Physical Characteristics for Biocomposite Material Using to Jawbones Compensation

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Abstract. The biocomposite material prepared from polymeric blend (bone cement) as a matrix, and hydroxyapatite powder as a reinforcement material to jawbones compensation is a material that has a good compatibility in terms of physical properties with oral environment in general, and jawbones in particular. The physical properties such as density, water absorption, and thermal properties were tested according to the weight fraction for the reinforcement material. It was found that the density of biocomposite material does not uniformly change, and it decreases with increasing the ratio of hydroxyapatite particles. While, the water absorption increases with increasing the weight fraction of hydroxyapatite particles. Also, the thermal properties were measured. It was found that the thermal conductivity and the thermal diffusivity of the prepared material were decreased with increasing the weight fraction of hydroxyapatite particles, and therefore the specific heat of the prepared material was also increased due to the porous nature for reinforcement material (hydroxyapatite powder) that enhances the formation of voids between the resulting material.

Keywords: Biocomposite materials, jawbones, density, water absorption, thermal conductivity, thermal diffusivity, specific heat.

1. Introduction

During the last few years, composite materials were widely used in medical applications [1,2] to compensate the damaged parts of the body such as bones, denture base, teeth implants, and dental fillings, etc. [3]. Generally, jawbones like other types of bones are a specialized type of connective tissue [4]. It consists of cortical and cancellous bone [5]. The biocomposite materials that used to compensate jawbone must have specific properties to be suitable for human’s body especially for oral environment [6]. Biocomposite material for jawbone is a single composition with a uniform structure [7], and should satisfy many significant criteria as well as specific characteristics such as biocompatibility, osteoconductivity, corrosion resistance, strength, fatigue durability, and tight chemical similarity to biological apatite that exists in the human hard tissues [7,8]. Therefore, the preparation of any biocomposite material for jawbone compensation must have special biological, chemical, mechanical, and physical properties to be compatible with other body tissues [8,9].

In recent studies, the preparation of many biocomposite materials for compensating jawbones, biopolymer composite prepared from silicone rubbers were widely used for this purpose. Nanocomposites prepared from silicone rubbers as a matrix, and poly (methyl methacrylate) (PMMA)
as a reinforcement were succeeding for maxillofacial prosthesis and studied the effect of adding different percentage of nanopowder of PMMA on the physical properties [10]. The silicone rubber reinforced with hydroxyapatite powder (HAP) was used to compensate the damaged parts of the jaws [11]. The mechanical properties of biocomposite material were studied, and the PMMA was found to be a good biopolymer material to compensate human bones in general, and jawbones in specific [12]. PMMA as a denture base material was used, and it proved itself as success choice in this field due to its compatibility with oral environment and the mechanical and physical characteristics [13]. The objective of this work is to prepare a biocomposite material to repair the damaged parts of the jawbones and improves the performance of the jawbones using bone cement polymer reinforced with nanohydroxyapatite, and to study the physical properties of the prepared material.

2. Materials and Methods

2.1 Composition of Biocomposite Material

The biocomposite samples for jawbones compensation were prepared using (Hand lay-Up) method as a function of the specific weight ratios for all selected raw and reinforcement materials as shown in Table 1, and Table 2. Figure 1 shows the three types of reinforcement materials used in biocomposites.

### Table 1. Materials used to prepare biocomposite material.

| Materials                  | Symbol | Type   | The use   |
|----------------------------|--------|--------|-----------|
| Poly (methyl methacrylate) | PMMA   | Powder | Matrix    |
| Benzoyl peroxide           | BPO    | Powder | Matrix    |
| Barium sulphate            | BaSO₄  | Powder | Matrix    |
| Methyl methacrylate        | MMA    | Liquid | Matrix    |
| N, N-Dimethyl para-toluidine | N, N-DMPT | Liquid | Matrix    |
| Hydroxyapatite             | HAP    | Powder | Reinforcement |

