Preparation and Characterization of Polymer- Ceramic Composite Bio-material

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Abstract

   This work focuses on studying the addition effect of the prepared HA powder as a filler material before and after the calcination process with different volume fractions (2.5, 5, 7.5, 10, 12.5, 15) vol% to the unsaturated polyester resin matrix. Many mechanical and physical tests were used to determine the properties of the prepared composite material which involved tensile strength, the modulus of elasticity, the elongation percentage at break, compression strength, compression modulus, bending strength, impact strength, fracture toughness, hardness and water absorption percentage. For the prepared HA powder, the Ca/P ratio was increased after the calcination process from 2.45 to 2.51. X- ray diffraction patterns for the prepared HA powder before and after the calcination process revealed an increase in the HA peak intensity after the calcination process. Secondary phases also appeared after the calcination process like α- $Ca_3(PO_4)_2$) and (β- $Ca_2P_2O_7$). For the prepared composite material with both groups of HA filler particles, the results had shown that the mechanical properties which included: tensile strength, modulus of elasticity, compression strength, compression modulus, bending strength, fracture toughness and hardness have been increased with increasing volume fraction of HA filler particles and reached their maximum value at (7.5 vol%). Furthermore, the increasing in volume fraction revealed a decreasing in the evaluated properties. Both the elongation percentage at the break point and the impact strength decreased with increasing volume fraction of HA filler particles. The water absorption percentage as a physical property for the prepared composite material showed an increase with increasing volume fraction of HA filler particles. The improvement of unsaturated polyester resin with calcined HA filler particles had shown greater values for the fore-mentioned properties than the improvement of unsaturated polyester resin with uncalcined HA filler particles.

تحضير ودراسة مادة حيوية متراكبة ذات أساس بوليمري مقواة بدقائق سيراميكية

الخلاصة

يركز هذا البحث على دراسة تأثير اضافة مسحوق الهيدروكسي اباتايت المحضر وبكـــلا الحالتين قبل وبعد عملية الكلسنة كحشوات تقوية بكسور حجميـــة مختلفـــة (10 ,7.5 ,5 ,2.5 vol% (15 ,12.5) الى راتنج البولي استر غير المشبع كمادة اساس. تم أجراء العديـــد مـــن الاختبارات الميكانيكية والفيزيائية لغرض تحديد خواص المادة المتراكبة المحضرة, حيث

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تضمنت تلك الاختبارات : مقاومة الشد , معامل المرونة , النسبة المئوية للاستطالة عند الكســــر , مقاومة الانضغاط , معامل الانضغاط , مقاومة الانحناء , مقاومة الصدمة , متانة الكسر , الصلادة و النسبة المئوية للامتصاصية . بالنسبة لمسحوق الهيدروكسي اباتايت المحضر بعد عملية الكلسنة , ازدادت نسبة Ca/P بزيادة مقدارها من 2.45 الى 2.51 . في حين اظهرت مخططـــات حيــود الاشعة السينية بعد عملية الكلسنة ظهور الطورين الثـــانويين [Ca₃(PO₄)₂ -α , Ca₂P₂O₇ -β]كما اظهرت زيادة في قمة الشدة للهيدروكسي اباتايت المكلســن . بالنســـبة للمـــادة المتراكبـــة المحضرة وبكلا المجموعتين من حشوات التقوية (مسحوق الهيدروكسي اباتايت الغير مكلســن والمكلسن) , وضحت النتائج بأن الخواص الميكانيكية التي تضمنت : مقاومة الشد , معامل المرونة , مقاومة الانضغاط , معامل الانضغاط , مقاومة الانحناء , متانة الكسر و الصلادة قد ازدادت بزيادة الكسر الحجمي لحشوات التقوية وبلغت أقصى قيمة لها عند الكسر الحجمي (7.5 Vol%) بعدها بدأت تلك الخواص بالانخفاض مع زيادة الكسر الحجمي . اما النسبة المئوية للاستطالة عند الكسر ومقاومة الصدمة فقد قلتا مع زيادة الكسر الحجمي لحشوات التقوية . اما الأختبار الفيزيائي في هذا البحث والمتمثل بالنسبة المئوية للامتصاصية للمادة المتراكبة المحضرة فقد اظهر زيادة نســـبة الامتصاصية بزيادة الكسر الحجمي لحشوات التقوية . لقد بينت النتائج العملية لهـــذا البحـــث ان تدعيم راتنج البولي استر غير المشبع بحشوات تقوية من مسحوق الهيدروكسي اباتايت المكلسن يعطي قيم اعلى للخواص الموضحة اعلاه من تدعيم الراتنج نفسه بحشوات تقوية من مســـحوق الهيدروكسي اباتايت غير المكلسن.

1. Introduction

Trauma, degeneration and diseases often make surgical repair or replacement necessary when a person has joint pain. The main concern is the relief of pain and return to a healthy and functional life style [1]. Tissue engineering has emerged as a promising alternative approach in the treatment of malfunctioning or lost organs. In this approach, a temporary scaffold is needed to serve as an adhesive substrate for the implanted cells and a physical support to guide the formation of the new organs. In addition to facilitating cell adhesion, promoting cell growth, and allowing the retention of differentiated cell functions, the scaffold should be biocompatible, biodegradable, highly porous with large surface/volume ratio, mechanically strong, and malleable [2]. Basic principles of tissue engineering are indicated in Figure (1). Over the last century, biocompatible materials such as metals, ceramics and polymers have been extensively used for surgical implantation. Polymer materials have received increasing attention and been widely used for tissue engineering because of easy control over biodegradability and processability [3, 4]. Resorbable polymers belong to the group of biodegradable polymers. The special feature of biodegradable polymers is their comparatively fast degradation to biologically harmless substances. Polymers degrading to substances that are not only non - toxic but also can be metabolized by the human organism are called resorbable or bioresorbable. This characteristic makes resorbable polymers particularly interesting for medical applications, especially as materials for implants [5]. On the other hand, the calcium phosphate ceramics have high potential in use as bio-substituent materials such as artificial bones or dental crowns because their chemical

2498

composition is close to that of hard human tissues [6].

The development of biocomposites offers great promise to improve the efficacy of current tissue substitutes, that are too frequently single-phase materials, that lack such property as diversification. Owing to similarities with the chemical composition of bone, polymer/ ceramic composites were one of the first biocomposites considered for orthopedic implant applications. Specifically for polymer/ ceramic biocomposites, it is possible to obtain a wide range of mechanical and biological properties by modifying the type and distribution of the ceramic phase in the polymer matrix. Importantly, increased interactions between biocomposites and host tissue can be achieved by optimizing polymer/ ceramic composites. For this reason, a wide variety of biocomposites have been synthesized and fabricated for various biomedical applications during these years [7].

