under applied strain [24]. The maximum value of compression strength found in the biocomposite specimens containing ((PMMA: 2%NR): 0.5%CP) reached to 340 MPa compared to the compression strength for the blend sample (PMMA:2%NR) which is about 230 MPa. Furthermore, Figure 13 also shows 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 comprises of alternating silicon and oxygen atoms and with two groups of atoms (CH₃) that are side-bonded to the backbone chain [23].

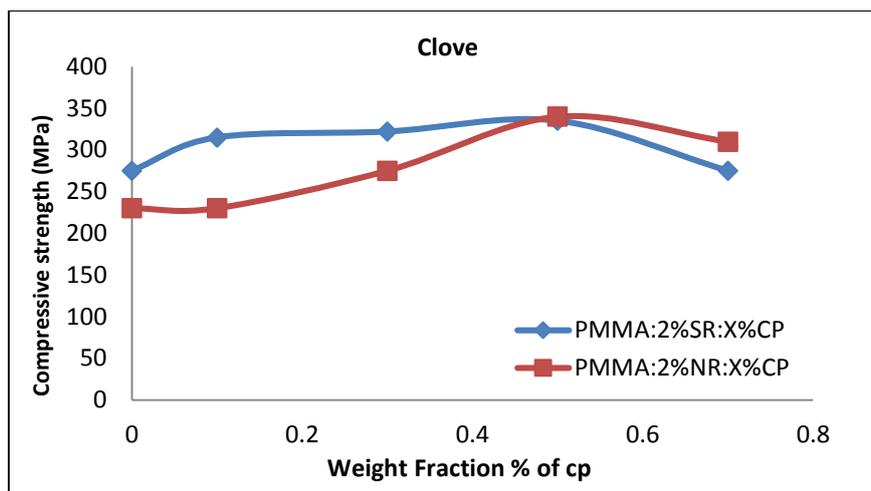


Figure 13. Compression strength for PMMA bio composite specimens as a function of weight fraction content for (CP powder) in composites.

5. CONCLUSIONS

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

- i. The FTIR spectra showed that no new peaks appeared, or any aberration in the positions of peaks for all samples of nanocomposites. This indicates that there is no chemical interaction in these samples of nanocomposites based on the polymer blend.
- ii. Mechanical properties of polymer blend improved with the addition of natural powder of clove material.
- iii. The bio-nanocomposites material ((PMMA: 2%NR): x% CP) got higher values in the mechanical properties (tensile strength, elastic modulus, the percentage of elongation at break and hardness), as compared with ((PMMA: 2%SR): x% CP). In contrast, the compression strength of nanocomposites samples ((PMMA: 2%SR): x% CP) gained higher values.
- iv. The maximum value of tensile strength, elastic modulus and elongation reached about 0.3% ratio of CP content in composite based on (PMMA: 2% NR), and the maximum value of hardness reached 0.7% ratio of CP content in the same group of nanocomposites. While the higher value of compression strength of biocomposite samples reinforced was found with 0.5% of CP powder for nanocomposites group samples ((PMMA: 2%SR): x% CP).
- v. The highest values of tensile strength, elastic modulus, elongation and hardness and compression strength for biocomposite specimens with matrix (PMMA: 2% NR) are 65MPa, 1.033 GPa, 5.88%, 89 and 340 MPa, respectively.
- vi. 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 polymethyl methacrylate material is a favourable material for improving the mechanical properties of the total or partial dentures base.

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