Egypt. J. Chem. **Vol. 64**, No. 2 pp. 859 - 867 (2021)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

The Mechanical Properties of Biopolymer Composite Material Using in Compensation Jaws bones

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Abstract

The aim of this paper is to prepare biopolymer composite material used to compensate for the jaws bones, where the characteristics of poly(methyl methacrylate) resin were developed by addition hydroxyapatite powder as a reinforcement material and studying the effect of selected weight fractures (1,2,3,4,5,6,7,8,9, and 10) wt.% to the polymeric blend (bone cement) on the tensile strength, maximum stress, modulus of elasticity and elongation percentage at break, and before that microstructure analysis was studied by using scanning electron microscope (SEM). The strength results are consistent with the (SEM) images that illustrated significantly increased agglomerates with increasing amount of hydroxyapatite powder. The results showed that the values of these properties increase at specific percentages of hydroxyapatite powder, while decreasing with increasing these percentages to a certain limit. The highest tensile strength value, elastic modulus and best elongation percentage at break were found in composite samples (45.159 MPa at 3%, 2.7365 GPa at 3%, 1.187 mm at 6%) respectively.

Key words: Biocomposite materials; Poly (Methyl methacrylate); Jaws bones; Hydroxyapatite; Tensile characteristic.

1. Introduction

The defects of the jaws bones were and still, whether genetic defects, pathological defects or defects caused by accidents are the attention of researchers, so the preparation of compensable biomaterials to treatment these defects is necessary and very important. Many biomaterials used in this field, particularly composite materials [1], where this type of biomaterial was adopted for its characteristics in terms of light weight, high durability and low cost etc. [2].

A composite material is composed primarily of two parts (two phases) that a matrix material, i.e. a continuous phase, which is armored with a reinforcement material (reinforcement is a secondary phase), which is usually the discontinuous phase [3,4].

Composite materials classification dependent on the matrix or reinforcement materials type, where the composites are classified based on the matrix material as polymer, metal, ceramic and carbon matrix composite, while depending on the material of reinforcement as fibers, filled, whiskers, flake, particulates and directionally solidified eutectics [5,6]. In addition to this, it is very important that the biocompatibility of the material used is chosen as biocompatible materials are compatible with the human body in general and with the oral environment in particular. The most common types of composite materials for compensation jaws bones are polymer matrix composite materials [7]. Poly (methyl methacrylate) is a polymeric material that is biocompatible with the oral environment, where it based resins are most widely used resins in dentistry, especially in fabrication of dentures and orthodontic appliances [8]. Hydroxyapatite is the bioceramic

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Receive Date: 07 June 2020, Revise Date: 09 August 2020, Accept Date: 02 October 2020
DOI: 10.21608/EJCHEM.2020.32055.2681

material most frequently used in orthopedics, because it is bioactive and thus, it promotes bone growth into available porosities [9], adding hydroxyapatite powder to the poly (methyl methacrylate) lead to an increase the biocompatibility and osteoblast reaction through direct reinforcement of osseointegration [10]. As well as, there are many materials used as reinforcement materials in biomedical field, such as titania [11], magnesia is another bioactive material and suitable additive to Poly (methyl methacrylate) [12], hydroxyapatite, magnesia and titania particles were also used as reinforcement by incorporation and added to poly (methyl methacrylate) [13]. Silicone rubber reinforced with hydroxyapatite powder was use with different percent from (nano and micro hydroxyapatite) powder to prepare composite material to compensation the damaged parts of the jaw bone [14], as polymer blends prepared (silicone rubber/ poly (methyl methacrylate)) reinforced with nanopowders (pomegranate peels powder, seeds powder of dates Ajwa and titanium oxide) was used at different percent to replace the facial parts of maxillofacial that lost through disease or trauma [15]. The objective of this present work is preparing biocomposite material used to reparation the damaged parts of the jaws and improves the performance of facial and maxillofacial prostheses by using bone cement polymer blends as matrix of polymers reinforced by hydroxyapatite and study the mechanical properties for the prepared material.

2. Materials and methods

2.1. Used materials

To prepare biocomposite material use as prostheses parts for jaw bone compensation, that need to matrix and reinforcement materials, as the following:

i. Matrix (Poly (methyl methacrylate) PMMA, Benzoyl Peroxide BPO and Barium Sulphate $BaSO_4$) powders, (Methyl Methacrylate MMA and N, N-Dimethyl Para-Toluidine N, N-DMPT) liquids.

ii. Reinforcement (Hydroxyapatite HAP) Powder.