2. Experimental Part

a- A suspension of (27gm) of $Ca(OH)_2$ was dissolved in (1000 ml) of distilled water and vigorously stirred.

b- A solution of (24.6gm) of H_3PO_4 was diluted in (1000 ml) of distilled water and was slowly added dropwise over 1 to 2hrs under a condition of pH= (8) to reveal a gelatinous precipitate.

c- The reaction mixture was aged at room temperature for a week.

d- The resulting slurry was cleared by removing floating liquid.

e- The resulting slurry was washed with distilled water followed by clearing floating liquid (three times) and then washed with ethanol and the floating liquid was also cleared.

f- The resulting slurry was filtered by filtration papers to remove maximum amount of liquid.

g- The resulting slurry was dried at $80°C$ in an oven overnight.

h- The resulting agglomerate was milled and sieved using sieve of ($<53\mu m$) aperture.

i- Half of the prepared powder was calcined at $800°C$ in an oven for 3 hours, and left inside the oven for 24hrs for cooling.

j- Again the re-milled powder was sieved using sieve of ($<53\mu m$) aperture [8, 9].

k- To prepare the specimens of the study, the accelerator and the hardener, were added as a weight % with an amount of (0.5%) and (2%) respectively.

l- The reinforcement filler of HA and the matrix (including the accelerator and the hardener) were mixed at room temperature continuously and slowly to avoid bubbling during mixing. The process was continued for (10) minutes until the mixture became homogeneous.

m- The mixture was poured from one corner into the mould (to avoid the bubbles formation which causes cast damage) and the uniform pouring is continued until the mould is filled to the required level.

n- The mould was placed on an electrical vibrator to remove any residual bubbles.

o- The mixture was left in the mould for (24) hrs at room temperature to solidify. Then the cast was placed inside an oven dryer for (1) hr at ($55°C$, this step was important to reveal complete polymerization, best

Eng. & Tech. Journal, Vol.28, No.13, 2010

Preparation and Characterization of
Polymer- Ceramic Composite Bio-material

coherency, and to relieve residual stresses.

p- X- ray diffraction inspection was done for identifying the phase purity of prepared Hydroxyapatite before and after calcination process, and also to introduce the role of calcination process on HA powder crystallinity. This inspection was performed in the Ministry of Sciences and Technology- Iraq, using X- ray diffractometer with the following analysis conditions:- target: Cu, wavelength: $1.54060(A^\circ)$, voltage: 40(Kv), current: 30(mA), range: 20- 60(deg), step: 0.05(deg), speed: 5(deg/min).

q- Evaluation of Ca/P ratio was done for (HA) powder before and after calcination in the Ministry of Industry and Minerals- State Company of Geological Survey and Mining - Iraq, and as shown in Table (1).

r- Optical microscope examination, whose result in Figure (2, a), appears as irregular shaped particles with a wide distribution before calcination process. After calcination process Figure (2, b) revealed a refined irregular shaped particle and the agglomeration of particles disappeared showing good distribution of the particles.

s- Real and apparent Ddensities of prepared (HA) powder were calculated by using pecnometer and gravity methods respectively and the results were presented in Table (2).

t- Water absorption test was performed according to (ASTM D 570- 98) at room temperature [10]. Tensile test was performed according to (ASTM D638M- 87b) at room temperature [11]. Compression test was performed according to (ASTM D695- 85) at room temperature [12]. Flexural strength test was performed according to (ASTM D790) at room temperature [13]. Impact test was performed according to (ISO- 179) at room temperature [14]. Hardness test was performed by using shore hardness (D) and according to (ASTM D 2240) at room temperature [15].

3. Results and Discussion
3.1 Physical Evaluation

Evaluation of Ca/P ratio shows that the Ca/P ratio increased from 2.45 before calcination process to 2.51 after calcination process. This increase in Ca/P ratio may be due to the loss of weight on heating that is associated with the adsorption of bound H_2O on the surface of HA powder [16]. The X- ray diffraction patterns in Figure (3, a&b) indicated that the presence of HA as a major constituent and α- $Ca_3 (Po_4)_2$ and β- $Ca_2 P_2 O_7$ as secondary phases, as in Table (3). The results of x- ray diffraction analysis for uncalcined HA referred to the existence of three strong intensity peaks of HA phase. Also, there are many weak intensity peaks of HA phase and other secondary phases which are α- $Ca_3 (Po_4)_2$ and β- $Ca_2 P_2 O_7$. After the calcination process at temperature $800^\circ C$ for 3hrs, the results clearly indicated that the intensity peak heights of HA phase increased with the existence of the two secondary phases α- $Ca_3 (Po_4)_2$ and β- $Ca_2 P_2 O_7$ which appear in weak intensity peaks. So, the calcination process led to

Eng. & Tech. Journal, Vol.28, No.13, 2010

Preparation and Characterization of Polymer- Ceramic Composite Bio-material

increase the grain growth that led to increase in crystallinity. Figure (4) shows the relationship between water absorption percentage and volume fraction of the filler particles of uncalcined HA and calcined HA, which were added to the unsaturated polyester resin, respectively. The figure illustrates that the water absorption percentage increases with increasing volume fraction of HA filler particles, for both groups of composite material. This increase may be due to the filler particles which have a higher water absorption percentage than the matrix material. It is known that the water absorption percentage increases with increasing particles size due to increase of surface area of the particles, but in this work the composite material filled with calcined HA particles shows a higher water absorption percentage than the other, which was filled with unclcined HA particles because of the bound H_2O, that is absorbed by the unclicend particles during the preparation process of HA powder. This means that these particles are saturated with water while the calcined particles are dried which will absorb more water. All of that will affect the water absorption percentage of the composite material, which is filled of these particles.

