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Development the Physical Properties of Polymeric Blend (SR/ PMMA) by Adding various Types of Nanoparticles, Used for Maxillofacial Prosthesis Applications

Abstract- *As maxillofacial defects increased due to cancer; it became necessary to select high-quality prosthetic materials in this field. Silicone rubber is widely used in damaged maxillofacial affected areas replacement surgery as bio material. The aim of this research, prepared a nano composites materials, from polymer blend (silicone rubber: 5% PMMA) reinforced by different types of nano-powders; pomegranate Peels Powder (PPP), Seeds powder of dates Ajwa (SPDA) and TiO_2 nano-powders with loading level (0.0, 0.1, 0.2, 0.3 and 0.4%). Some physical properties such as density, water absorption, and Thermo-Physical test, FTIR analysis, as well as, FTIR, antibacterial tests were done on prepared samples. The results showed that the composites material based of polymer blend with optimum percent are of 0.2% of pomegranate Peels Powder (PPP), 0.3% of Seeds powder of dates Ajwa (SPDA) and 0.1% of TiO2 nano-powders that have ideal characteristic. Also for antibacterial tests, polymeric blend composites with optimum percent of this nano-powders show that more antibacterial efficiency against S.aureus bacteria than E.coli bacteria.*

Keywords- *Maxillofacial Prosthesis, Nano powder, Polymer Blend, Physical Properties, PMMA, Silicone rubber.*

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Study the Microstructure and Mechanical Properties of High Chromium White Cast Iron (HCWCI) under Different Martempering Quenching Mediums

Abstract *This study aims to find the effect of hydroxide mixture as a quenching medium in martempering heat treatment on microstructure and mechanical properties of high chromium white cast iron. This mixture is cheaper and more available than the ordinary nitrate mixture in Iraqi markets. High chromium white cast iron is used in mining, crushing and cement plants as mill liners and it is subjected to extreme conditions of wear and impact that cause failure, reduction in life and raise the cost of repairing. Hence it is important to improve its mechanical properties. In this study, two types of quenching mediums were used:(50% NaOH: 50% KOH) mixture and (50% NaNO3 + 50 % KNO3) mixture. The specimens were austenitized at 950°C for 1 hr then the first group was quenched in nitrate mixture, and the other was quenched in hydroxide mixture both at about 350°C for (1/2, 2,4,6,8) hr. The results showed an increase in hardness and decrease in toughness for both mixtures, and the higher hardness value was found for both of the mixtures at martempering temperature 350°C for 4hr quenching time.*

Keyword: *Hardness, High Chromium White Cast Iron, Martempering, microstructure, quenching mediums.*

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1. Introduction

High chromium white cast irons represent a class of materials commonly used for materials handling, crushing and grinding applications in the minerals and mining industries due to their excellent wear resistance [1,2]. After heat treatment, High chromium white cast irons microstructures are comprised of eutectic carbides and a metallic matrix comprised of secondary chromium carbides, martensite, austenite and in some cases ferrite. During solidification, the eutectic carbides are formed, and they do not undergo a further transformation, but the matrix can be altered. Microstructures in the matrix are affected by three factors: thermal cycle, chemical composition and the initial state of alloy (as-cast or annealed). This will, in turn, affects the hardness and wear resistance of the material. In general, at the end of the solidification process, the microstructure of high-chromium white cast iron is composed of a primary phase (austenite dendrite) and a eutectic compound (M_7C_3) [3]. The reference [4] studied the influence of variables such as holding temperatures and times during aus tempering of High Chromium White Cast Iron (HCWCI). It is found that the best performance of alloy against the abrasion was at austempering at 450 ºC with 6 hours holding time. The reference [5] studied the effects of soft annealing and hardening and tempering on the hardness and microstructure of high chromium cast iron. Their results showed that a proper selection of parameters of heat treatment could control the hardness and composition of phase microstructure of chromium cast iron. Hardening has positive effects on the hardness of castings, but tempering and soft annealing enhance ductile properties. The reference [6] observed the effect of changing the chemical composition and heat-treatments on the microstructure and mechanical properties of high chromium white cast iron. The results revealed marked improvements in mechanical properties and wear performance by adding carbide forming elements .on the other hand, the Destabilization heat treatment employed to obtain the martensitic structure for improving the toughness and abrasion resistance of these irons. In addition, the subcritical (tempering) heat treatments following the hardening are commonly utilized to relieve the internal stresses and to control the final hardness before service. The reference [7] studied the effects of heat-treatment on microstructure and mechanical properties of high chromium white cast iron alloyed with

1. Introduction

Prostheses which are applied to acquired tissue deficiency in the maxillofacial area are called "Maxillofacial prostheses" [1]. Maxillofacial patients have problems related with facial distortion, particularly after surgery [2]. The fundamental purpose of maxillofacial prosthetic is the effective treatment to recondition missing parts of the face, maxilla and mandible and solve the lost aesthetic look and function [3]. There are many materials have been used for maxillofacial prosthesis, maxillofacial silicone elastomer has been used for manufacture facial prostheses and repairing the maxillofacial defects because of its biocompatibility, chemical inertness, durability, easy of manipulation and coloration. [4].