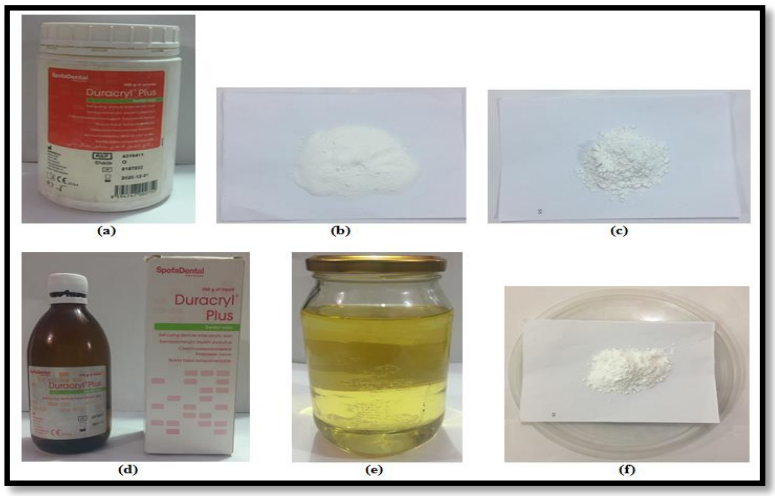

Fig. 1. The used materials in the composite material: (a) Poly (methyl methacrylate); (b) Benzoyl peroxide; (c) Barium sulphate; (d) Methyl methacrylate; (e) N, N-Dimethyl para-toluidine; (f) Hydroxyapatite powder.

Figure (1) represents the used materials in biocomposite material as matrix and reinforcement, where figure (a), (b) and (c) show the powder materials, while (d) and (e) illustrate the liquid materials, all that as matrix, whilst figure (f) represents reinforcement material (hydroxyapatite powder), in addition, the chemical structure for all components of matrix and reinforcement materials were illustrated in table (1).

Table 1: Chemical structure for used materials to prepare biocomposite material

Materials	Symbol	Chemical Structure	References
Poly (methyl methacrylate)	PMMA	$(C_5O_2H_8)_n$	[16]
Benzoyl peroxide	BPO	BPO	[17]
Barium sulphate	$BaSO_4$	$BaSO_4$	[18]
Methyl methacrylate	MMA	$C_5H_8O_2$	[16]
N, N-Dimethyl para-toluidine	N, N-DMPT	$CH_3C_6H_4N(CH_3)_2$	[19]
Microhydroxyapatite	m-HAP	$Ca_{10}(OH)_2(PO_4)_6$	[20]

Poly (methyl methacrylate) PMMA also known as acrylic is a transparent thermoplastic [21]. Currently, the acrylic resin (PMMA) is used almost universally for denture base fabrication [22]. PMMA is classified as a hard, rigid, but brittle material, with a glass transition temperature of 105°C [23]. The physical properties of the final polymer are important in the medical applications [24]. The density of acrylic ranged between 1.17-1.20 g/cm^3 which is half less than that of glass. The impact strength of PMMA is greater than that of glass and polysterene [21]. PMMA or acrylic has good mechanical strength [21,23], acceptable chemical resistance and extremely good weather resistance [23], PMMA is lightweight material, therefore, it is often used in sheet form due to its properties such as lightweight and shatter resistance as an alternative to glass [21]. PMMA has favorable processing properties, good thermoforming and can be modified with pigments, flame retardant additives, UV absorbent additives and scratch resistant coatings [23].

Acrylic also has good compatibility with the human tissue [21]. Major factors affecting the thermal, physical and mechanical properties of particulate (i.e., filler) reinforced polymer composites include the polymer structure and molecular weight, the filler type and volume fraction, and the interfacial state between the filler and the polymer [25]. Methyl methacrylate liquid (MMA) is the monomer, that used with the polymer (PMMA) as matrix material [26].

Benzoyl peroxide (BPO) is an odorless, white or colorless crystalline powder. It is using in component bone cement, addition for the polymer powder PMMA, where it represent initiator of the radical polymerization [17].

N, N-Dimethyl para-toluidine (N, N-DMPT) is the accelerator in the redox initiator-accelerator system used commercially to cure methyl methacrylate monomers [19].

Barium sulphate ($BaSO_4$) using in components bone cement to be easily identifiable radiographically [18].

