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Development of Surface Roughness and Mechanical Properties of PMMA Nanocomposites by Blending with Polymeric Materials

Abstract- This work aims to a development of mechanical properties of PMMA that is utilized in denture material, by using two types of polymers; blends (PMMA:2%NR) and (PMMA:2%SR) as a matrix materials strengthen with natural nanoparticles from the pomegranate peel powder (PPP) that were added at different weight fractions (0.0, 0.1%, 0.3%, 0.5% and 0.7%). Two groups of bio nanocomposites specimens were prepared, using (Hand Lay-Up) method. Experimental tests were carried out on surface roughness, hardness and wear rate as well as analyzing of FTIR test. The minimum values of surface roughness and wear rate were reached 1.51 nm and 0.317×10^{-8} g/cm respectively for polymer blend nanocomposite ((PMMA:2%NR): 0.7% PPP). Whereas, the maximum value of Shore D hardness reached 90 for the same sample of nanocomposites. According to these results, it can be a concluded that the addition of Nano pomegranate powder and natural rubber can develop the mechanical properties of PMMA material used in medical applications.

Keywords- PMMA, NR, SR, pomegranate peel powder, wear rate.

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1. Introduction

Poly methyl methacrylate (PMMA) is one of the oldest synthetic materials used in dentistry for denture base fabrication. PMMA polymer shows better mechanical and physical properties compared with other polymer types. Although, it has poor mechanical and physical properties when used alone, where it is easily ruptured during an accident, or when a patient applies high mastication force on the denture base [1]. One of the studies developments of the mechanical properties of acrylic resin used by glass fiber as strengthening material with 2mm in length, the result showed increased in the transverse strength, resistance to deflection and increased the modulus of elasticity [2]. A comparative study of resistance to fracture of heat polymerizing PMMA acrylic resin strengthen by glass and nylon fibers with 2% weight fraction of each other, the result showed the fiber-reinforced samples showed more resistant to impact and flexural fatigue than PMMA specimens and glass fiber reinforcement acrylic resin improves impact and flexural strengths of heat cure PMMA denture resin when compared with nylon fiber [3]. There are many attempts to strengthen polymers using varied procedures, one of them the effect of glass fiber strengthens on fracture resistance and flexural strength of denture base resin, this study was exhibited the possibility for

improving the flexural strength of heat cure polymerized PMMA after strengthening with glass fiber, and may be possible to apply distal tension on partial and whole denture bases [4]. It was reported that hydroxyapatite Nano powder that had been used in dental material as an effective biocompatible filler to reinforce the self-cured polymer matrix [5]. 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. In addition, the values showed that the impact of 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 [6]. Nanotechnology invaded the prosthodontics field for enhancement purposes of medical and material. The properties of strengthening PMMA resin by nanoparticles depends on the type, shape, size, and concentration of the particles [7]. 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 modulus, hardness and maximum shear stress improved by adding these Nano powders [8]. Studied to evaluate the effect of E-Glass fiber force (GFF) with 65 mm long and 10 mm wide place in the different region in the bending heat cure acrylic samples on the flexural strength, flexural toughness, and flexural modulus. The result illustrated presence GFF fibers near to the surface on its tensile stress side improves its flexural modulus, flexural toughness and flexural strength of the GFF in the middle of the samples did not change the flexural strength or the flexural modulus, but increased toughness by delaying fracture via continued absorption of energy through delayed fracture propagation without additional applied stress. Presence of the GFF near to the compressive side surface extends the flexural modulus but illustrate the only improvement in flexural strength or flexural toughness [9]. Investigated of some mechanical properties include: tensile, hardness, impact and infrared spectroscopy FTIR of (PMMA) resin as a denture base material strengthen with siwak fibers as a natural material. The results exhibited improvement in fracture toughness, tensile strength, Young modulus, hardness, with increasing ratios and length of siwak fiber, whereas the percentage of elongation at break and impact strength decreased with increasing of fiber concentration in bio composite specimens [10]. Studied the effect of different types of natural powders pomegranate peels (PPP) and seed powder of dates Ajwa (SDP) with weight fraction of (0.0, 0.4, 0.8, 1.2 and 1.6%) with average diameter of 53.38 nm and 93.78nm respectively on mechanical properties of PMMA bio composites specimens, the result showed a considerable improvement in these properties for both groups of bio composites, moreover, all bio composite specimens reinforced with pomegranate peels Nano powder (PPP) showed the highest properties compare with seed [11]. This study aimed to develop some mechanical properties of a PMMA material by adding silicone rubber or natural rubber and nanoparticle from pomegranate peel powder, to use in medical applications.