For maxillofacial prosthesis, it should focus on developing the physical and mechanical properties of the material so that it will make more like human tissue and raise the service life of the prosthesis. Also, it should finding color-stable coloring agents for coloring the facial prostheses and improving a scientific method of color matching to human skin [5]. In the fact, there are many disadvantages of the materials used in facial prosthodontics are discolorations on orbital prosthesis after 1 year and tearing the silicone material of ear prosthesis. Thus for improving the performances of polymers and expand their application scopes, nanoparticles were incorporated into the polymer to obtain a nanocomposite [6]. The usual objective for preparing a novel blend of two or more polymers is to improve processability, product uniformity, quick formulation changes, plant flexibility and high productivity [7]. Many fruits and natural products have been analyzed for their antioxidant activity and medicinal properties [8].

Anna Karin Hulterstrom et al. studied the water sorption, wettability, and solubility of silicone rubber that used for facial prostheses. The results showed that condensation type polymers should not be used in prosthetic implants because it so large volumetric changes when exposed to liquid. While the addition type of silicones offered less sorption and solubility [9]. Al-qenae studied the physical properties of VST-50HD silicone elastomer maxillofacial material after mixed with nano alumina ceramic fiber as fillers. The results showed that there is no significant improvement of properties (tensile, tear and Shore A hardness strengths) [10]. Shakir and Abdul-Ameer developed the mechanical properties of two kinds of maxillofacial silicone elastomers (VST50FRTV and Cosmesil M511 HTV) by added nanoparticles TiO_2. The results show that the incorporation of (0.25 % and 0.2 %) of nanofiller increased the tensile strength, tear strength, and hardness of the materials [11]. Salih et al. studied the mechanical properties of blends (SR/PMMA) after addition of different loading level (5%, 10%, 15% and 20%) of PMMA material to silicone rubber. The results offered that the tensile strength, modulus of elasticity, hardness and compression strength increased and reaches to the highest values at 10% of PMMA content, so, this sample may be a favorable material to obtain the properties required for maxillofacial prosthetic applications [12]. Salih et al. studied the physical properties of polymer blend material (SR/PMMA). The results display that the optimum percent of PMMA is 10% which has perfect characteristic. So, this sample may be an appropriate material used for maxillofacial prosthetic applications [13].

Maxillofacial material must be tested for different physical properties in order to obtain ideal medical prosthetic material. In present research, the physical properties and anti-bacterial test of polymer blend composite reinforced three different nano powders pomegranate Peels Powder (PPP), Seeds powder of dates Ajwa (SPDA) and TiO_2 for maxillofacial prostheses were studied. The polymer blends nanocomposite was tested by density, water absorption, and thermal conductivity test.

2. Experimental Work

I. Materials Used

There are two materials used to prepared binary polymer blend as matrix for composites samples include Versiltal RTV Silicone elastomer type (VST-50F) is supply from Factor II Inc., Lakeside, USA that it is consisting of two parts, one is a liquid, and another is the catalyst. polymethyl methacrylate (PMMA) is a second material of the blend which cold curing resin with Castavaria type, provided from Spofa Dental Company. The reinforcement's materials for composites samples are Pomegranate Peels Powder (PPP) taken from pomegranate fruit (Punica granatum) which were supply from Saudi Arabia with particles size (102.45 nm), Other one is Seeds powder of dates Ajwa (SPDA) taken from date's ajwa fruit which were supply from Saudi Arabia with particles size (59.26 nm) and

TiO_2 which was supplied from USA with particles size (82.13 nm).. The Atomic force microscope AFM was used to determine the average diameter of nanoparticle and its distribution. Figure 1, 2 and 3 show the size and distribution for pomegranate peel powder and seed powder of dates ajwa and TiO_2 nanoparticles respectively.