### Table 2. Biocomposites materials and its chemical composition.

| HAP wt. % | PMMA | BPO | BaSO₄ | MMA | N, N-DMPT |
|-----------|------|-----|-------|-----|-----------|
| 0%        | 98   |     |       |     |           |
| 1%        | 96.5 |     |       |     |           |
| 2%        | 95   |     |       |     |           |
| 3%        | 93.5 |     |       |     |           |
| 4%        | 92   |     |       |     |           |
| 5%        | 90.5 | 0.294| 1.706 | 49  | 1         |
| 6%        | 89   |     |       |     |           |
| 7%        | 87.5 |     |       |     |           |
| 8%        | 86   |     |       |     |           |
| 9%        | 84.5 |     |       |     |           |
| 10%       | 83   |     |       |     |           |
2.2 Preparation of Biocomposite Material

Biocomposite material for jawbones compensation was prepared according to the amounts mentioned in Table 2. The amount of all components was (2:1) powder per liquid. It is necessary to pour the powder into the liquid not the opposite and mix them in a glass bowl with a thin wooden stick. Three groups of biocomposite materials were prepared with (nanohydroxyapatite, hydroxyapatite prepared from fish bones, and hydroxyapatite prepared from natural egg shells) as reinforcement materials at different weight ratios of (0, 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10)%.

By mixing the powder and the liquid, many changes will take place due to the solubility of a polymer in the monomer. The stages of mixing monomer and polymer acrylic materials include (sandy or granular, sticky, full dough, rubbery, and hard). The speed for the polymer and monomer mixture to reach the dough stage depends on the solubility of the polymer powder in the liquid monomer and increasing the temperature [14]. The components are mixed for 30 seconds and quickly poured into the molds cavities because the dough stage turned to hard stages quickly. A weight of approximately (2N) is putting above the prepared sample to obtain a smooth surface and to prevent the entry of gases vapor into the acrylic as well as the formation of bubbles during the curing process. In addition, the weight leads to coordinate the specimen molecules and the interference between them taking into account the lubrication of the mold of all directions to prevent the specimen adhesion. The prepared specimens are kept in the molds for 24 hours until the curing and polymerization processes for the acrylic specimens are completed. After the curing process was completed, the specimens were removed from the mold cavities with a very smooth upper and lower surface and polished with sandpaper to remove the small cracks resulted from the specimen's sides as a result of the specimen's adhesion with the mold cavity sides. All specimens were prepared and tested under the same conditions.

2.3 Physical Tests

The physical tests are carried out at room temperature 23±2˚C. All the tested specimens were stored in distilled water and heated at (37± 1˚C) for 48 hours.

2.4 Density Test

Density or (Bulk density) is the mass per unit volume of a material. Specific gravity is a measure of the ratio of the mass of a given volume of material at (23°C) to the same volume of distilled water. The specific gravity and density can be calculated from equations (1), and (2), respectively [15]:

\[
\text{Specific Gravity (S.G.)} = \frac{m_1}{m_1 + w - m_2}
\]
\[
\text{Density (\(\rho\))} = \text{Specific Gravity (S.G.)} \times 0.9975
\]

where:
- \(S.G.:\) Specific Gravity.
- \(m_1:\) Mass of a specimen in the air (gm).
- \(m_2:\) Mass of a specimen in the water (gm).
- \(w:\) Mass of partially immersed wire.
- \(\rho:\) Density (g/cm\(^3\)).
The density (g/cm$^3$) can be obtained by multiple specific gravities and the density of distilled water which is equal to (0.9975 g/cm$^3$) [15,16]. The specimens were prepared according to (ASTM D-792) standard [17], and the specimens weights were measured according to Archimedes method by examining the weight of the specimen in the air and then check the weight with water [18]. But, the volume should not be less than (1 cm$^3$). This test was done by accurate balances PS 360/C/1device, as shown in Figure 2.

![Figure 2. The density test device.](image)

2.5 Water Absorption Test
Various polymeric materials are susceptible to water absorption during its life exposures which cause dimensional instability. Thus, subjecting the material to degradation internal stresses may result in crack formation, and eventually lead to fracture or failure of the jawbones [19]. The moisture content of a polymer is very intimately related to such properties as mechanical strength, appearance, and dimensions stability. The effect upon these properties is the change in water absorption that depends largely on the type of exposure to high humidity, the rigidity of 3-D structure, the thickness of the specimen, immersion time, temperature, type of solution, type of materials, bounding type, the geometry of the specimen, cross-linking, curing method, and inherent properties of the polymer [20]. The water absorption can be calculated according to the following equation:

$$\text{Water absorption percentage} = \frac{(W_s - W_d)}{W_d} \times 100 \quad (3)$$

where:
- $W_s$: Mass of the specimen after immersion for (24 hours in distilled water).
- $W_d$: Mass of the specimen before immersion (dry).