2. Materials and Methods

I. Materials

In this work, the complete or partial dentures base samples consist of poly methyl methacrylate (PMMA) blending with either natural rubber (NR) or silicone rubber (SR) and reinforcement with pomegranate peel in nanometer sized. Matrix contain (PMMA (heat curing): 2%NR),

(PMMA: 2%SR) as control sample. PMMA material used as liquid resin matrix, type (Spofa Dental Company, Czech Republic). A reinforced material as natural powder of pomegranate peel (PPP) was select with concentrations of (0.0, 0.1, 0.3, 0.5 and 0.7% wt.) and average diameter of 88.93nm. The mold used in this work made from low carbon steel with (160×5×20 mm) for tensile test and for hardness test (4 mm) thickness with diameter (50 mm) as showed in the Figure 1. The atomic force microscope AFM was used to verify the average diameter of nanoparticle and its distribution shown in Figure 2.

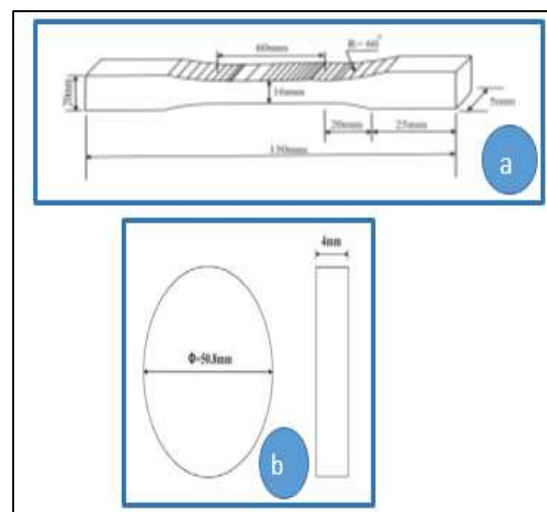


Figure1: Illustrates (a): The standard dimensions of tensile test specimens in (mm), (b) the schematic sample for standard hardness test sample.

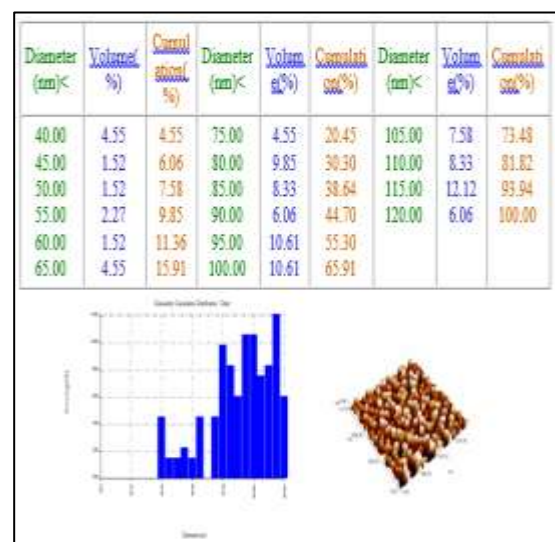


Figure 2: AFM test of Pomegranate peel nanoparticles (Average diameter 88.93nm).

II. Preparation of specimens

In this work, the bio nanocomposite materials consist of 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 this work to compose a polymer blends samples and bio nanocomposites samples. Initially it was mixed the liquid (MMA) part of acrylic resin 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 (pomegranate peel powder (PPP)) it was added to this mixture, with continuous mixing process, then the mixture was poured into metallic mold prepared for this purpose. The mold pressed by using a hydraulic compressor with pressure of about 2.5 bars to gain a smooth surface and to block gases vapor to entry into PMMA during the curing. The curing process for acrylic was carry out under the conditions of 70 °C and 2.5 bar for 30 min according to company instructions. And then raise temperature to 100 °C and remain at this temperature for one hour. Then the cooling the mold start into the curing device to oust the residual monomer. The samples were extracted from the metallic mold, with very smooth surfaces. Then, final heat treatment at 55°C for 3 hrs. was done to remove the residual stresses if it's found within samples.