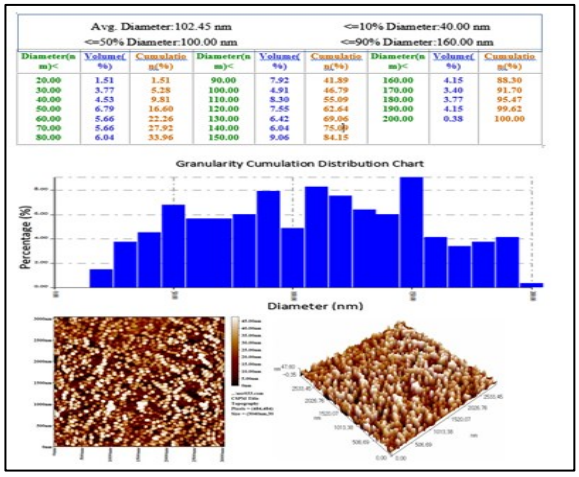

Figure 1: AFM Test of Pomegranate Peels Particles (Average Diameter (102.45 nm)).

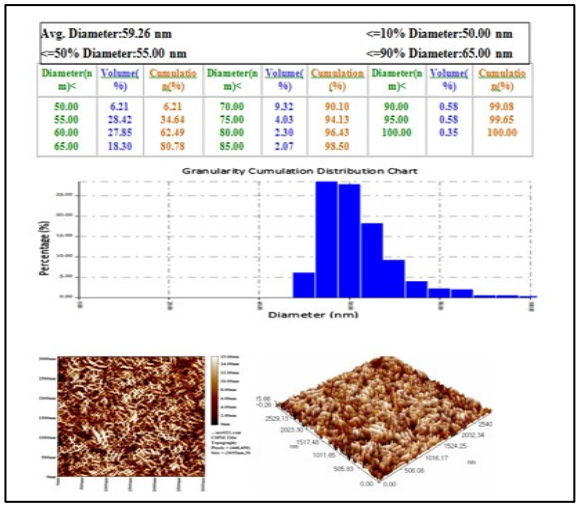

Figure 2: AFM Test of Seeds powder of dates Ajwa (average diameter 59.26 nm).

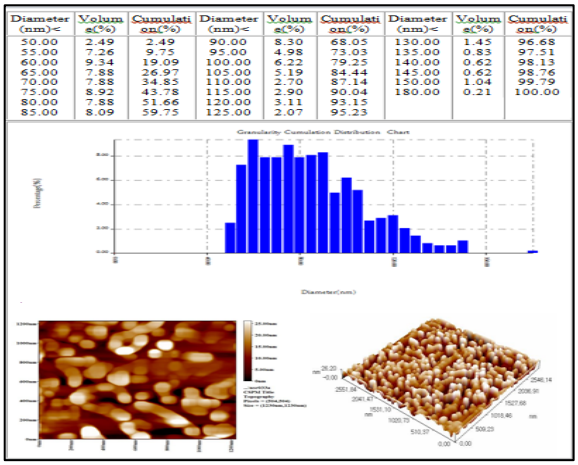

Figure 3: AFM Test of TiO_2 particle (Average Diameter (82.13 nm)).

II. Preparation Method

In this work, mechanical mixing was used to prepare polymer blends composites by binary polymer blends (SR: 5%PMMA) as a matrix material reinforced with different percentages ratios of pomegranate Peels Powder (PPP), Seeds powder of dates Ajwa (SPDA) and TiO_2 nanoparticles in individually form as reinforcement materials. For binary polymer blend (SR (VST-50F): 5% PMMA) were prepered in two part by mixing 95% wt from Part A of silicone rubber with 5% wt of acrylic powder (PMMA) by the vacuum mixer for 10 minutes as a part one. Then part B of silicone rubber and liquid monomer (MMA), were added to the base (part one) and mixed in the vacuum mixer for 5 minutes at speed of 360 rpm and under vacuum of (-10) bars. Then, putting the blend into the mold. Sample were left for 6 hour to vulcanization. For composites samples, the polymer blend (SR (VST-50F): 5% PMMA) were reinforced by pomegranate Peels Powder (PPP) with avg. diameter (102.45 nm), Seeds powder of dates Ajwa (SPDA) with avg. diameter (59.26 nm) and TiO_2 with avg. diameter (82.13 nm) with selected weight ratios (0, 0.1, 0.2, 0.3 and 0.4). Preparation of composites samples were carried out In the same practical methoed as in preparation the polymer blend sample mentioned above.

3. Characterization and Testing:

I. Test Methods

Fourier transform infrared spectrometer (FTIR) test is carried on according to (ASTM E1252) [15], made by (Bruker Optics Company, Germany), type is (TENSOR-27). FTIR was used to characterize the neat silicone rubber and binary polymeric blends samples (SR/PMMA). All tests were performed in according to (ADA Specification No.12, 1999), where all the test specimens after preparation and polishing processes was stored in distilled water at (37± 1℃) for 48 hr.