3.2 Mechanical Evaluation

An axial tensile load being applied to a standard tensile specimen of a rectangular cross section with a constant strain rate at about 5mm/min. Figure (5) shows the stress- strain diagram under tension for unsaturated polyester resin without addition of HA filler particles. It can be noted that, the initial part of the curve is linear where the specimen behaves in an elastic manner. Beyond the limit of proportionality, the specimen may undergo a sudden extension without a corresponding increase in load, which refers as a yield point. After the yielding of material, the (stress-strain) curve deviates due to the deformation of specimen in plastic manner. An increase in the plastic deformation continued with increasing stress, till the fracture occurred in the specimen (the deformation of polymeric material will lead to break their bonding), at this time, the ultimate tensile strength can be estimated. Figure (6) shows the (stress- strain) diagram under tension for a composite material consisting of unsaturated polyester resin filled with the prepared HA powder before the calcination process with different volume factions (0, 2.5, 5, 7.5, 10, 12.5 and 15) vol%. The diagram illustrates that, the stress increases with increasing volume fraction of HA filler particles until the addition ratio of (7.5 vol%), then the stress decreases. The reasons behind such behavior, that the strengthening mechanism of HA reinforcing filler in which, the amount of these filler particles plays an important role by impeding increasing the slipping of polyester resin chains. Knowing that, the chains require high stress to bend them in narrow space among the particles. The strengthening increases and reaches maximum values at the ratio of (7.5 vol%), after which the increase of volume fraction causes the fraction between the particles and the matrix causes slipping among the particles in tension. Also the nature of the bonding between the matrix and the filler particles has an important role, i.e. the good ability of a liquid polyester to spread on the solid particles (good wettability) and may cause increasing bonding force

between the matrix and the filler material, so the resultant composite will require high stress to break their physical bonding. Until the volume fraction becomes (7.5 vol%), the interface region between the matrix and the additive represents strengthening region. Beyond this ratio, the interface region will increase to a limit at which the discontinuity between the matrix and the filler material becomes clear and increases due to the decreasing in wettability, so that the composite material requires low stress to break their physical bonding.

Figure (7) shows the (stress- strain) diagram under tension for a composite material consisting of unsaturated polyester resin filled with the prepared HA powder after the calcination process with different volume fractions (0, 2.5, 5, 7.5, 10, 12.5 and 15) vol%.

The behavior of this material is similar to that obtained in the material fillered with uncalcined HA powder, except that in the second (after calcination) the material reveals a higher stress values. This is due to the effect of particles size difference and crystallinity, as mentioned earlier. The calcination process causes a decrease in particle size due to the desorption of the bound H_2O (crystalline water) on the surface of HA powder. Also, it causes an increasing in crystallinity, so the calcination process increases the mechanical properties. So the composite material, that used calcined HA as a filler shows higher stress values than obtained in the uncalcined one. In figure (5) the strain increases with increasing stress, while in figures (6) and (7), the strain increases with increasing stress and decreases with increasing volume

fraction of HA filler particles. The reason behind such behavior is because the increasing of particles number will decrease their movement, so the strain will decrease. Also, the strain values of the composite material filled with calcined HA particles are lower than the same values for the composite material filled with uncalcined HA particles. This is due to the improvement of the mechanical strength that is revealed from the calcination process. Figure (8) shows the relationship between the tensile strength and the volume fraction of the filler particles of uncalcined HA and calcined HA which were added to the unsaturated polyester resin, respectively. It can be noted that the addition of calcined HA filler particles have a noticeable effect on the tensile strength more than the uncalcined one, and this is due to the improvement in the mechanical properties that is associated with the addition of calcined HA. Figure (9) shows the relationship between the modulus of elasticity and the volume fraction of the filler particles of uncalcined HA and calcined HA, which were added to the unsaturated polyester resin, respectively. It can be noted that the modulus of elasticity increases with increasing volume fraction until (7.5 vol%) is reached, then the elastic modulus decreases with increasing volume fraction. So, the volume fraction (7.5vol%) represents the greatest value for the modulus of elasticity. This may be due to the strengthening mechanism and the nature of bonding that mentioned before. Also, the increase in elastic modulus of the composite material may be due to the fact the elastic modulus of HA range is (40 - 117) GPa and it is much higher than

Eng. & Tech. Journal, Vol.28, No.13, 2010

Preparation and Characterization of
Polymer- Ceramic Composite Bio-material

that for unsaturated polyester resin [17]. The decrease in the modulus of elasticity with increasing volume fraction after the addition passed more than (7.5 vol%) may be due to the fraction between the particles and the matrix that develops a slipping among the particles. Also it may be due to the decrease of wettability, which is mentioned earlier. In addition, the filler particles are brittle and when they are added to the matrix, they will increase their strength, but with the higher addition (%) (i.e. more than 7.5 vol%) the brittleness of the composite material will increase too much while, the elastic modulus will begin to decrease. Here, also the addition of calcined HA filler particles will improve the elastic modulus more than the addition of uncalcined HA filler particles, and this is due to the effect of the particles size difference and the crystallinity, which are associated with the improvement of the mechanical properties as mentioned before. Figure (10) shows the relationship between the elongation percentage calculated at break point and the volume fraction of the filler particles of uncalcined HA and calcined HA, which were added to the unsaturated polyester resin, respectively.The figure illustrates the elongation percentage decreases with the increasing volume fraction of HA filler particles for both groups of composite materials. This is because of increasing the particle number, which act as localized stress concentration regions, so the elongation percentage at break will be decreased. Also, the elongation percentages at break for the composite material filled with calcined HA particles are lower than the same percentages for the

composite material filled with uncalcined HA particles. This is due to the effect of particles size difference and crystallinity which will increase the mechanical strength for the same volume fraction in the composite material filled with calcined HA particles and then decrease the elongation percentage.

Figure (11) shows the relationship between the compression strength and the volume fraction of the additive particles of uncalcined HA and calcined HA which were added to the unsaturated polyester resin, respectively. The figure illustrates that the compression strength increases with increasing volume fraction of HA filler particles until (7.5vol %) is reached, and then the compression strength decreases with further addition increase. This is may be due to the strengthening mechanism and the nature of bonding, which are mentioned before. The decrease of the compression strength after adding (7.5 vol%) of HA filler particles may be because of the decrease in wettability and the fraction between the particles and the matrix which were mentioned before. Again, it can be noted that the addition of calcined HA filler particles improves the compression strength more than the addition of uncalcined HA filler particles, this is due to the effect of particle size difference and crystallinity which are mentioned before. The same explanation for the compression strength can be said for the compression modulus which is shown in figure (12). The properties of bending usually depend on the nature of the bonding between the filler and the matrix materials. Figure (13) shows the relationship between the bending strength and the volume

fraction of the filler particles of uncalcined HA and calcined HA powder, which were added to the unsaturated polyester resin, respectively. The figure illustrates that the bending strength increases with increasing volume fraction of HA filler particles, and reaches its maximum amount at (7.5 vol%), then the bending strength decreases with increasing volume fraction. This can be related to the strengthening mechanism and the nature of the bonding which were mentioned before. The increase in bending strength may be also due to the high elastic modulus of the filler material compared with that of the matrix material which is in agreement with [101]. The decrease in the bending strength after adding (7.5 vol%) of HA fillers can be related to the decrease in the wettability that was mentioned before, especially the bending strength largely depends on the bonding force between the matrix and the filler material. The composite material containing calcined HA filler particles has a greater bending strength than the one with uncalcined HA filler particles because of the effect of particle size difference and crystallinity which are mentioned before. Impact test is different from the other tests described above. It is a fast dynamic test with a fast leading to a change in material behavior. Figure (14) shows the relationship between the impact strength and the volume fraction of the filler particles of uncalcined HA and calcined HA, which were added to the unsaturated polyester resin, respectively. The figure illustrates that the impact strength decreases with increasing volume fraction of HA filler particles for both groups of composite materials. This is because of the filler