3. Experimental Tests

I. 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) [12]. By using Fourier transform infrared spectrometer, model (SENSOR 27) made in Germany, by (Bruker Optics Company). Infrared spectrum was used within range of (400- 4000 cm^{-1}).

II. Surface roughness test

The surface roughness was tested for the prepared bio composites specimens, by using the Atomic force microscope (AFM) device (Tapping mode probes with constant amplitude 200mv), the test was done according to the (ASTM E2865) [13]. AFM test gave some of the properties that included (Roughness average and root mean average) which is indicates surface roughness. The test specimens were in disk form with a thickness 40mm diameter.

III. Hardness test

Hardness test was used (Shore D) the test was done due to ASTM- D-2240 [14]. To estimate the

hardness of the specimens, and the utilized ones should have a plain surface, smooth with thickness at a minimum more than (4mm) and should not be subjected to mechanical vibrations, the test sample made with diameter (50 mm).

IV. Wear rate test

The rotating Pin -on- Disc wear testing machine was done within a conditioned laboratory environment. The weighing method was used to determine the mass loss of the test specimens before and after the test. The sample was fixed in the holder and was abraded under load (5 N) and a sliding speed (2 m/sec.) with different times (5 and 10 min). The dimensions of the specimen were (10 mm) width and (20 mm) length, based on the standard wear tests described in ASTM standard D5963-97a [15].

4. Results and Discussion

I. Fourier transform infrared spectra (FTIR) test result

This test is utilized for full characterization of neat PMMA (heat curing), binary polymers blends (PMMA: 2NR) and (PMMA: 2SR) and bio nanocomposites specimens as a function of added nature powders of (pomegranate peels powder (PPP)) based to the 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 Figure 3. Is quite similar to that reported in the literature [11 and 16]. 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 appears 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 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 the plane and out of plane bending, respectively. These results are in a good agreement with that previously reported elsewhere [16 and 17]. Figures 4 and 5 show the FTIR spectra for two groups of bio Nano composites, which are

((PMMA: 2%SR): X%PPP) and ((PMMA: 2%NR): X%PPP) as a function of PPP content in composites materials. It can be seen from Figures 4 and 5 that all the characteristics vibration bands of neat PMMA Figure 3 are presents in the FTIR spectra for polymers blends specimens (PMMA: 2% SR) and (PMMA: 2% NR), which appears in the color is blue in Figures 4 and 5 respectively. Moreover, it can be seen from Figure 4 that the infrared spectra of the first group of bio nanocomposites specimens ((PMMA: 2%SR): X%PPP) and from Figure 5 for the infrared spectra of second group specimens of the bio nanocomposites ((PMMA: 2%NR): X%PPP); these spectra are quite similar to the FTIR spectrum for the neat PMMA Figure 3, moreover, it's also are matching with the infrared spectra of polymers blends (PMMA: 2%SR) and (PMMA: 2%NR) that mentioned above in Figures 4 and 5 respectively. On the other hand, no other new peaks have appeared or shifts in peaks locations were observed in the spectra of these bio nanocomposites when addition the nanoparticle of PPP to its.

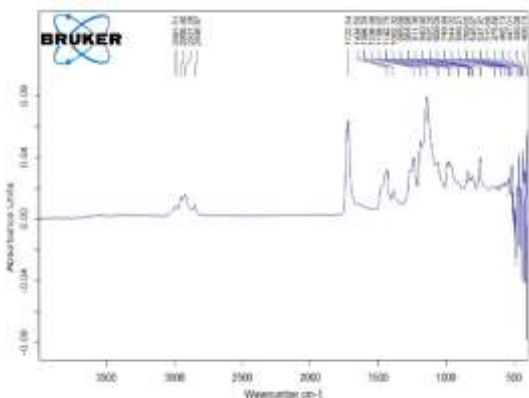


Figure 3: The Fourier Transform Infrared Spectrum for neat PMMA (heat curing).