For density tests, the samples were prepared according to ASTM standard (D-792) [16-18] and samples weights were measured according to Archimedes method by accurate balances kind: PS 360/C/1device. The specific gravity can be determined by the definition shown in equation (1) [19]:

$$\text{Specific Gravity (S.G)} = \frac{W_D}{W_D - W_i + 0.02} \quad (1)$$

Where:
W_D: Mass of dry sample (gm.).
W_i: Mass of sample after immersing and suspended in water (gm).
0.02: Mass of practically immersed wire.
Specific gravity can be converted to density (gm/cm^3) by multiplying the specific gravity by D which represents the density of distilled water (gm/cm^3) that equal to (0.9975).
Water Absorption test is preformed according to (ASTM D570) [20,21]. In this test, the samples were immersed in the distilled water at room temperature for (24hr), then samples were removed from distilled water and weighed by digital balance. The water absorption can be calculated according to the following equation (2):

$$\text{Water Absorption } \% = \frac{W_S - W_D}{W_D} \times 100 \quad (2)$$

Where:
W_D: Mass of dry sample before immersion
W_S: Mass of sample after immersion in distilled water for (24 hr) at room temperature.
For thermal analysis test, this test was performed according to apparatus manual of standard specifications instrument [22]. Thermal properties test was carried out by using thermal properties test device, manufactured by (Kelthley), type (Transient Plane Source (TPS) - 500). The hot disk sensor is placed between two pieces of the same sample material. The values of thermal conductivity, thermal diffusivity and specific heat are read from the computerize gauge. The relationship between these properties is shown by the equation (3) [23-25].

$$D_{th} = K / (Cp.\rho) \quad (3)$$

Where:
D_{th}: Thermal diffusivity (mm^2/s).
Cp: Specific heat (heat capacity) at constant pressure (MJ/ m^{3}ºK).
K: Thermal conductivity (W/m.ºK).
ρ: Mass Density (Bulk Density) (kg/m^3).
For anti-bacterial Analysis, in this test 1000 µl suspension of each bacterium, which approximately contains 106 CFU (colony forming unit), was poured onto the surface of biofilm samples and were kept at 37°C with a relative humidity of 90%. After 24 hours, the bacterial solution was collected and transferred to 96-well culture plates. Bacteria counting were performed using OD method with ELISA, ELx808 at 600 nm.

4. Results and Discussion

I. FTIR Test

The FTIR a spectrum of neat silicone rubber (VST-50F) is shown in Figure (٤). In general, the absorption peak at 2962.78 cm^{-1} is assigned to stretching vibration of CH_3. The absorption peak at 1413.15 cm^{-1} is assigned to the rocking vibration of $–CH_2$-. The absorption peaks at 1258.50 cm^{-1} and 863.93 cm-1 are assigned to bending vibration and rocking vibration of Si-CH_3. The absorption peaks at 1009.28 cm^{-1} are assigned to the stretching vibration of Si-O-Si on backbone of silicone rubbers. The absorption peak at 787.O8 cm^{-1} is assigned to the coupling of stretching vibration of Si-C and rocking vibration of $–CH_3$ [26,27].

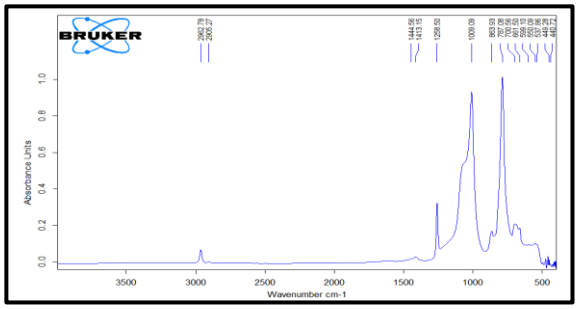

Figure 4: FTIR spectrum for neat silicone rubber (VST-50F) material

The FTIR spectra of polymers blend (SR (VST-50F): 5% PMMA) is shown in Figure 5. It can be seen from the infrared spectrum of polymeric blend specimen; these spectra are quite similar to the FTIR spectrum of neat silicone rubbers (VST-50F) which shown in Figure 4, no other new peak or peak shifts were observed for the polymeric blend of ((silicone rubber (VST-50F): 5% PMMA) specimen. This is due to the find physical bond and absence of any cross linking and chemical reaction between constituents of polymeric blend.