For the most parts, the polymer may suffer expansions and contractions when alternately soaked in water and dried. This parameter is important in clinical applications of the matrix resin since water uptake may affect the dimensional stability, physical, and mechanical properties of jawbones. The water absorption by acrylic jawbone is nearly independent of temperature from (0 - 60 °C), and the thin specimens reach a steady-state within a short time [21].

The test of water absorption was performed according to (ASTM D570) [22]. The dimensions of the specimens are (L×W×H) (50 × 50 × 4) mm. Figure 3 illustrates the experimental specimens and standard dimensions of the specimen for the water absorption test.

![Figure 3. Water absorption test: (a) Experimental specimens; (b) Standard dimensions of the specimen for water absorption test.](image)
2.6 Thermo-Physical Test

To evaluate the thermal properties of specimens, Hot Disk thermal analyses were used. The Transient Plane Source (TPS) technique was used to determine the (thermal conductivity, thermal diffusivity, and specific heat). The Hot Disk includes a heat source, and temperature probe as a flat sensor with a continuous double spiral of electrically conducting nickel (Ni) metal with a resistance of (11.56 Ω) to transform the thermal power which passes through the specimen to measure thermal transport properties, etched out of thin foil, sandwiched between two layers of insulating material. During the test, the sensor was normally placed between the surfaces of the two pieces of the specimen to be measured [23,24]. Measurements can be performed on many different materials; solids, liquids, powders, viscous materials, composites, etc. including various types of geometry, and dimensions. The Hot Disk (TPS) can also be used under various environmental conditions from very low temperature (-45°C) up to (1000°C) [25].

The relationship between the thermal properties is illustrated by [25,26]:

\[
D_{th} = \frac{K}{C_p \rho}
\]

where:

- \(D_{th}\): Thermal diffusivity (mm\(^2\)/s).
- \(K\): Thermal conductivity (W/m.K).
- \(C_p\): Specific heat at constant pressure (MJ/m\(^3\).K).
- \(\rho\): Mass density (Bulk density) (kg/m\(^3\)).

This test was performed according to the apparatus manual of standard specifications instrument [27]. The test was carried out by using the thermal properties test instrument, manufactured by (Kelthley), type (Transient Plane Source (TPS) - 500). Figure 4 (a) illustrates the instrument used for the thermo-physical properties test. This apparatus was used for investigating the thermal transmission properties, such as thermal diffusivity (\(C_v\)) or (\(D_{th}\)), volumetric specific heat (\(C_p\)), and thermal conductivity (K) which were determined by utilizing the Hot Disk sensor. A hot disk sensor was placed between the two pieces of the same specimen material prepared at the same dimensions of the standard specifications instrument, which are a least as thick as the radius of the sensor, functioning mechanical support, and electrical insulation as shown in Figure 4 (b), and then heated by an electrical current for a short period of time [28]. The lavished heat generates a temperature rise of both the sensor and the surrounding specimen material. To avoid the influence from the outside boundaries of the specimen, the specimen should be larger than the sensor diameter to ensure stable values of both thermal diffusivity, and conductivity. The values of thermal diffusivity, thermal conductivity, and specific heat were taken from the computerized gauges.

![Figure 4. Thermo-physical properties test: (a) Thermal properties test instrument; (b) Hot disk sensor operation.](image-url)
3. Results and Discussion

3.1 Density Property

From important measurements, the density of biocomposite material is the basic indicator for knowing the lighter prepared biocomposite materials [15]. Generally, the biocomposite materials have a lighter weight than biometals or bioceramic. This is one of the main reasons why using biocomposite materials in vivo applications [29].

The density of biocomposite specimens in this study is measured according to the Archimedes method using an equation (2). Figure 5 illustrates the relationship between the density, and the weight fraction of the reinforcing particles (HAP powder), which were added to the polymeric matrix (bone cement). It can be seen from the curve that the density values for biocomposite specimens increase with increasing weight fraction of HAP powder to the mixture (polymeric matrix) because the density of HAP particles is higher than that of PMMA [30,31].