A hydroxyapatite is known as one of vital materials and common use in biomedical field and concentrated in clinical area [27]. Biocompatible and their bioactivity can strengthen bone-bond formation with other tissues through an osteoconductive mechanism [28]. In bones, the minerals are mainly deposited in the form of calcium phosphate compounds with the great majority existing as apatite and only a small amount of them are carbonate containing apatites [29]. Calcium phosphate based biomaterials have received great interest since the main constituent of inorganic mineral component in human bone is hydroxyapatite (HAP) [30].

2.2. Preparation of Biocomposite Material Specimens

2.2.1. Composition Rates of Biocomposite Material

The biocomposite specimens for jaws bones material were prepared according to the relevant ASTM standard for each test by utilizing (Hand lay-Up) method according to the specific weight ratios for all components of matrix and reinforcement materials of polymeric blend samples that were selected, as in the table (2).

The weight ratios mentioned in the below table were applied with microhydroxyapatite as reinforcement materials, that were prepared from egg shells, where the particle size for m-HAP filler laying between 0.77 µm and 24.67 µm with a mean size of 5.005 µm.

Table 2:Composition of biocomposite material that prepared in this study

Materials	Powder				Liquid	
	HAP wt.%	PMMA wt.%	BPO wt.%	$BaSO_4$ wt.%	MMA wt.%	N, N-DMPT wt.%
Ratios	0%	98%	0.294%	1.706%	49%	1%
	1%	96.5%				
	2%	95%				
	3%	93.5%				
	4%	92%				
	5%	90.5%				
	6%	89%				
	7%	87.5%				
	8%	86%				
	9%	84.5%				
	10%	83%				

2.2.2. Steps to Prepare Biocomposite Material

To prepare biocomposite material (bone cement) for jaws bones compensation, the following steps were carried out:

- In this study, the used amount of powder per liquid for all components was (2:1) (wt./wt.) (powder/liquid). Therefore, the amount PMMA acrylic resin required per the weight of the liquid monomer resin (MMA) was also (2:1) (wt./wt.) (PMMA powder/MMA liquid), as in the previous table, while the other components with the weight ratios that mentioned.
- Prepare specimen polymeric blend (bone cement) without hydroxyapatite powder, by adding the mixture of the powder components to the liquid components gradually.
- The stages in mixing monomer and polymer acrylic materials include (sandy or granular, sticky, full dough, rubbery and hard). The speed with which the polymer and monomer mixture reaches to dough stage depends upon the solubility of the polymer powder in the monomer liquid and increasing the temperature [31]. It is necessary to pour the powder into the liquid and not the opposite. As well as the mixing in glass bowl with a thin wooden stick.
- Mixing the components at time 30 seconds, and mold the mixture in the mould cavity with high speed, because the dough stage reaches for the rubbery and hard stages quickly, after pressed by using cover with size similar to the size of the mould cavity, to obtain smooth surface and to prevent gases vapor entry into the acrylic during the curing process. As well as put the weight of the amount (6 N) to the above sample.
- To prepare other specimens, liquid monomer (MMA) and reinforcement particles (m-HAP) were be mixed together at room temperature, continuously and homogeneously to make sure of homogeneity of the mixture, after repeat the same previous steps to produce biocomposite material.
- The prepared specimens leave in the moulds for 24 hours to curing process and to complete polymerization process of acrylic specimens.
- After the curing process was completed, the samples were remove from the mould cavities with very smooth upper and lower surface. Then they were finished using special hand grinder to remove the cracks from the specimen's sides as a result of the specimen's adhesion with the mould cavity sides.
- All specimens were prepared and tested under the same conditions at room temperature 23±2˚C.
- All the test specimens after preparation and polishing processes must be stored in distilled water at (37± 1℃) for 48 hours.
- Figure (2) illustrates the standard sample, the mould and the specimens before and after test.