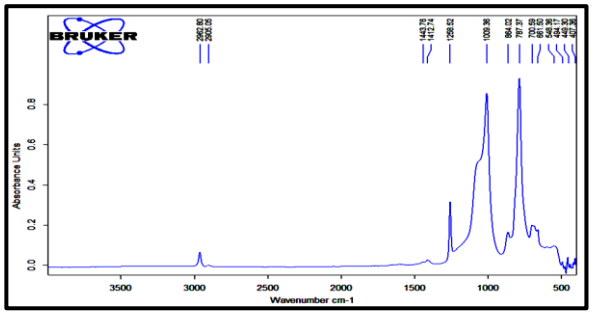

Figure 5: FTIR spectrum for polymeric blend (silicone rubber (VST-50F): 5% PMMA) specimen.

Figure 6 shows the FTIR spectrum of pomegranate peel powder (PPP). The spectrum confirmed the complex nature of these materials and proved the presence of wide variety of compounds. From this figure it can observed that

the spectra for PPP showed long bandwidth 3402 cm-1 which indicates the O-H stretching band confirms the presence of alcohols compounds and carboxylic acids. The C═C stretching band of alkyne group was detected at bandwidth 2929 cm^{-1}. The sharp mid-intense peak at 1726 cm^{-1} attributed to carbonyl group C=O which lead to presence of aldehydes, ketones and carboxylic acids. The moderate sharp peak at 1616 cm-1 indicates the presence of unsaturated compounds (alkenes). The band at 1338 cm^{-1} (CH_2 bending), related to the presence of cellulose, it can observe that the spectra of PPP is quite similar to that reported by [28,29].

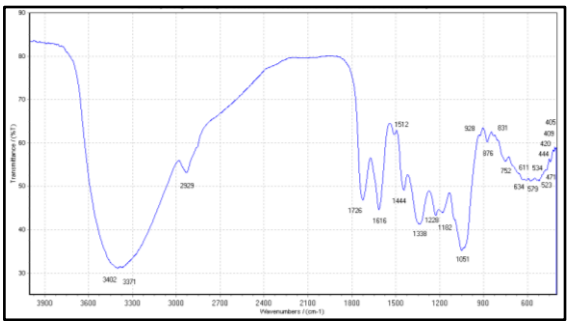

Figure 6: FTIR spectrum of pomegranate peels powder.

Figure 7 shows the FTIR spectrum of Seed powder of dates Ajwa (SPDA). From this figure it can observed that the FTIR spectrum of the Seeds powder of a dates Ajwa (SPDA) is very similar to the spectrum of pomegranate peels powder, since the two articles are from natural origin. The characteristics of the bands observed at 3431 cm^{-1} which indicates the O-H stretching band confirms the presence of alcohols compounds and carboxylic acids. The C═C stretching band of alkyne group was detected at bandwidth 2856 cm^{-1}. The sharp mid-intense peak at 1743 cm^{-1} attributed to carbonyl group C=O The moderate sharp peak at 1624 cm^{-1} indicates the presence of unsaturated compounds (alkenes). The infrared spectrum of SPDA is quite similar to that reported for many different natural compounds by [30,31].

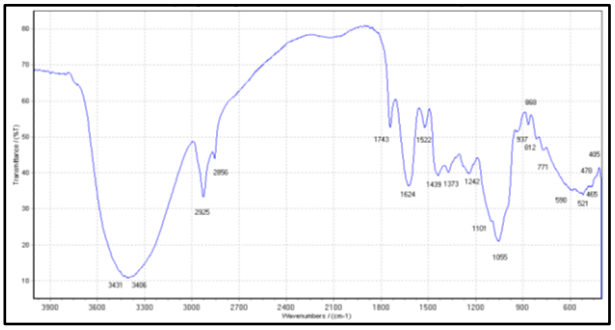

Figure 7: FTIR spectrum of Seed powder of dates Ajwa.

Figure 8 shows the FTIR spectrum of TiO_2. The peak around (3473 cm^{-1}) is due to the stretching mode of the O-H bond of free water. The peak around (1633 cm^{-1}) is due to the stretching mode of the O-H bond of chemisorbed water, peak at 1369 cm^{-1} related to Ti-O modes. The IR band at (424, and 1400 cm^{-1}) corresponds to the Ti-O and Ti-O-Ti stretching vibration mode in TiO_2 respectively. The titania nano-particle used in this work show an infrared bands around (694, 525 and 424 cm^{-1}) are associated to (Ti-O-Ti) stretching vibrations, all the characterize peak for these spectrum are similar to that reported by [32,33].