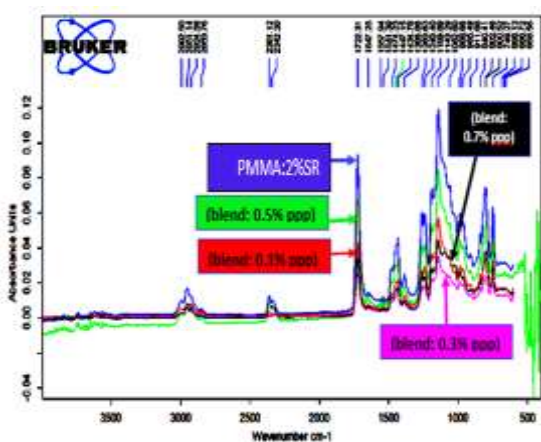


Figure 4: FTIR spectra for polymer blend (PMMA: 2%SR) in blue color and polymer blend composites ((PMMA: 2%SR): X%PPP) as a function of pomegranate peels powder content in composite.

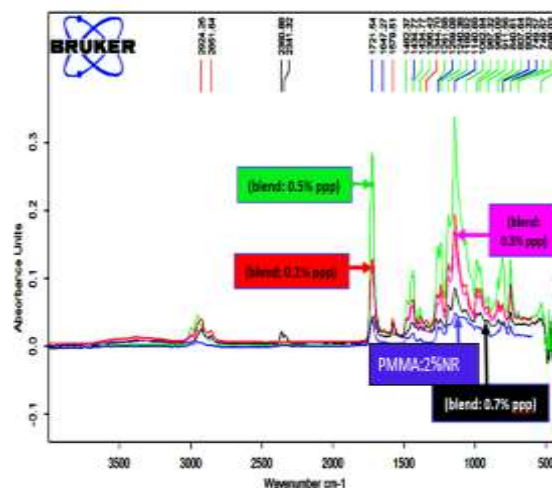


Figure 5: FTIR spectra for polymer blend (PMMA: 2%NR) in blue color and polymer blend composites ((PMMA: 2%NR): X%PPP) as a function of pomegranate peels powder content in composite.

II. Surface roughness test results

Figure 5 shows the morphology of surfaces roughness which was tested by AFM technique, from this figure it was observed, there are different construction in the surface structure morphology of the samples depending on the roughness of the surface, which varies according to the nature of their components, where Figure 6 a represent surfaces morphology of neat PMMA, figure 6 b and c represent surfaces morphology of polymeric mixtures (PMMA: 2%SR) and (PMMA: 2%NR) respectively. It is evident from the surface's construction that surface structure of neat PMMA sample Figure 6 a is smoother with surface roughness 4.69nm as compared to the blended samples. Also, it is observed that the sample of polymer blend (PMMA: 2% SR) Figure 6 b has the highest surface roughness compared to blend (PMMA: 2% NR) Figure 6 c. The surfaces morphology for nanocomposite's specimens reinforced by nanoparticle of pomegranate peel powder (PPP) with weight fraction ratios (0%, 0.3%, 0.5%, 0.7%) shown in Figure 6 d, e, f, g, h and i, where d, e and f represent the nanocomposites that based on polymer's blends (PMMA: 2% SR) and (g, h and i) represent the nanocomposites based on polymer's blends (PMMA: 2% NR). From these surface's construction, it was noticed that surfaces structure for the nanocomposites based on polymer's blends (PMMA: 2% NR) have the lower value of roughness surfaces morphology as compared with their counterparts' samples of nanocomposites based on blends (PMMA: 2% SR).