particles, which may represent points for a localized stress concentration, from which the failure will begin. Also, it can be noted that the decrease in the impact strength of the composite material, which tends to improve by calcined HA filler particles is less than the other, which was improved by uncalcined HA filler particles. This is due to the effect of particle size difference and crystallinity which are mentioned before. Figure (15) shows the relationship between the fracture toughness and the volume fraction of filler particles of uncalcined HA and calcined HA which, were added to the unsaturated polyester resin, respectively. The figure illustrates that fracture toughness increases with increasing volume fraction of HA filler particles, and reaches its maximum amount at (7.5 vol %), then the fracture toughness decreases with increasing volume fraction. This is due to the dependence of the fracture toughness on the elastic modulus, as mentioned in the equation in [18], and the elastic modulus had shown a similar behavior to the behavior of the fracture toughness as explained. Also, the composite material filled with calcined HA filler particles has higher fracture toughness than the other, which was filled with uncalcined HA filler particles according to the elastic modulus of the first group of composite material, which is higher than that for the second group of composite material. Figure (16) shows the relationship between the hardness and the volume fraction of the filled particles of uncalcined HA and calcined HA, which were added to the unsaturated polyester resin, respectively. The figure illustrates that, the hardness increases with increasing volume fraction of HA

Eng. & Tech. Journal, Vol.28, No.13, 2010

**Preparation and Characterization of
Polymer- Ceramic Composite Bio-material**

filler particles, and reaches its maximum amount at (7.5 vol %), then the hardness decreases with increasing volume fraction. This is because of the filler particles being hard and brittle compared with the matrix material, till the ratio (7.5)vol% the added filler particles increases the hardness of the composite because of increasing the wettability or the bonding between the matrix and the fiiler particles, but after this ratio the composite becomes brittle due to the large content of the particles and the hardness will decrease. Here, also the composite material of calcined HA filler particles has a higher hardness than the other, which was filled with uncalcined HA filler particles due to the effect of particle size difference and crystallinity as mentioned before.

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Preparation and Characterization of
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Table (1) some standard properties of unsaturated polyester resin at Room temperature:

Density (gm/cm^3)	Tensile Strength (MPa)	Tensile Modulus (MPa)	Compression Strength (MPa)	Bending Strength (MPa)	Fracture Toughness (MPa.m$^{1/2}$)
1.2	50-75	2.5-4.5	100	125	0.6

Table (2) Some standard properties of HA at room temperature:

Density (theoretical, gm/cm^3)	Tensile Modulus (MPa)	Compression Strength (MPa)	Bending Strength (MPa)
3.16	4-117	294	147

Table (3)Analysis of Ca/P ratio of prepared HA powder:

Sample No.	Ca%	P%	Ca/P
(HA) before calcination	35.10	14.34	2.45
(HA) after calcination	43.90	17.50	2.51

Table (4)The magnitude of real and apparent densities:

Real density(gm/cm^3)	Apparent density(gm/cm^3)
(HA) before calcinations:1.71	(HA) before calcinations: 0.405
(HA) after calcinations: 0.95	(HA) after calcinations: 0.32

Table (5) the different phases formed according to the X- ray diffraction pattern analysis:

Before Calcination	After Calcination
HA	HA
α- Ca$_3$ (Po$_4$)$_2$	α- Ca$_3$ (Po$_4$)$_2$
β- Ca$_2$ P$_2$ O$_7$	β- Ca$_2$ P$_2$ O$_7$

Eng. & Tech. Journal, Vol.28, No.13, 2010

Preparation and Characterization of
Polymer- Ceramic Composite Bio-material
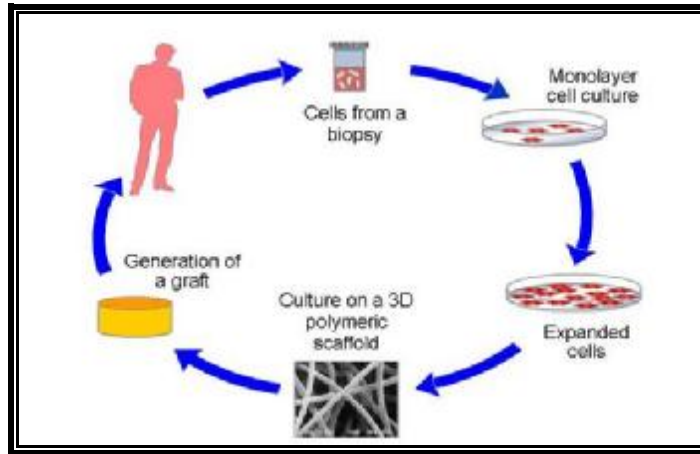
Figure (1) Basic Principles of Tissue Engineering.
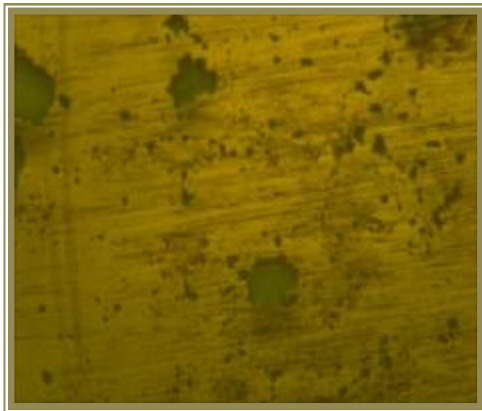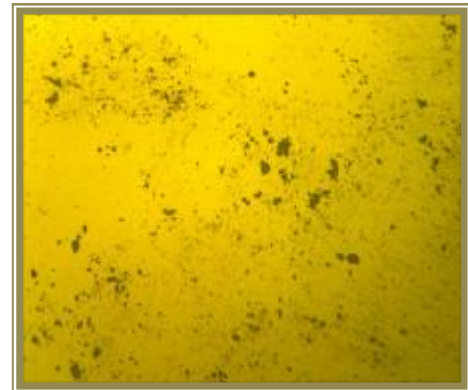
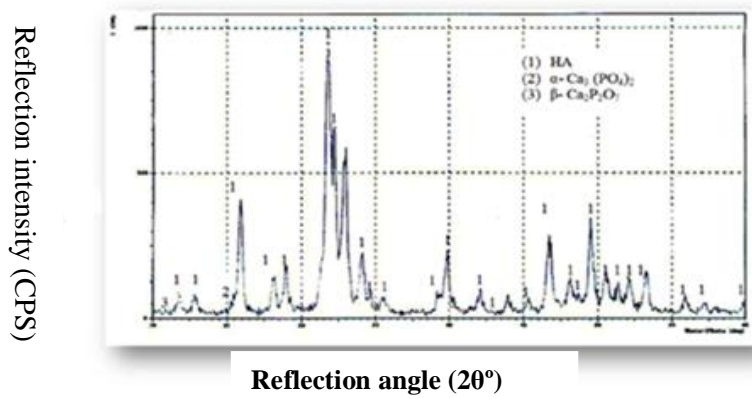(a)   Before calcination.   (b) After calcination.