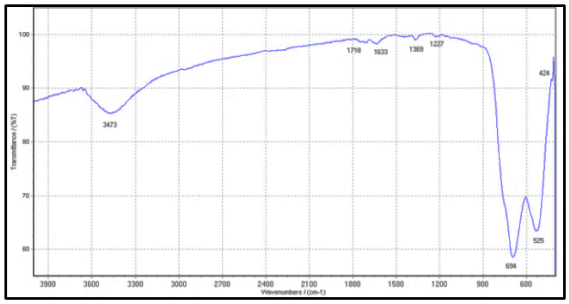

Figure 8: FTIR spectrum of TiO_2 powder.

Figures 9, 10 and 11 show the FTIR spectra for three groups of polymeric blends nano composites, which are [(SR (VST-50F): 5% PMMA): X%PPP], [(SR (VST-50F): 5% PMMA): X%SPDA] and [(SR (VST-50F): 5% PMMA): X% TiO_2]. It can be seen from the infrared spectrum of these group of polymeric blend composites specimens; these spectra are quite similar to the FTIR spectrum of neat SR (VST-50F) Figure 4 and polymer blend (SR (VST-50F):5% PMMA) Figure 5, no other new peak or peak shifts were observed for the polymeric blends of ((silicone rubber (VST-50F): 5% PMMA) specimens with the addition (PPP, SPDA and TiO_2). This is 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.

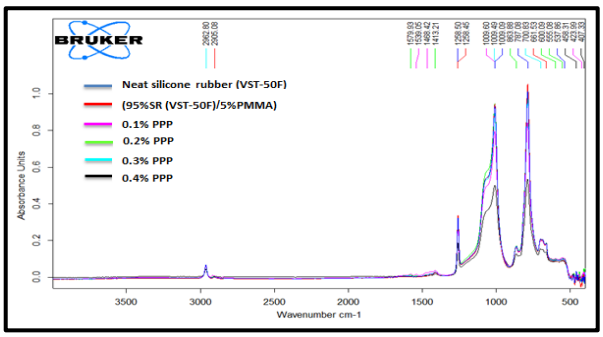

Figure 9: FTIR spectra for neat silicone rubber (VST-50F) and (95%SR (VST-50F) / 5%PMMA) polymer blend reinforced with Pomegranate peels powder (PPP).

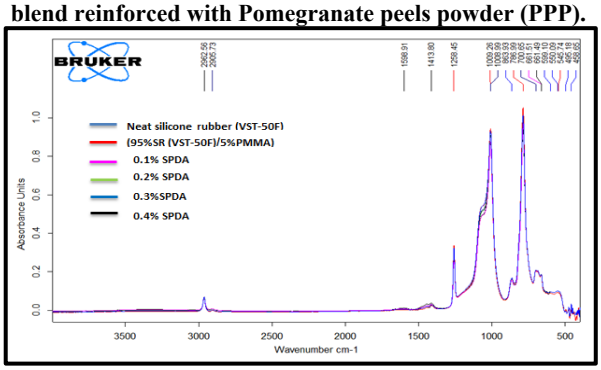

Figure 10: FTIR spectra for neat silicone rubber (VST-50F) and (95%SR (VST-50F) / 5%PMMA) polymer blend reinforced with Seed powder of dates Ajwa

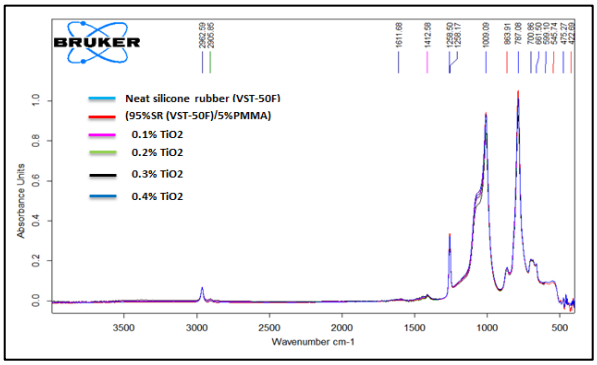

Figure 11: FTIR spectra for neat silicone rubber (VST-50F) and (95%SR (VST-50F) / 5%PMMA) polymer blend reinforced with Pomegranate peels powder (TiO_2).

