Effect of Sintering Temperature on Some Physical And Mechanical Properties of Fabricated Hydroxyapatite Used For Hard Tissue Healing

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Abstract

This work focuses on studying the role of drying and calcination on stiochiometry and crystallinity of deposited HA. Also, studying the effect of sintering temperature on phases generated, physical and mechanical properties of sintered HA powder compact at a range of (800-1200)˚C.

Both Ca/P ratio and crystallinity were increased after calcination, where the Ca/P ratio raised from 1.7 to 1.9 and the height of Hydroxyapatite peak intensity was also increased .Secondary phases also appeared.

X-ray diffraction patterns and electrical microscopic pictures of polished surfaces of the Hydroxyapatite compact after sintering had revealed the process of densification and crystallization of Hydroxyapatite . The increase of sintering temperature leads to grain growth, while surface cracking and other defects became lower i.e. porosity and surface voids.

Both hardness and fracture strength were increased for samples sintered at high temperatures where they reached their maximum values at sintering temperature (1200)ºC .The maximum linear shrinkage was 20% at sintering temperature 1200ºC. The maximum bulk density was (2.173) g/cm^3 at sintering temperature of 1200ºC and the maximum apparent density was (2.58) g/cm^3 at sintering temperature 900ºC. The maximum apparent and open porosities were 47.136% and 47.058% respectively at sintering temperature 900ºC. The maximum water absorption was 39.13% at sintering temperature 800ºC.

Keywords: Bioceramics, preparation of Hydroxyapatite, Bioactive Hydroxyapatite, Reinforcement of Hydroxyapatite.

تأثير درجة حرارة التلبيد على بعض الخصائص الفيزياويه والميكانيكيه للهيدروكسي اباتاتيه المصنع والمستخدم لمعالجه الانسجه الصلبه

الخلاصة

يركز هذا البحث على دراسة دور عمليتي التجفيف و الكلسنة على التوازن الكيميائي و تبلور الهيدروكسي اباتايت المحضر . وكذلك دراسة تأثير درجة حرارة التلبيد على الأطوار المتكونة و الخواص الفيزياوية و الميكانيكية لمسحوق الهيدروكسي اباتايت المكبوس و الملبد ضمن المدى (800-1200)°م.

لوحظ ازدياد كل من التبلورية و نسبة P\ Ca بعد عملية الكلسنة , حيث تزداد نسبة P\ Ca من 1.7 إلى 1.9 بالإضافة إلى ازدياد ارتفاع شدة قمة الهيدروكسي اباتايت و ظهور الأطوار الثانوية.

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أظهرت مخططات الأشعة السينية و صور المجهر الضوئي للسطوح المكبوسة و المصقولة بعد التلبيد ، عملية التبلور و التكثف للهيدروكسي اباتايت . أدت الزيادة في درجة حرارة التلبيد إلى النمو الحبيبي كما تراجعت الشقوق السطحية و العيوب الأخرى (المسامات والفجوات السطحية). ازداد كل من الصلادة و مقاومة الكسر للنماذج الملبدة في درجات الحرارة العالية حيث تصل إلى أعلى قيمها عند (1200)°م.

أعلى قيمة للتقلص الخطي كانت (20)% عند درجة حرارة تلبيد (1200) °م أعلى قيمة للكثافة الكتلية كانت(2.173) غم\سم ^{3} عند درجة حرارة تلبيد(1200)°م . أعلى قيمة للكثافة الظاهرية كانت(2.58) غم\سم ^{3} عند درجة حرارة(900) °م وأعلى قيمة للمسامية المفتوحة و الظاهرية كانت (47.058)% و (47.136)% على التوالي عند درجة حرارة تلبيد (900) °م ،و أعلى قيمة لأمتصاصية الماء كانت (39.13) %عند درجة حرارة تلبيد(800)° م.

1. Introduction

The remarkable progress of ceramics in last decades has resulted in the development of materials with chemical, physical and mechanical properties that are suitable for biomedical application. Ceramic materials used for this purpose are known as bioceramics[1] .

Bioceramics can be used as structural parts like: joint or tissue replacements, can be used as coatings to improve the biocompatibility of metal implants, and can function as resorbable lattices which provide transient structures and a frame work that is dissolved replaced as the body rebuilds tissue. The thermal and chemical stability of ceramics, their high strength, wear resistance and durability all contribute to making ceramics good candidate materials for surgical implants. Some ceramics even feature drug delivery capability [2, 3].