Figure (2, a&b) Optical microscopic image shows the particle shapeof the
prepared HA powder (a) before and (b) after calcination practices process
at a magnification of (X400).
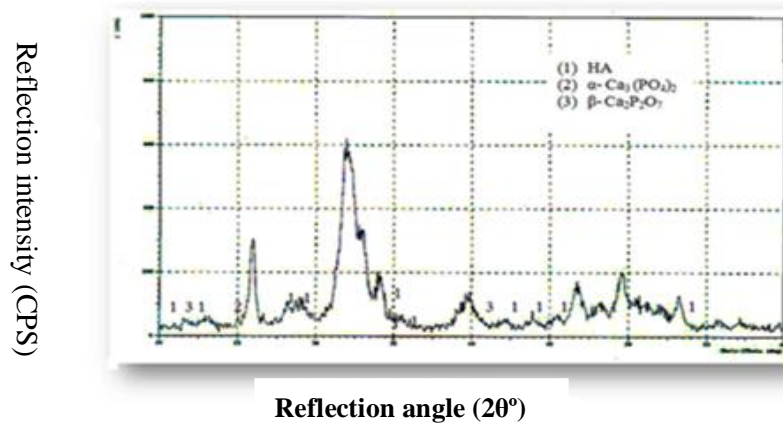
(a) Before calcination.

(b) After calcination.

**Figure (3) X- ray patterns of HA powder before and after the calcination
process.**

Eng. & Tech. Journal, Vol.28, No.13, 2010

**Preparation and Characterization of
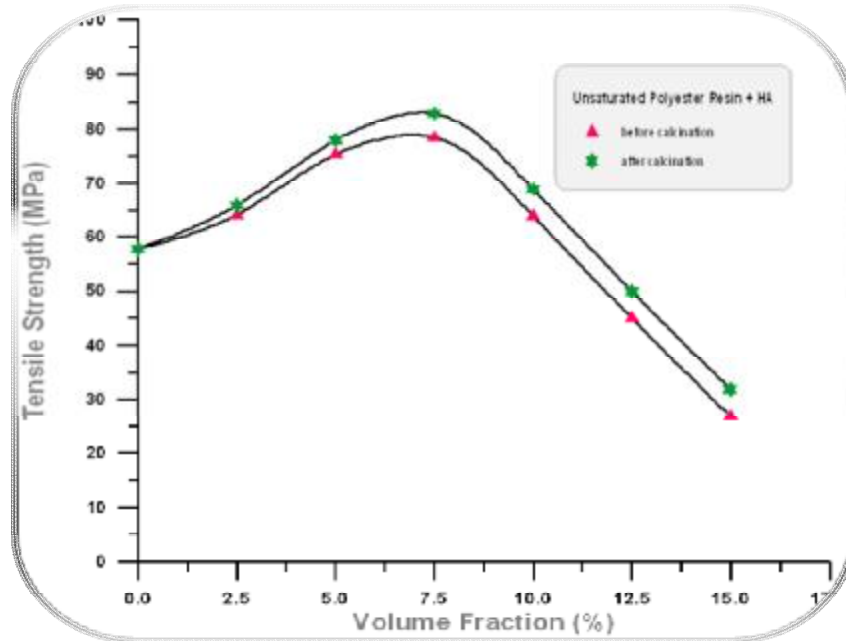Polymer- Ceramic Composite Bio-material**
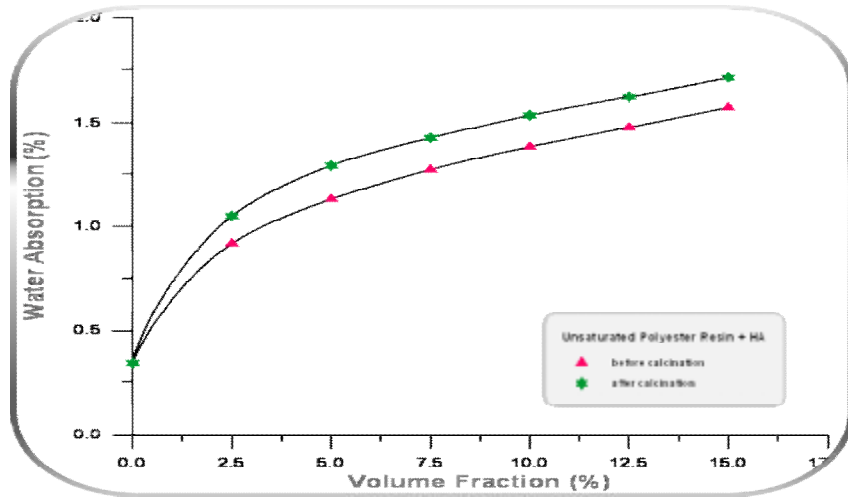
**Figure (4) the relationship between the water absorption percentage and
volume fraction for unsaturated polyester resin filled with uncalcined (HA)
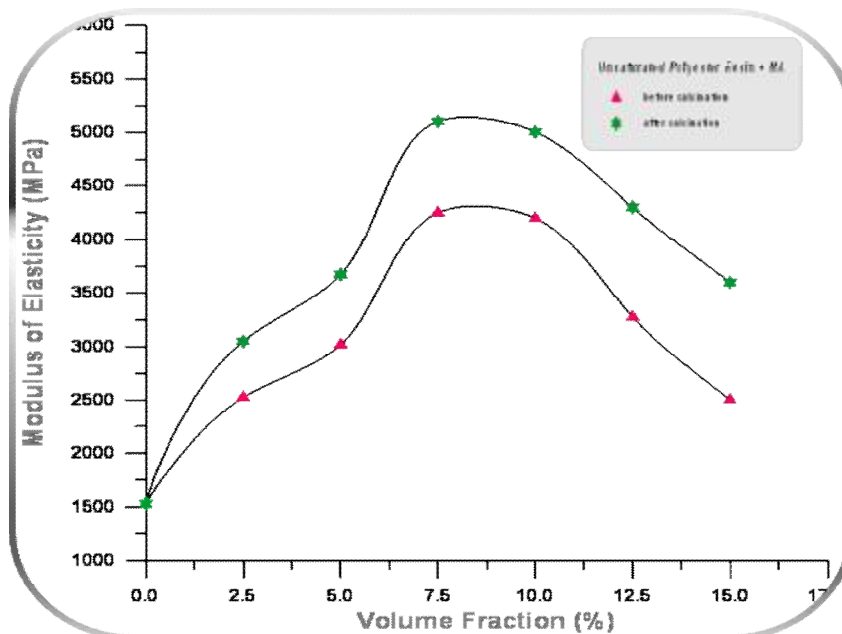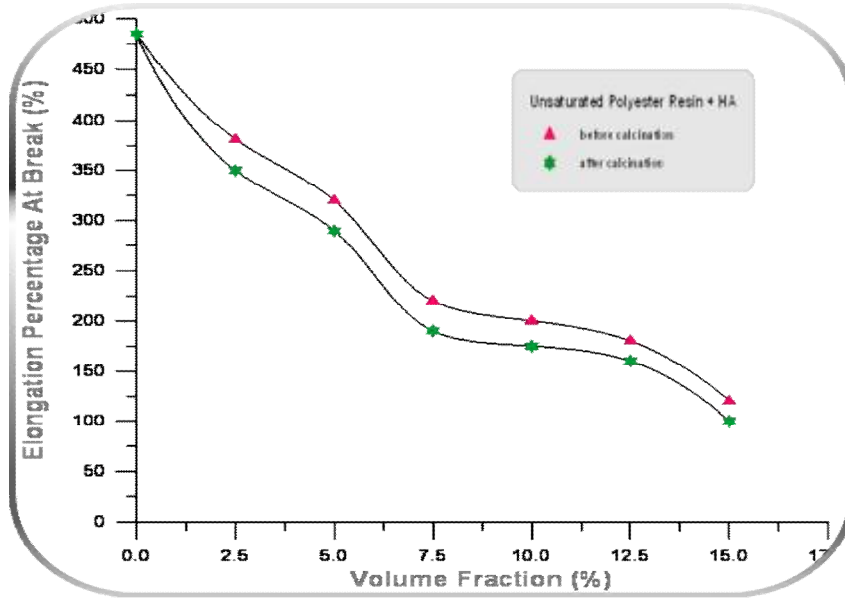and calcined (HA) powder**.
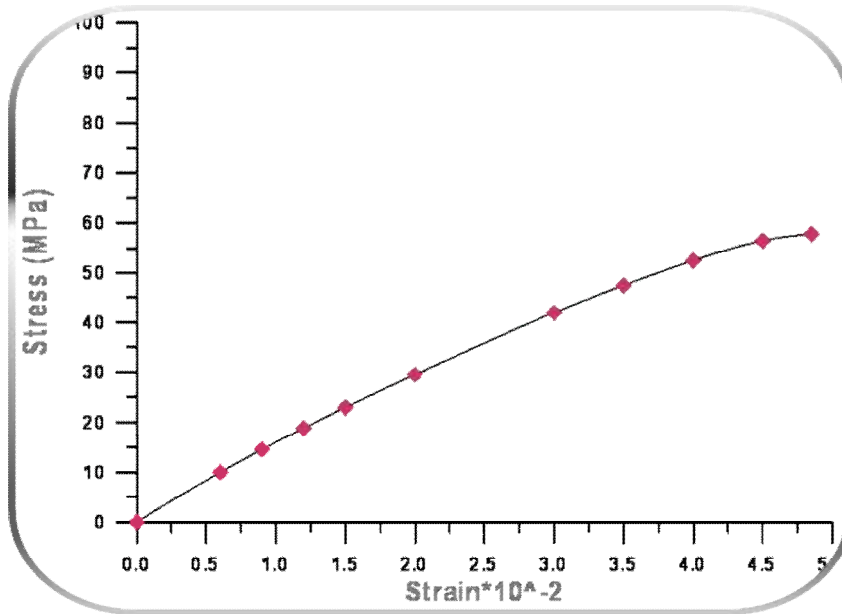
Figure (5) the stress- strain diagram for unsaturated polyester resin.