II. Density property and water absorption test

The density and water absorption property of polymeric blends composites ((SR: 5% PMMA): X% nano filler) reinforced with different nano powder (PPP, SPDA and TiO_2) with ratios (0.0, 0.1, 0.2, 0.3 and 0.4 %) are shown in Figures 12, 13. For all three types of polymeric blends composites, the density decreased as compared with the matrix material of polymeric blends (SR: 5% PMMA) blend. It can be noted from this figure that the density values decreased with increased nanoparticles content in composite, and the least value reached to 1.0863 g/cm^3 at 0.4% ratio of pomegranate peels powder. While for seed powder of dates Ajwa and nano TiO_2 powder, the density reached to 1.095g/cm^3 and 1.093 g/cm^3, respectively at 0.4%. These materials may be in good compatibility [34].

From Figure 13 showing the relation between water absorption as function of nano powder content in composites, it was found that this property for all types of (pomegranate peels powder, seed powder of dates Ajwa and nano Tio_2) polymeric blend composites increased with increased nano powder percent. Polymeric blend composites with 0.4% percent of nanoparticles have a higher value of this property for all types of nano powder (PPP, SPDA and TiO_2) which reaches to 1.32%, 1.92%, and 2.11%, respectively [35, 36].

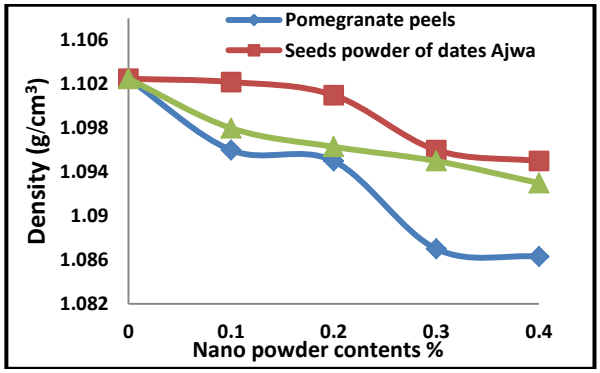

Figure 12: Density Test of polymer blend composite ((SR (VST-50F): 5% PMMA): X% nanofiller) as a function of (PPP, SPDA and TiO_2) Nano powders content in composites.

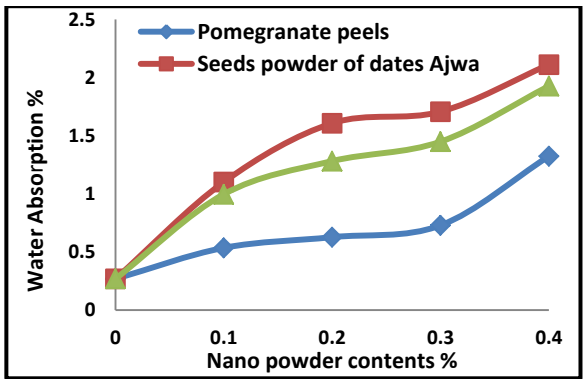

Figure 13: Water absorption of polymer blend composite ((SR (VST-50F): 5% PMMA): X% nanofiller) as a function of (PPP, SPDA and TiO_2) Nano powders content in composites.

III. Thermo-Physical Properties

The thermo-physical property (thermal conductivity values) of polymeric blend specimen (95%SR / 5% PMMA) and the addition of three types of nanopowder (Pomegranate peels powder, Seeds powder of dates Ajwa, TiO_2 Nanopowder) are illustrated in Figure 14.

The effect of the addition different ratios (0.1%, 0.2%, 0.3%, 0.4%) of nanopowder to polymeric blend (SR/PMMA) specimen on the thermal

conductivity of polymeric blend composites can be noted from the figure that the thermal conductivity of (Pomegranate peels powder, Seeds powder of dates Ajwa, TiO_2 Nanopowder) decreased reached to (0.1148, 0.1143, 0.1142 W/mk), respectively, with an increase of nanopowder contents in the polymeric blend composites [37].

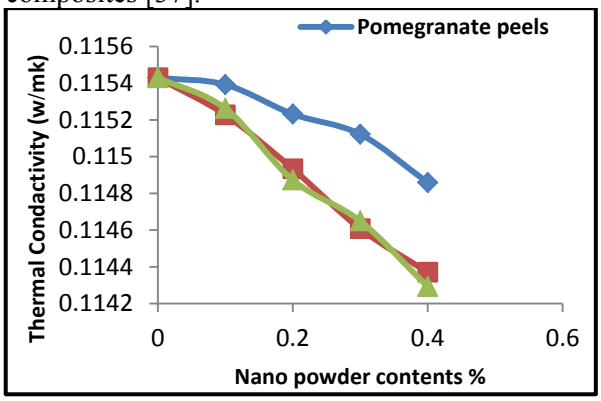

Figure 14: Thermal conductivity of polymer blend composite ((SR (VST-50F): 5% PMMA): X% nanofiller) as a function of (PPP, SPDA and TiO_2) Nano powders content in composites.