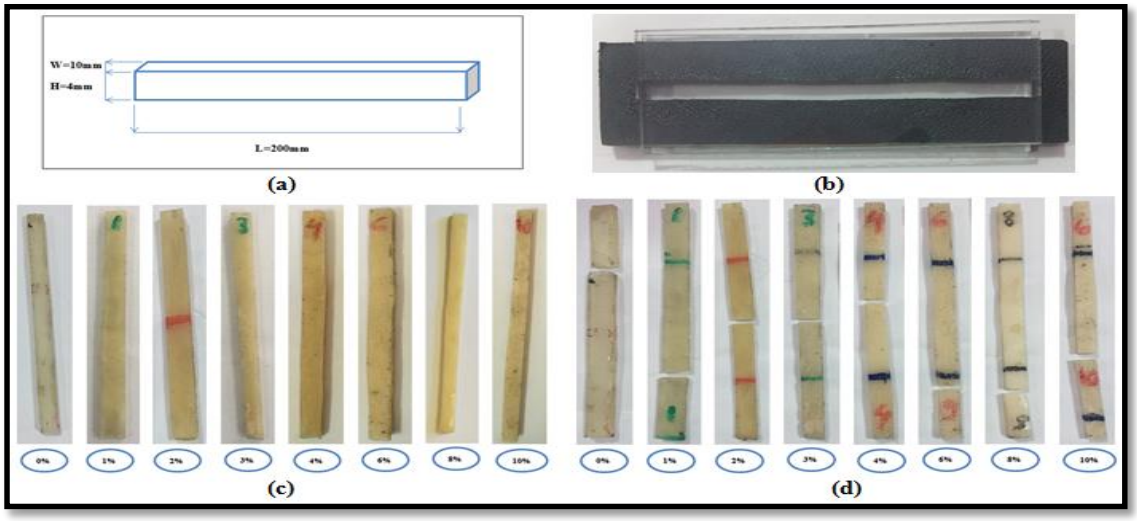

Fig. 2. Tensile test: (a) Standard dimensions of the sample; (b) The used mould; (c) Some of specimens before test; (d) Some of specimens after test.

3. Results and discussion

3.1. Morphological analysis

In order to correlate the mechanical properties with the fracture surface morphology of the bone cement as polymeric blend and biocomposite specimens, as a function of (hydroxyapatite particles) at different weight fractions content in composites. Scanning electron microscopy SEM micrographs were done to the tensile tested fractured surface for the polymeric blend (bone cement) and some of biocomposites specimens with different weight fractions as in figures (3), (4), (5), and (6), respectively.

The morphology of polymer biocomposite material depends on the processing conditions, nature components, weight fractions, and component melt viscosities. The fractured surface morphology of the polymeric blend specimens, as illustrated in figure (3), in the most case, it appeared as a homogeneous morphology, some agglomerations of hydroxyapatite powder material can be observed with the reinforcement material additions, the agglomerations increase in the biocomposite materials with increase the weight fracture of the reinforcement material, because of the porous nature of the hydroxyapatite powder, with a small percentage of the hydroxyapatite powder, powder particles overlap between polymer molecules, with higher concentrations of hydroxyapatite powder, the distribution of inorganic phase in the matrix where precipitates are closely spaced so forming a semicontinuous network along grain boundaries. It can be clearly seen with increasing amount of bioceramic particles that these particles are located in the grain boundaries of polymer blend as the irregular atoms arrangement at grain boundary provides lower atomic packing and high energy. Atoms are thus able to spread more rapidly to form precipitates. This high energy makes the grain boundary more "chemically reactive" than the grain itself, therefore, boundaries are an ideal position for both nucleating and growth of precipitates, this corresponds to reference [13].

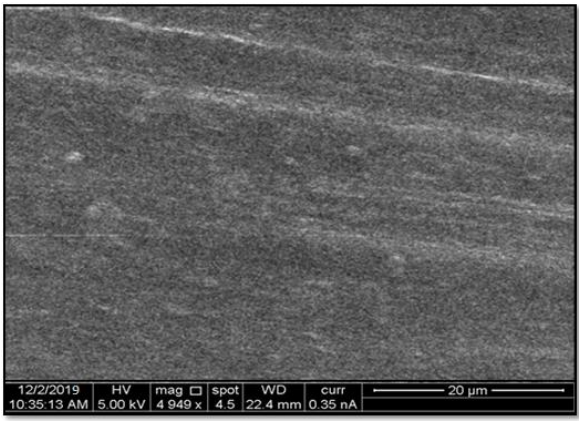

Fig. 3. Scanning electron microscopic image for polymer blend (bone cement).