Eng. & Tech. Journal, Vol.28, No.13, 2010
        Preparation and Characterization of
        Polymer- Ceramic Composite Bio-material
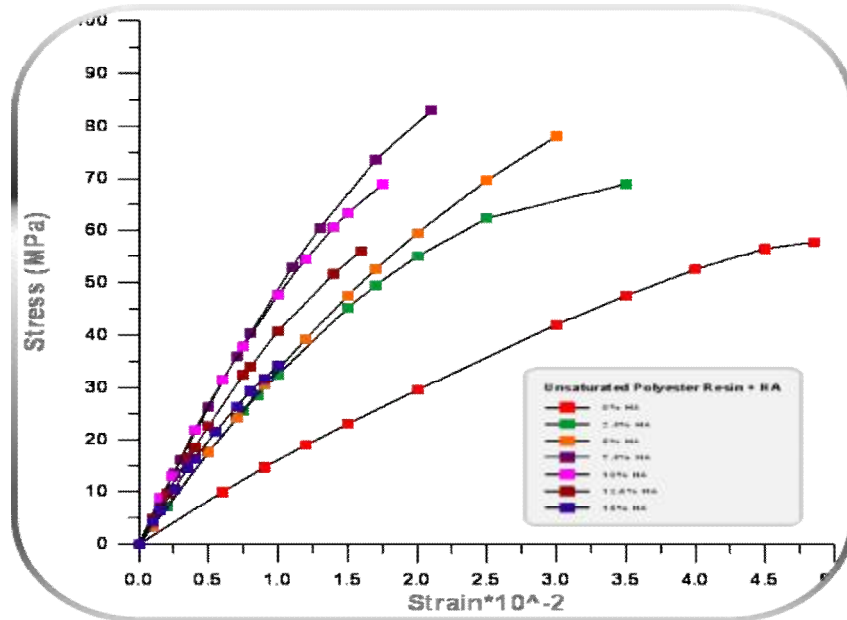
Figure (6) the relationship between the stress and strain for unsaturated polyester resin filled with uncalcined (HA) powder with different volume fractions.
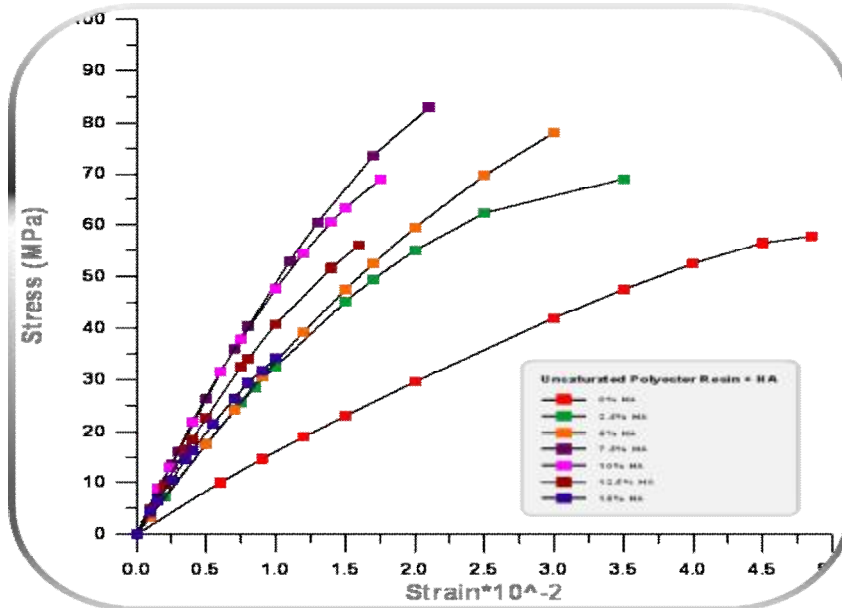
Figure (7) the relationship between the stress and strain for unsaturated polyester resin filled with calcined (HA) powder with different volume fractions.