IV. Anti-Bacterial Test

Based on the foregoing results of the physical properties of polymers blends Nano composites, one sample was selected from these nano composites samples as the optimum ratio of polymer blend nanocomposite ratios, which is (SR (VST-50F): 5% PMMA): 0.1% TiO_2. This selected sample is then exposed to anti-bacterial test.

E. coli (Escherichia coli), is a type of bacteria that normally lives inside the body which can cause some disease. In Figure 15 show the relation between antibacterial efficiency of polymer blends composite for E.coli bacterial. From these figure, the results show that the antibacterial efficiency increased with adding 5%PMMA to (SR/PMMA) blend reaching to (51.4%) compared with pure silicone rubber which reached to (20.2%) respectively. Also, the antibacterial efficiency of polymeric blend composites reinforced by three type of nanopowder (PPP, SPDA and TiO2) improved reached to (42.21%, 43.81%, and 42.92% respectively) compare with that of pure silicone rubber. But stay it less than the antibacterial efficiency of (95%SR /5%PMMA).

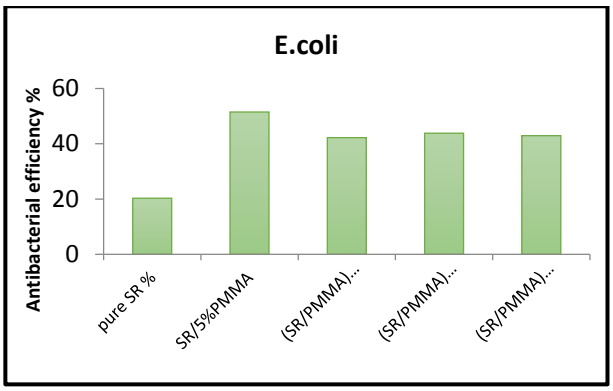

Figure 15: the effect of E.Coli bacterial on polymeric blend composites prosthetic for maxillofacial applications.

Staphylococcus aureus (S.aureus) found in the upper respiratory tract and on the skin. itcan cause a range of illnesses, from minor skin infections. Figure 16 shows the relation between antibacterial efficiency of polymer blends composite for S.aureus bacterial. From figure the results show that the antibacterial efficiency also increased with adding 5%PMMA to blend reached to (55.03%) compared with pure silicone rubber which reached to (24.70%). For polymeric blend composites reinforced by three type of nanopowder (PPP, SPDA and TiO2), the antibacterial efficiency was increased reached to (48.29%, 48.48%, and 50.54%), respectively compared with that of pure silicone rubber. Also, it stays less than the antibacterial efficiency of (95%SR /5%PMMA) [38,39]. But from result, it can be noticed that the antibacterial efficiency of polymeric blend composites to the S.aureus bacteria is more than that of antibacterial efficiency to E.coli bacteria.

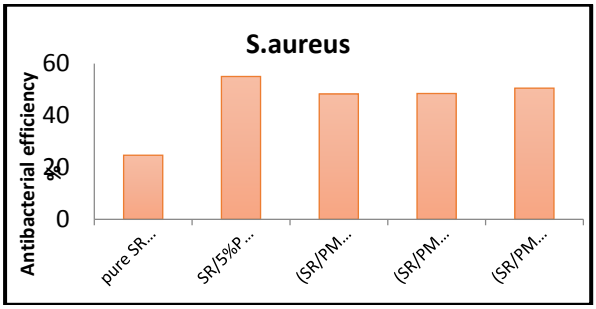

Figure 16: the effect of S.Aureus bacterial on polymeric blend composites prosthetic for maxillofacial applications

5. Conclusions

From the test results of the prepared polymeric blend nanocomposites with pomegranate Peels Powder (PPP), Seeds powder of dates Ajwa (SPDA) and TiO_2 nanopowder, it was concluded the following: -

1.The density property decrease after addition of pomegranate Peels Powder (PPP), Seeds powder

of dates Ajwa (SPDA) and TiO_2 nano filler to the base polymer blends, whereas the water absorption increase with addition these nanoparticles to base polymer blends.

2. The thermal conductivity of polymeric blend composites decreased reached to 0.1142 (W/mk) with an increase of nanopowder content in the polymeric blend composites.

3. At 0.2% of pomegranate Peels Powder, 0.3% of Seeds powder of dates Ajwa and 0.1% TiO_2, the antibacterial efficiency of polymeric blend composites improved compare with that of pure silicone rubber.

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