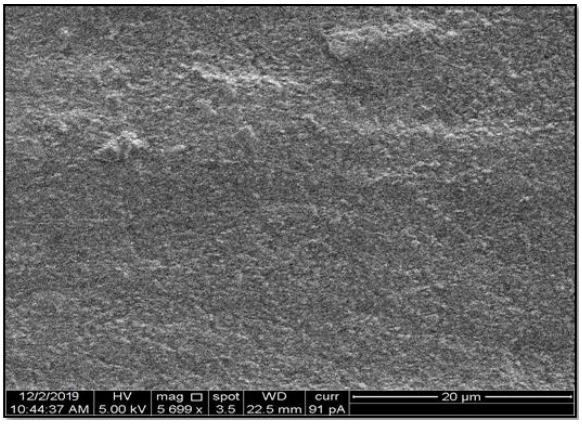

Fig. 4. Scanning electron microscopic image for biocomposite material with (3%) of (HAP).

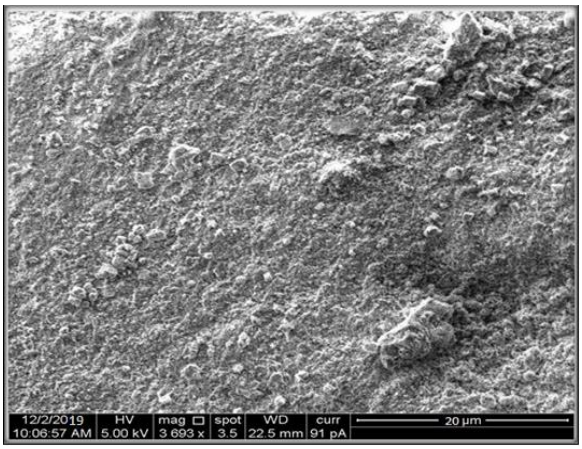

Fig. 5. Scanning electron microscopic image for biocomposite material with (6%) of (HAP).

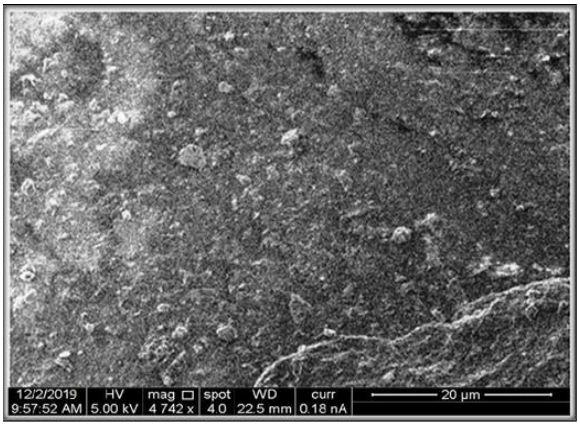

Fig. 6. Scanning electron microscopic image for biocomposite material with (9%) of (HAP).

3.2. Tensile test

This test was performed according to the international standard (ASTM D638-87b) [32]. Tensile test is performed on the specimens to obtain the (load–elongation) curves then from which the (stress–strain) curves are plotted. The ultimate tensile strength, tensile modulus and elongation percentage at break are obtained from this test.

The calculation of tensile strength, elastic modulus and poison ratio can be done by the following equations [33]:

$$\text{Tensile strength } (\sigma) = \frac{F}{A} \quad \ldots. (1)$$

$$\text{Elastic modulus } (E) = \frac{\Delta\sigma}{\Delta\varepsilon} \quad \ldots. (2)$$

$$\text{Poison ratio } (v) = \frac{\Delta\varepsilon_t}{\Delta\varepsilon_a} \quad \ldots. (3)$$

Where: F: maximum load; A: cross section area; $\Delta\sigma$: difference in stress; $\Delta\varepsilon$: difference in strain; $\Delta\varepsilon_t$: difference in transverse strain; $\Delta\varepsilon_a$: difference in axial strain.

3.2.1. Stress-strain curve

The (stress-strain) curves of polymer blend and the composite specimens reinforced with microhydroxyapatite powder prepared from egg shells at different weight percent of (1,2,3,4,6,8 and 10) wt.% are presented in figure (7). The (stress-strain) curves show difference in the behavior due to reinforcement particles, where the stress for the biocomposite increase with a little percentage of hydroxyapatite powder, after the stress decrease with high percentage of it's in the biocomposite material, where this corresponds to reference [13].