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Figure (8) the relationship between the tensile strength and volume fraction for unsaturated polyester resin filled with uncalcined (HA) and calcined (HA) powder.
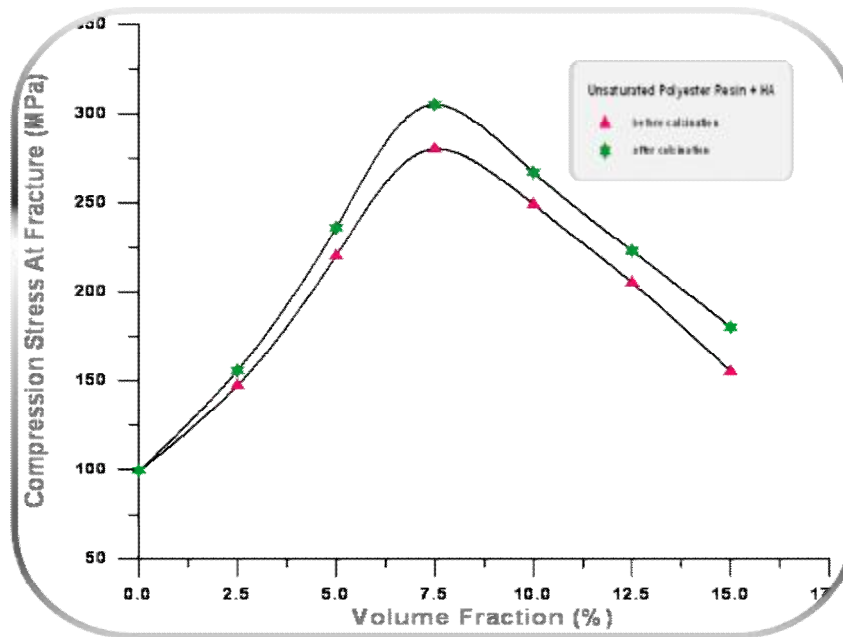

Figure (9) the relationship between the modulus of elasticity and volume fraction for unsaturated polyester resin filled with uncalcined (HA) and calcined (HA) powder.

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**Figure (10) the relationship between the elongation percentage at break and
volume fraction for unsaturated polyester resin filled with uncalcined (HA) and
calcined (HA) powder.**

**Figure (11) the relationship between the compression stress at fracture and volume fraction
for unsaturated polyester resin filled with uncalcined (HA) and calcined (HA) powder**
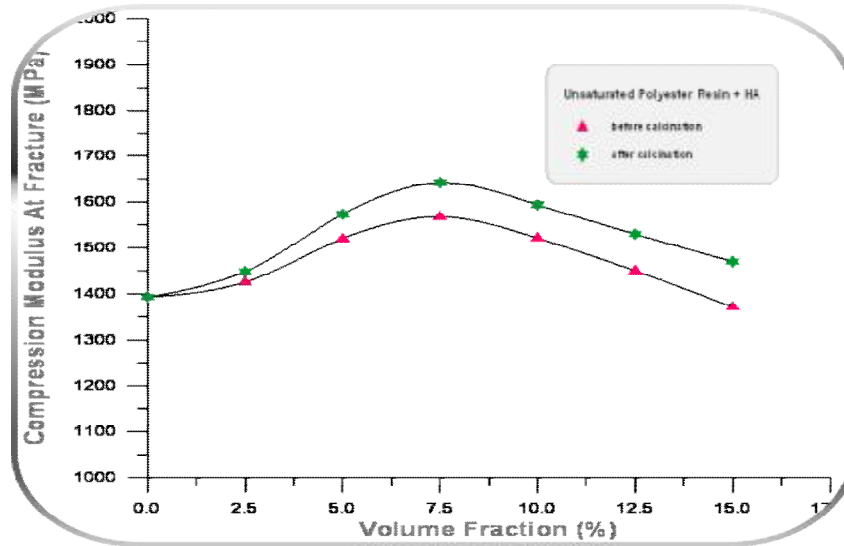
2512

Figure (12) the relationship between the compression modulus at fracture and volume fraction for unsaturated polyester resin filled with uncalcined (HA) and calcined (HA) powder.
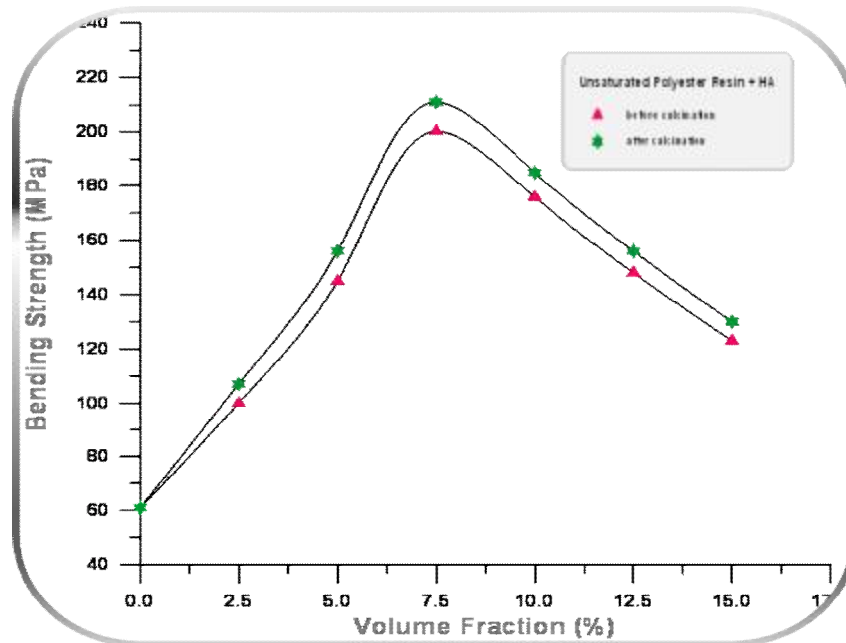

Figure (13) the relationship between the bending strength and volume fraction for unsaturated polyester resin filled with uncalcined (HA) and calcined (HA) powder.