Generally, prepared specimens had different densities, where the density for specimens has not constant change in proportion with the weight fraction of the reinforcement material (HAP powder) because other influential factors such as no homogeneity through the preparation process may affect the density of the specimen.

Pure PMMA has a density equals to (1.2 g/cm$^3$) [31], and MMA has a density equal to (0.93 g/cm$^3$) [32]. Other components of the matrix also have specific densities, while the density of the reinforcement (HAP powder) is (2.62 g/cm$^3$) [30]. The density for prepared specimens was between (1.32-1.91) g/cm$^3$, and the lower density was found at (2%) of (n-HAP) and (3%) of (HAP prepared from egg shells and fish bones). The higher density was found at (9%) of (n-HAP) and (10%) for (HAP prepared from egg shells and fish bones), respectively. These results differ from the results in reference [13] due to the difference in the reinforcement material, while it is in good agreement with the results in reference [10].

![Figure 5. Density as a function of weight fraction of the reinforcement particles (HAP powder) in biocomposite material.](image)

3.2 Water Absorption Property

The relationship between the water absorption of biocomposite material and reinforcement material HAP particles is illustrated in Figure 6. The water absorption values of the polymeric matrix and biocomposite material with HAP particles for all specimens that fabricated in this work are illustrated in figure 6. It can be observed from the figure that the water absorption decreases with increasing the weight fractions of HAP particles. This is due to the attitude of the particles towards the moisture, and also may be due to the porosity nature of HAP particles. Therefore, the water absorption is very little. In previous study, HAP particles were used as reinforced material with silicone rubber as a matrix, and the water absorption was not found [11]. Also, PMMA powder was used as a matrix with (bamboo, and siwak) fibers as a reinforcement, and it found that the water absorption increases with increasing the weight fraction of fibers and fiber's length [10].
In general, biocomposite specimens with HAP powder graded of absorption for water-dependent on the weight fraction of reinforcement, as well as the specimens with lower density have higher water absorption. The density decreases with increasing the moisture absorption [33]. Polymeric matrix (bone cement) specimen without reinforced material have a high value of water absorption equal to (1.443%), while biocomposite specimens with HAP particles have a low value reaches (1.1%) for a specimen with weight fraction (10%). This increase in water absorption is interpreted as a result for the existence of a bioceramic material (HAP) in the prepared biocomposite material, and these results are identical to the results in reference [34].

![Figure 6. Water absorption as a function of weight fraction of the reinforcement particles (HAP powder) in biocomposite material.](image)

### 3.3 Thermo-Physical Properties

The thermal-physical properties (thermal conductivity, thermal diffusivity, and specific heat capacity) are properties obtained from the hot disk test for biocomposite specimens at room temperature. The relationship between these properties is given by equation (4) which illustrates decreasing the thermal conductivity and improving the specific heat to decrease the thermal diffusivity of specimens [35].

**Thermal Conductivity Results:** Thermal conductivity for biocomposite material represents the thermal energy that causes movement of the molecular chains of polymer in biocomposite specimens at a rate proportional to the weight fraction of the conductive materials [36]. Thermal conductivity results are illustrated in Figure 7.

This figure illustrates that when increasing the weight fraction of HAP particles, the thermal conductivity of the biocomposite specimens decreases. This is due to the role of HAP particles that have thermal conductivity lower than that for PMMA matrix. Also, the presence of bioceramic HAP particles of a porous structure and conglomerate of biocomposite specimen as a result of increasing the weight fracture from HAP powder that lead to the difficult process of heat transfer through the composite specimens, and can considerably poor thermal conductivity. This is made better molds for the characterization of bone cement thermal behavior [37].