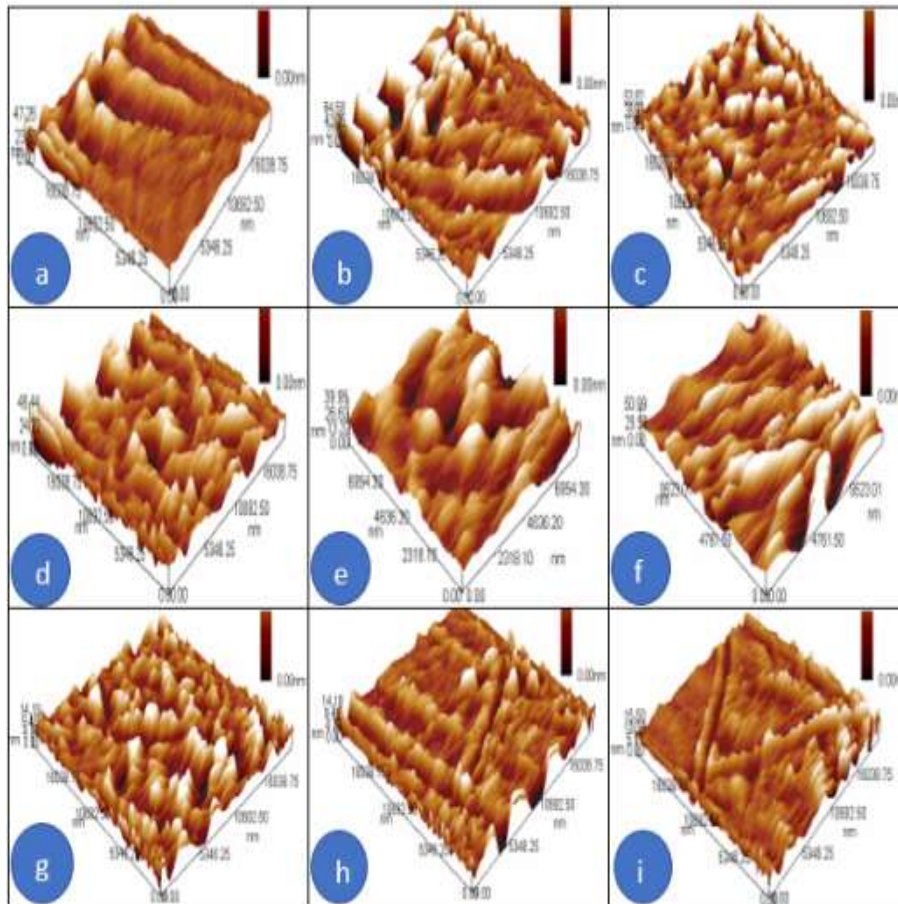


Figure 6: The structure of surfaces roughness by AFM technique where a: Neat (PMMA), b: polymer blend (PMMA:2%SR), c: polymer blend (PMMA:2%NR), and (d, e and f) nanocomposites that based on polymer's blends (PMMA: 2% SR), as a function of PPP content respectively, as well as, (g, h and i) nanocomposites that based on polymer's blends (PMMA: 2% NR) as a function of PPP content respectively.

Figures 7 illustrate, the surfaces roughness for the nanocomposite's specimens, that based on two types of the polymer's blends (PMMA: 2% NR) and (PMMA: 2%SR) as a matrix material. This figure shows the effect of addition natural nanoparticle of pomegranate peel powder (PPP) with weight fraction ratios (0%, 0.3%, 0.5%, 0.7%) on the surface's roughness for the nanocomposite's specimens. It can be noted from these figures that the values of surfaces roughness for bio nanocomposites specimens decreased with an increase in the weights fractions ratios of pomegranate peels powder (PPP) in the content of blended matrixes, and reach to lower value of surfaces roughness (1.51 nm) at 7% ratio of PPP for composite group samples that based on (PMMA: 2%NR) as a matrix. This result is related to the nature of components of polymer blend and the ratio of reinforcement material in composite sample and according to the mode distributed of reinforcement material as

nanoparticles inside the polymer blend matrix [18]. As well as, due to the nature of bonding force between the components of polymer blends and nanoparticles that does not let any defects formation or formation of porous gaps in the microstructure of composite material all that lead to earn the smooth surfaces [19]. Moreover, it was observed from Figures 6 and 7, that the surfaces roughness of bio nanocomposite's specimens, that based on the polymers blends (PMMA: 2% NR) as matrix material lower than their counterparts of the second group of bio nanocomposite's specimens, which based on the polymers blends (PMMA: 2% SR) as matrix material, this result may be attributed to the nature and behavior each of SR and NR in composite. So that, the blending of PMMA with SR material may be lead to the formation of a heterogeneous structure with the components of the composite material, this eventually lead to increase the proportion of defects in the grid of

polymeric composite which lead to increases the roughness of the surfaces, so, the smoother structure of surfaces roughness have more noteworthy for flawless polymer blend grid. especially when the bonding between the components of polymer blend and the Nano fillers material good sufficiently, Therefore, the high smoother surfaces quality of the flawless and filled composite be higher than that of the filled composite and contain on the flaws [20]. As well as, it was noticed that the lower value of surface roughness for bio composite specimens with (PMMA: 2%NR) as matrix and strengthen with 0,7% ratio of PPP particles equal to (1.51nm) compared with polymer blend matrix which equal to (9.3nm).