Eng. & Tech. Journal, Vol.28, No.13, 2010

**Preparation and Characterization of
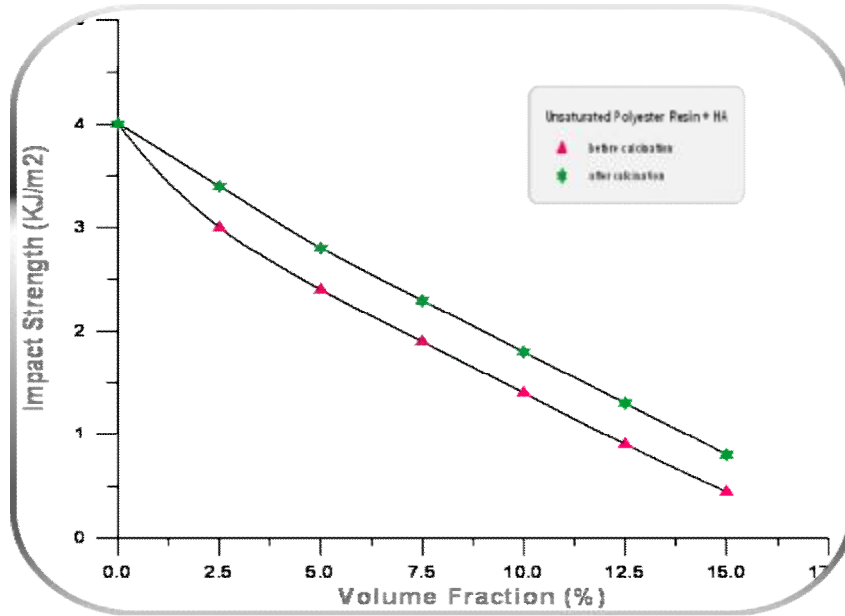Polymer- Ceramic Composite Bio-material**

**Figure (14) the relationship between the impact strength and volume fraction for
unsaturated polyester resin filled with uncalcined (HA) and calcined (HA) powder.**
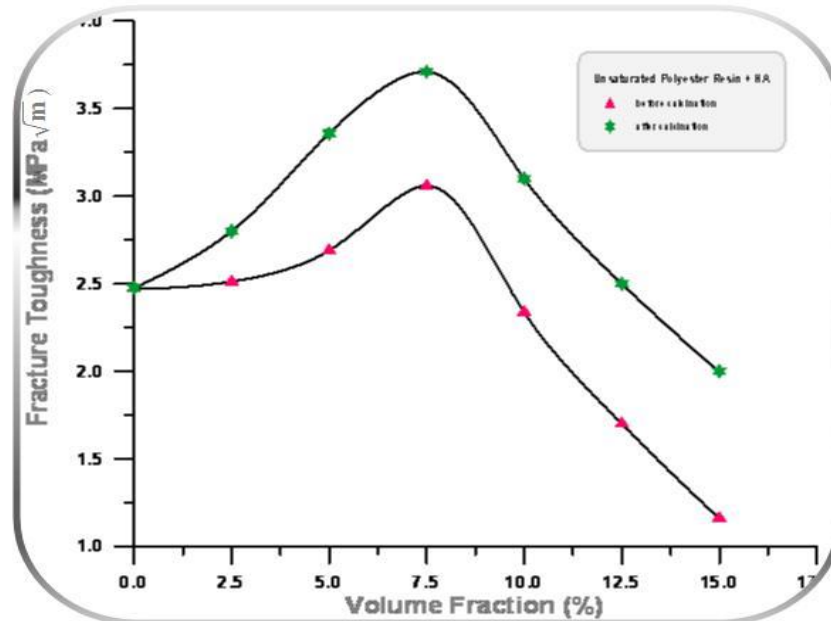

**Figure (15) the relationship between the fracture toughness and volume
fraction for unsaturated polyester resin filled with uncalcined (HA) and
calcined (HA) powder.**

Eng. & Tech. Journal, Vol.28, No.13, 2010

**Preparation and Characterization of
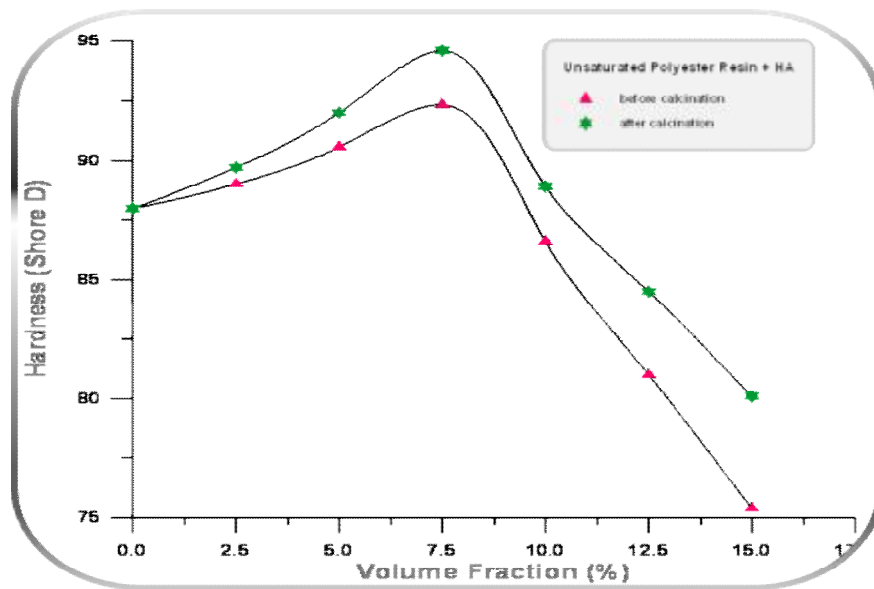Polymer- Ceramic Composite Bio-material**

**Figure (16) the relationship between the hardness and volume fraction for unsaturated
polyester resin filled with uncalcined (HA) and calcined (HA) powder.**