Figure 7 illustrates the polymeric matrix (pure) has a higher thermal conductivity value equal to (0.1622 W/m.K), while the biocomposite specimens have lower values than the polymeric matrix. The maximum values of thermal conductivity for biocomposite specimens with (n-HAP, HAP prepared from egg shells and HAP prepared from fish bones) particles at the optimum condition of weight fraction (1%) equal to (0.1604 W/m.K), (0.1525 W/m.K) and (0.1551 W/m.K), respectively, and have minimum values at (10%) (0.0918 W/m.K), (0.0645 W/m.K) and (0.0775 W/m.K) respectively. The biocomposite specimens with less weight fraction of HAP have higher thermal conductivity value than biocomposite specimens with high rates.
Thermal Diffusivity Results: The thermal diffusivity of specimens indicates the change in the composite specimen’s temperature in the test when the heating is applied [38]. Figure 8 shows the relationship between the thermal diffusivity and the weight fraction of the reinforcing HAP particles added to the matrix (bone cement). The thermal diffusivity of the composite specimen decreased with increasing the weight fraction of (n-HAP) reinforcing particles added to the matrix (bone cement). The pure matrix (bone cement) has a higher value of thermal diffusivity than the composite specimen reaches (0.06182 mm$^2$/s). So, the higher values of biocomposite specimens reinforced by weight fraction equal (1%) reach (0.0602 mm$^2$/s, 0.0515 mm$^2$/s and 0.0549 mm$^2$/s) for composite specimens having (n-HAP, HAP prepared from egg shells and HAP prepared from fish bones) particles, respectively. While, composite specimens have thermal diffusivity decreased with increasing weight fraction of HAP particles. These results differ from those obtained for composite specimens PMMA reinforced with fibers (bamboo and siwak) which show that the thermal diffusivity of specimens is increased with increasing the weight fraction and the fiber length of (bamboo and siwak). In addition, siwak specimens have higher values of thermal diffusivity than bamboo specimens [10].
Figure 8. Thermal diffusivity as a function of the weight fraction of the reinforcement particles (HAP powder) in biocomposite material.

Specific Heat Results: The specific heat for biocomposite specimens represents the energy required to raise the temperature $T_g$ of biocomposite specimens by one degree [36]. Figure 9 illustrates the relationship between the specific heat, and the weight fraction of the reinforcing particles (HAP powder) added to a pure matrix (bone cement). Specific heat magnitudes could be increased by increasing the weight fraction of HAP particles compared with the magnitude of PMMA alone. Therefore, the highest values of specific heat for biocomposite specimens are obtained for biocomposite with (10%) weight fraction of HAP particles. This could be attributed to the formation of spaces and voids between the particles because of the porous nature for HAP particles, which consequently led to an increase in specific heat. The pure matrix (bone cement) has lower value than biocomposite specimens with reinforcing HAP powder that equal to (2.319 MJ/m$^3$.K).

The highest values of specific heat were found with biocomposite specimens at (9%) weight fraction of (n-HAP) particles, and (10%) of (HAP prepared from egg shells and HAP prepared from fish bones), that reach to (3.246 MJ/m$^3$.K), (3.59 MJ/m$^3$.K), and (3.36 MJ/m$^3$.K), respectively. This is different from composite specimens with (bamboo & siwak fibers) that have a lower value of specific heat than pure PMMA [10].

Figure 9. Specific heat as a function of the weight fraction of the reinforcement particles (HAP powder) in biocomposite material.

4. Conclusions
The experimental physical tests used for the biocomposite materials prepared in this study led to the following conclusions:

The biocomposite material prepared from biopolymer (bone cement) as a matrix and reinforced by bioceramic (HAP powder) as filler is a material with good compatibility, as the physical properties of the prepared material are suitable with the jawbone material in particular, and with the body in general.

The prepared biocomposite materials have different densities, where the density for prepared specimens lay between (1.32-1.91) g/cm$^3$. Therefore, the density of these samples increases with the weight fraction of HAP powder.

The rate of water absorption of biocomposite specimens with HAP particles is grading dependent on the weight fraction of reinforcement, where polymeric matrix (pure) specimen without reinforcement have
the higher value of water absorption, while biocomposite specimens with HAP particles have lower values, and decreases with increasing the weight fraction of HAP powder. The maximum values of thermal conductivity and diffusivity of the biocomposite specimens are at a weight fraction less than the HAP powder, and the minimum values of them at the weight fraction higher than it.

Specific heat is inversely proportional to both thermal conductivity and diffusivity, so, it increases with increasing the weight fraction of the reinforcement material.

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