Calcium phosphate is found in a variety of forms which are well-known in the art, such as hydroxyapatite HA($Ca_{10}(PO_4)_6(OH)_2$, Ca/P=1.67) , Tricalcium phosphate TCP ($Ca_3(PO_4)_2$, Ca/P=1.5), and brushite ($CaHPO_4$-$2H_2O$, Ca/P=1), as well as in amorphous form ACP($Ca_x(PO_4)_y$ NH_2O, Ca/P= ≈ 1.5). The relative stability of the predominant form of calcium phosphate is ACP<TCP<<HA [4]. Among calcium phosphate forms: HA, bioactiveglass and glass-ceramic are used in biomedical field [5].

Among the calcium phosphate ceramics, i.e.,$Ca_4P_2O_9$, HA and TCP(β-whitlockite), are known to lead to differences in biodegradation behavior that biodegradable TCP ceramics give rise to extensive bone remodeling around the implant[1].

Apatite is one of very few minerals which are produced and used by biological systems[6].

Apatite is a group of minerals which usually refer to hydroxylapatite , flouroapatite , and chloroapatite , named for high contaminations of OH^- , F^- , Cl^- ion respectively , in the crystal lattice[7,8].

HA is the major component of tooth, enamel, and a large component of bone material[9].

Synthetic carbonate apatites usually display reduced crystallinty whereas biological apatites appear to become more crystalline as the carbonate content increases[9].

The naturally occurring form of calcium apatite is with the formula $Ca_5(PO_4)_3(OH)$, but is usually written $Ca_{10}(PO_4)_6(OH)_2$ to denote that the crystal unit cell comprises two molecules. Pure hydroxyl apatite powder is white , odorless and tasteless[6,10].Table (1) illustrates the identity of hydroxyapatite[6,10,11,12,13] . Synthetic hydroxyapatite is of great importance as a biomaterial. It is one of the few materials that are classed as bioactive (it is one of two materials capable of forming a chemical bond with bone. In vivo the other being bioactive glass of various compositions [14].)Unlike other materials such as alumina and zirconia, which are identified as foreign materials and become encapsulated by fibrous tissue. HA is sometimes used in powder form, but in most cases it is used as a bulk material [15].

HA powder is sensitive depending on preparation conditions e.g. the reaction system used, starting material, pH, temperature, ripening time, calcination temperature, etc [18] .Pore volume and size distribution greatly depends on both sintering temperature and sintering method[19].

Pure HA degrades rapidly and easily if conditions are correct: pH, chemical attack, phagocytosis[20].

HA powders have generally been synthesized from aqueous solutions for use in bioceramic applications. It is known that calcium HA is the least soluble and the most stable compound of calcium phosphate phases in aqueous solutions at pH values higher than 4.2.However; HA powders synthesized in highly alkaline media were recognized by their relatively high thermal stability and phase purity even after high temperature (1100-1300) ºC sintering [21].

Some secondary phases are formed during HA sintering process and there are related to its[22]:

a-Original Ca/P ratio. b-Chemical composition . c-Sintering temperature

2. Experimental work

The following materials were used for the preparation of HA powder:-Calcium hydroxide $Ca(OH)_2$,-Phosphoric acid H_3PO_4 ,-poly-vinyl alcohol (PVA) as a binder,- drops of sodium hydroxide (NaOH) used as pH fixer [18] ,-Distilled water ,-Ethanol(99.9%) .

A suspension of (27)g of $Ca(OH)_2$ was dissolved in 1000ml of distilled water and vigorously stirred , then a solution of (24.6)g of H_3PO_4 was diluted in 1000ml of distilled water was slowly added as dropwise over 1 to 2hrs under a condition of pH=8 to produce a gelatinous precipitate .

The reaction mixture was aged at room temperature for a week. The resulting slurry was cleared by removing floated liquid and then washed with distilled water followed by clearing from floating liquid and then washed with ethanol and the floated liquid was also cleared.

The resulting slurry was dried at 80ºC in an oven over night; the resulting material was re-milled and calcined at 800ºC for 3hrs.

The finely ground powder was mixed with 2% PVA and 250ml warm distilled water and mixed for 15 mins, and then dried in an oven at 80ºC and then the re-milled powder was sieved to(<106μm) [23,24].

Pressing was done by a hydraulic pressing machine with capacity of (384.31)Mpa in a tool steel die of (2.55cm) in diameter with pressing pressure of (76.756) Mpa for two minutes.

Sintering of samples were done at different temperatures of (800, 900, 1000, 1100, 1200) ºC and at holding time of (3) hrs. With a heating rate of 5ºC/min [9] is set up to the required temperatures, then left in the furnace for cooling.