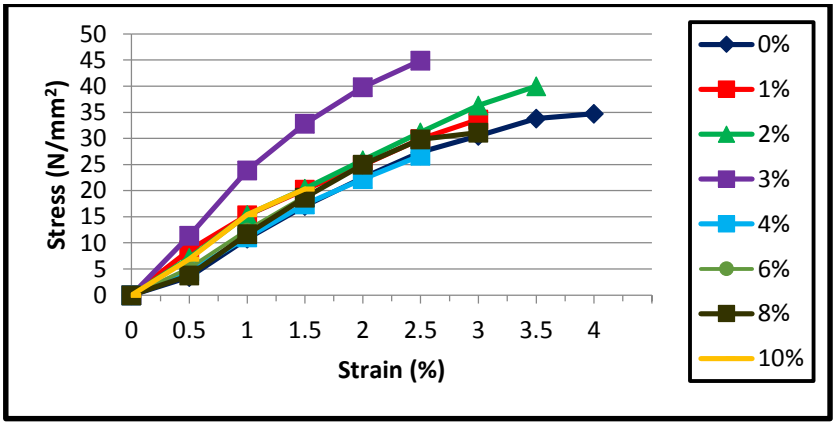

Fig. 7. Stress-Strain curve for polymer blend and composite specimens with (1,2,3,4,6,8 and 10) wt.% hydroxyapatite powder.

The essentially deformation in the prepared composite material due to the reinforcement powder, because the conglomeration of the reinforcement particles in the polymer blend and the particle size of this filler, all of these lead to the failure stress-stain curve.

3.2.2. Tensile strength

In the case of adding small quantities from m-HAP powder, the tensile strength increases as in figure (8) due to the large surface area of HAP powder, so the contact strength between them and the polymer chains increases and gives it more strength than in the case of high ratios where m-HAP powder work as filler to fill the voids between the polymer chains and give a hardening structure, where the microparticles are agglomerated and difficult to move on one side

more than on the other side so the area of contact with the polymer decreases and it is difficult to distribute them between the chains, which are clusters and conglomerates that lead to form defects, and the beginning of cracking will weaken its tensile strength increased percentage of m-HAP powder.

The composite specimens with reinforcing particles (hydroxyapatite) have tensile strength at specific limit than polymer blend (bone cement), after the observed failure in tensile strength with increasing the percentage of the (HAP) powder. This corresponds to the references [13,34], where increased content of inorganic particles lead to reduce cement strength, the reduction in strength due to reinforcement particles forming agglomerates. Particle agglomeration is likely to decrease the load resistance of the composite, even though the ceramic additives were strong enough to raise the modulus. Agglomerates constitute weak areas, so with load application and stress transfer to these weak areas, the bonds linking the agglomerated particles break and the stress is transferred back to matrix initiating cracks and stress concentrations and eventually leading to cement failure [13,35].

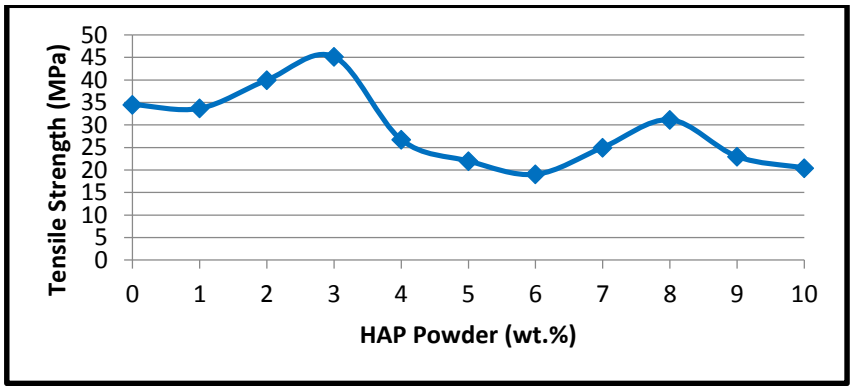

Fig. 8. Tensile strength and weight ratio of hydroxyapatite powder for composite specimens.

3.2.3. Modulus of elasticity

Figure (9) show the relationship between the modulus of elasticity and the weight fraction of the reinforcing particles (HAP powder), which were added to the (polymer blend) matrix. It can be noted that increase in modulus of elasticity appears if small quantities of HAP particles was added due to the high surface area and smoothness of HAP particles and the polymer chains are restricted within the HAP structure and become easy to guide between the polymer chains. As for increasing the added quantities of HAP particles leads to agglomeration particles and weak interconnection between chains, it slip one by one and are difficult to direct, so the elastic modulus decreases with increasing weight fraction of (HAP powder). It can also be seen that HAP powder improve modulus of elasticity at specific limit, this corresponds with references [13]. So, the weight fractions of (3 wt.%) represents the greatest value for the modulus of elasticity for polymer blend reinforced with HAP powder. The value of modulus of elasticity for the polymer blend was (1.3446 GPa), but when adding little quantity of HAP powder as reinforcement material, the modulus of elasticity reaches to (2.7365 GPa) at (3 wt.%).