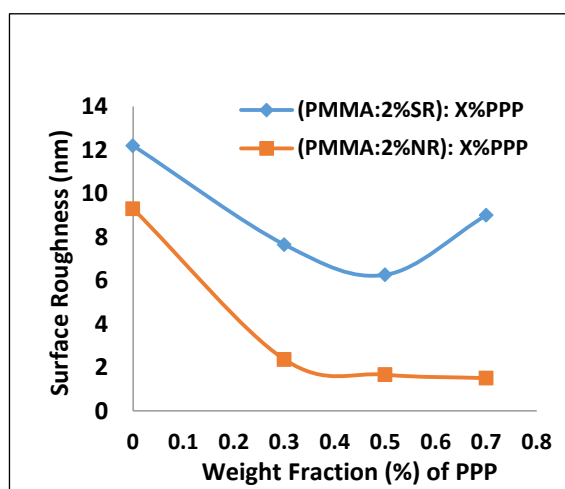


Figure 7: Surface Roughness for two groups of bio nanocomposite specimens ((PMMA: 2%SR): X%PPP) and ((PMMA: 2%NR): X%PPP) as a function of PPP content in composites.

III. Hardness test result

Figure 8 illustrates the relationship between hardness and weight fraction of PPP nanoparticles. It can be noted the hardness of the bio composite samples slightly increased with increasing the weight fraction of PPP for both types of bio nanocomposites. This behavior is due to good compatibility between the constituents of nanocomposites and the nature of chains structure for each of natural rubber or silicone rubber as a secondary material. Also, the hardness of natural powders (PPP) may be a stronger and stiffer as compared with polymer blend reference samples. As well as, that related to the bonding strength and wettability between the polymer blends and these particles, which lead to make surface harder by inhibit the polymeric chains motion along the stress direction. Moreover, it was found that, the bio nanocomposites specimens which based on the polymer blends (PMMA: 2%NR) matrix and

reinforced with PPP nanoparticle have great value of hardness compare with all other group. This result related to natural of PPP nanoparticle to improve resistance to deform plastically [21, 22]. In addition, the high hardness of PPP powder as compared to polymer blend materials that used in this work led to slightly increase in the hardness values of bio composite specimen. The higher value of hardness for nanocomposite for the specimen that content on blend (PMMA: 2%NR) reinforced with (0.7 wt.%) of PPP nanofiller reach to (90) compare with that based on blend material of (PMMA:2%NR) equal to (86.8).

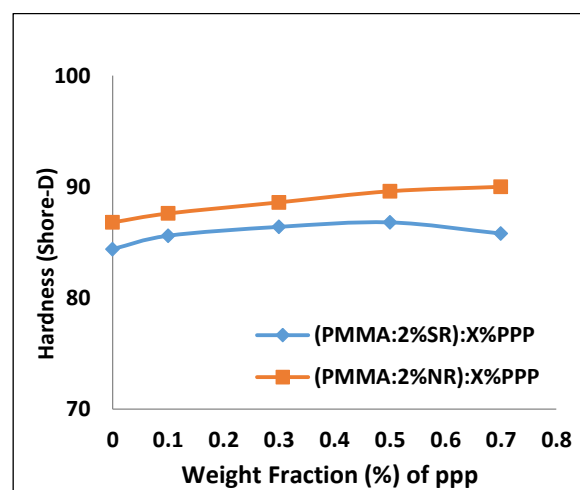


Figure 8: Hardness for PMMA bio composite specimens as a function of weight fraction content for (PPP powder) in composites.