Characterization of sintered samples

X-ray diffraction inspection was performed by using Philips diffract meter pw 1840 with a Cu Kα radiator tube and Ni filter and the diffraction angle (2Θ) was between (20-60) º.

And Ca/P ratio for the HA powder was evaluated before and after calcinations by atomic absorption and the ratio was 1.7 before sintering and 1.9 after sintering.

Electron microscopic examination was used to evaluate the particle shape .Figure (1) shows the particle shape of HA powder which obviously seems irregular and semicircular with a wide distribution .And Particle size distribution was carried out in a laser diffraction particle size analyzer "SHIMADZU SALD-2101", The mean value of diameter was (87.821)μm and the median value was (123.483)μm.

Apparent and real densities were measured by the conventional methods i.e. cup cone and pecnometer respectively[25,26].

Sintered samples were prepared by grinding and polishing then etched with (0.1)M of acetic acid CH_3COOH , followed by washing with distilled water then with ethanol . And the sample surfaces were colored with ink to enhance the microstructural observation which was examined by an optical microscope at a magnification of (X 63).

Linear shrinkage percentage was measured depending on diameteral change by using micrometer, before and after sintering

$$Linear\ shrinkage\ \% = \frac{D_o - D}{D} * 100$$

L.sh: Linear shrinkage.

D_o : Sample diameter after pressing (mm).

D : Sample diameter after sintering (mm).

Apparent and bulk densitiesas well as apparent and open porosities of sintered HA compact powder were measured, using the standard Archimede's method[25,26] .

Fracture strength test was carried out by using the diameterial compression disc test (Brazilian test) where the disc was placed between two surfaces and a load was applied in the rate of (0.5 mm/min) , then the force obtained at fracture was recorded , then the following equation was applied to calculate the strength of material[29]:-

$$\sigma_f = \frac{2p}{pdt}$$

σ_f :fracture strength (N/mm^2).

P : applied load (N).
d :sample diameter (mm).
t :sample thickness (mm).

Vickres method was used to measure the hardness of the sintered samples at loading force of (5N) then using the following equation:-

$$HV = 1.8544 \frac{P}{(d_{av})^2}$$

P : applied load (kg)
d_{av} : indentation of average diameter (mm)
HV : hardness value (kg/mm^2)

3.Resullts and Discussion

Both Ca/P ratio and crystallinity were increased after calcination, where the Ca/P ratio was increased from 1.7 to 1.9 and the height of (HA) peak intensity was also increased. Secondary phases appeared i.e.(β-$Ca_3(PO_4)_2$,$Ca_2P_2O_7$,β-$Ca_2P_2O_7$,$Ca_4P_2O_9$), according to the X-ray diffraction patterns as explained in appendix (1a,b).The existense of secondary phases such as $Ca_2P_2O_7$ and $Ca_3(PO_4)_2$ associated with (HA) will enhance the biological activity of (HA) .The increase in Ca/P ratio is due to the appropriate pH and calcination temperature which leads to more easily increase of Ca/P ratio up to about 1.9 .Such increases in Ca/P ratio may be related to ease of release of $(PO_4)^{3-}$ ions from the solid phase to the liquid phase at high pH and high temperature [18] .

HA phase was the major phase at almost X-ray patterns reflections, also $Ca_2P_2O_7$ is observed at many X-ray diffraction patterns. Other secondary phases indicated were α-$Ca_2P_2O_7$, β-$Ca_2P_2O_7$, γ-$Ca_2P_2O_7$, δ-CaP_2O_6,$Ca(PO_3)_2$,$Ca_3(PO_4)_2$,β-(CaP_2O_6) and $Ca_4P_2O_9$.(EM) pictures of polished surfaces of the sintered HA powder compacts after sintering revealed the process of densification and crystallization of HA , They were used to evaluate the surface defects (cracks and pores) that could be formed in HA samples during sintering with the varying of sintering temperature.

It can be seen in appendix (2 a-e) that intensity peak heights of HA vary with the increase of temperature , and it reaches the maximum height at 900ºC, then decreases at 1000ºC to increase again at 1100ºC and decreases again at 1200ºC. The decrease of the peak heights is due to the formation of different phases .The increase of temperature leads to grain growth at which the maximum grain size can be observed at 1200ºC. It also seems that at 1200ºC the surface crack and other defects become lower, as shown in figure (2,e).

As temperature of sintering increases the linear shrinkage increases because of the evaporation of binder, moisture and the densification [31].The maximum linear shrinkage was 20% at 1200ºC and the minimum L.sh % was 0.67% at 800ºC. The linear shrinkage percentage increases with the increase of temperature because of surface area of fine particles that have their effect on components. After firing, surface area is much reduced [32].Linear shrinkage behavior with temperature is shown in figure (3).