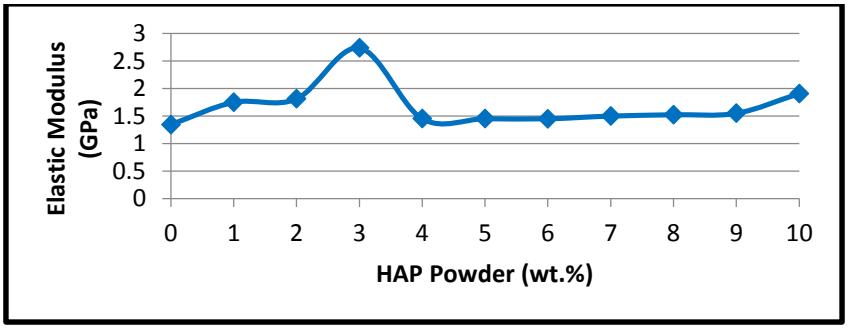

Fig. 9. Elastic modulus and weight ratio of hydroxyapatite powder for composite specimens.

3.2.4. Elongation percentage at break

The polymer blend matrix (bone cement) has the highest elongation percentage equal to (2.192 mm), while the elongation percentage at break of the composites reinforced by HAP powder is varying according to the percentage added of the powder, some are lower than that of the polymer blend matrix, while some are higher its. This due to the higher mechanical properties of reinforcement as compared with polymer blend. Figure (10) show the relationship between the elongation percentage calculated at break point and the weight fraction of (HAP) powder, which was added to the bone cement as reinforcement material. Increasing the weight fraction of (HAP) powder leads to reduce the percentage of elongation for samples. This is due to the presence of fillers imparts the stiffening effect within the matrix and thus imposes a mechanical restraint on the composites, where this corresponds with references [34,35].

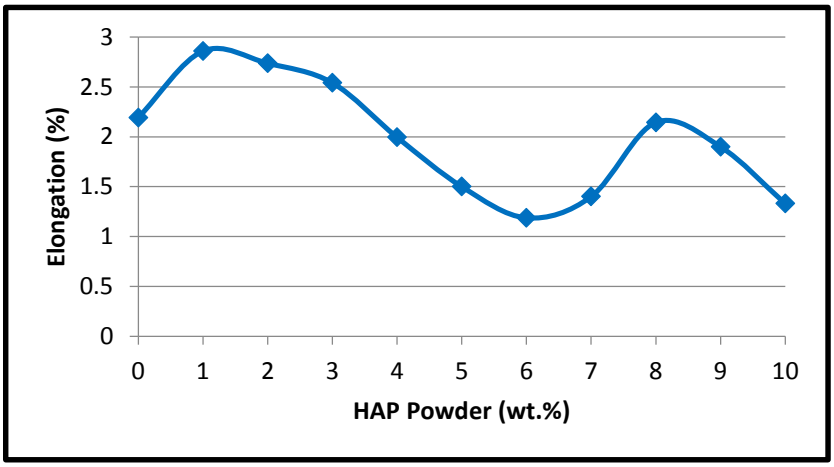

Fig. 10. Elongation property and weight ratio of hydroxyapatite powder for composite specimens.

4. Conclusions

After reach to biopolymer composite dough reinforced with hydroxyapatite powder "the main component of the bone", concluded that, the possibility of preparing this composite material with good mechanical properties according to the standard specifications to compensate the damaged parts of the jaws bones, where the addition of a little percentage of HAP powder to the polymer blend enhances mechanical properties, where the tensile strength, stress maximum, modulus of elasticity have best values at (3 wt.%) of HAP powder. The strength results are consistent with the SEM images that showed significantly increased agglomerates with increasing amount of hydroxyapatite particles.

5. Acknowledgments

The authors would like to thank Mr. Khaleel Fadel Abdalkeder, the teacher at Al-Furat Al-Awsat Technical University/ Technical Institute – Samawa for his cooperation regarding the completion of the tests.

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