IV. Wear Rate Test Result

The wear rate was tested under load (5 N) and a sliding speed (2 m/sec.) with different times (5 and 10 min). Figure 9 a and b show the influence of add pomegranate peel powder as nanofillers on the wear rate of bio nanocomposite specimens that based on the polymers blends (PMMA: 2%SR) or (PMMA: 2%NR) respectively. From this figure, it can be noted that the wear rate of all bio composites specimens decreases with increasing weight fraction of PPP content in composite material, that test at a constant time (5 min) and loading force (5N). Increase test time to 10 min also, a decrease in wear rate continues of all bio composites samples with increasing PPP content in composites materials. On the other hand, the wear rate decreases with the increased test time from 5 min to 10 min. This behavior may be attributed to the addition of natural nanoparticles into a polymer blend, led to increase the hardness values of the bio composites specimens (as mention earlier), and this may be resulting in a decrease in the rate of wear. As well as, it may be associated with good

adhesion between nanoparticles (PPP) and components of the polymer blend, this limits the release of materials from the composite's samples. This result indicates that the wear resistance process is more fixed for bio composites by filling it with natural nanoparticles (PPP) and have good adhesion force and compatibility between their components [22,23]. Samples of bio nanocomposite that based on the polymers blends (PMMA:2%NR) and reinforced by PPP nanoparticle achieved the minimum values of wear rate that equal to $(0.317 \times 10^{-8} \text{ g/cm})$, through 10 min of test time, as compared with their counterparts for bio nanocomposite samples that based on the polymer blend (PMMA:2%SR) where the minimum value of wear rate reached to $(0.67 \times 10^{-8} \text{ g/cm})$ and compared with blend matrix (PMMA:2%SR) which reached to $(0.84 \times 10^{-8} \text{ g/cm})$ under the same test condition.

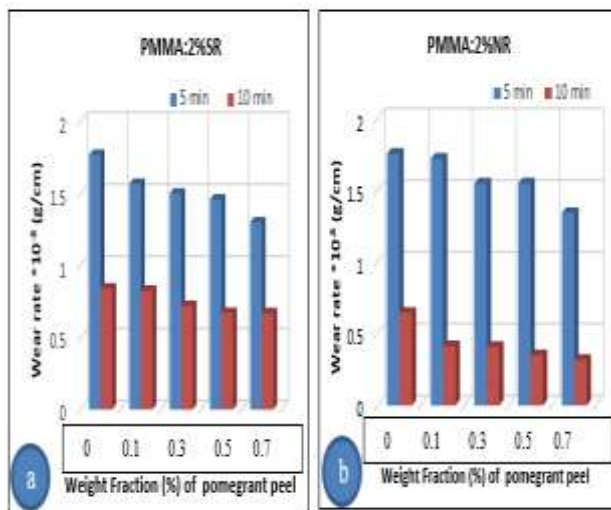


Figure 9: Wear Rate of bio Composite Specimens where (a): bio Composite based on the blend of (PMMA: 2%SR) (b): bio Composite based on the polymer blend of (PMMA: 2%NR), as a Function of weight fraction content of PPP and at different times of loading (5min and 10min).

5. Conclusions

1. Mechanical properties of polymer blend improved with adding the natural powder of pomegranate peel powder material to it.
2. The bio nanocomposites material ((PMMA: 2%NR): x% PPP) get lower values in the (Surface roughness and wear rate), as compared with that based on (PMMA: 2%SR) and slightly increase in shore D hardness.
3. The minimum value of surface roughness was reached to (1.51nm) at 0.7% ratio of PPP content for composite based on (PMMA: 2% NR).
4. The lower values in wear rate for bio composite specimens based on matrix (PMMA: 2% NR) and strengthen with 0.7% ratio of PPP

reached to $(0.317 \times 10^{-8} \text{ g/cm})$, as well as, minimum value of wear rate for nanocomposite samples based on matrix (PMMA: 2% SR) with same ratio of PPP reached to $(0.67 \times 10^{-8} \text{ g/cm})$.

5. It was noticed from surface's construction by AFM test, that the nanocomposite sample based on polymer's blend (PMMA: 2% NR) and reinforced by 0,7% ratio of PPP has smoother structure for morphology of the roughness surfaces as compared with their counterparts that based on blends (PMMA: 2% SR) and the neat PMMA material. Based on what was mentioned above, it can be concluded that the addition of 2% from each of natural or silicone rubber with 7% ratio of natural nanoparticles of pomegranate peel powder to PMMA material produce composite materials are favorable materials to use in medical applications.

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