Apparent density and bulk density relatively increase with the increase of temperature as shown in figure (4, 5), because of the increase of water loss

and linear shrinkage due to densification and the differences between densities are affected by chemical composition [33]. Particle size and shape of particles of powder which is varying from semicircular to irregular shapes with different size distribution of small particles .It is obvious that at temperature of 900ºC the apparent density is increased due to the wide presence of (HA , Dx=3.156) depending on the heights of peak intensities and (β-$Ca_2P_2O_7$, Dx=3.128) and ($Ca_2P_2O_7$, Dx=3.09) which have high densities compared with the other phases in 800ºC and 1000ºC .It seems that the appearance of ($Ca_3(PO_4)_2$,Dx=2.87)phase at these temperatures is the reason for being that apparent density lower at 900ºC , and both densities (apparent and bulk) increase again after 1000ºC due to the presence of (HA) and β-$Ca_2P_2O_7$ in high intensities. The maximum bulk density was (2.173) g/cm^3 at 1200ºC and the maximum apparent density was (2.58) g/cm^3 at 900ºC .The minimum bulk density was (1.2) g/cm^3 at 800ºC and the minimum apparent density was (2.27) g/cm^3 at 800ºC.

Apparent and open porosities are decreased with increasing temperature as shown in figure (6,7) due to the reduction in pore volume and the total volume of the sintered compact powder because of the increase in density[34] , while the (OH) as a vapor phase released from (HA) could represent the major reason that caused the remaining micro pores [32].The maximum apparent and open porosities were 47.136% and 47.058% respectively at 900ºC and the minimum apparent and open porosities were 12.379% and 12.57% at 1200ºC. Water absorption decreases with the increase of sintering temperature as shown in figure (8) since the water absorption is proportional to apparent porosity [31].The maximum water absorption was 39.13% at 800ºC and the minimum water absorption was 5.785% at 1200ºC.

At low temperatures fracture strength is relatively high as temperature increases the fracture strength decreases and it reaches its minimum amount (1.15)N/mm^2 at (1000) ° C as shown in figure (9) i.e. fracture at this regime is brittle and occurs by extension of inherent flaws , at all intermediate temperature regime , the strength falls because the formation of low strength phases (β-$Ca_2P_2O_7$,γ-$Ca_2P_2O_7$ and $Ca_3(PO_4)_2$), and the fracture occurs from flaws now produced by limited plastic flow . At high temperatures regime where the strength raises again with temperature increase until it reaches its maximum value (6.57)N/mm^2 at 1200ºC due to the increase in mechanical bonding [34] and fracture at this regime is accompanied by extensive plastic deformation[35].

Hardness increases slightly at low temperatures and then decreases at 1000ºC, then it start increasing at higher temperatures as shown in figure (10). The maximum hardness was (87.4)Kg/mm^2 at 1200ºC and the minimum hardness was (36.2)Kg/mm^2 at 1000ºC .The slight increase in hardness in the first stages of sintering is due to the formation of grain boundaries by atomic diffusion

in solid state sintering process. The decrease in hardness may be attributed to the formation of multi-phases which comprise different thermal expansion coefficient, which may exhibit cracking near to grain boundary phases [31].Hardness increases again due to the decrease in porosity and due to the fracture of the walls between contiguous pores which is lower at high temperatures [36].

4. Conclusions

1-At 1000ºC, a critical point exists in most of the physical and mechanical properties due to the formation of multiple phases.

2-1200ºC, is preferred to get high fracture strength and hardness, where the fracture strength was (6.57) N/mm^2 and the hardness was (87.4) Kg/mm^2.

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Table (1) illustrates the identity of hydroxyapatite.

Chemical formula	**$Ca_{10}(PO_4)_6(OH)_2$**	**Hazard and safety**	**Non flammable**
Abbreviation	**HA**	**Solubility**	**Soluble in mineral acid**
Melting point(ºC)	**1670**	**Insolubility**	**Insoluble in water, ethanol, acetic acid.**
Molecular weight	**Mol. Wt=1004.69**	**Porosity**	**Open, interconnective.**
density	**3.08-3.18**	**Clinical results**	**Available from 10 years of application.**
Ca/P	**1.67**	**Mineral phase**	**CaO<1% , TCP<1%**
Mohs hardness	**5**	**Implant bone connection**	**Enabling the stablishment of positive material connection.**
Refractive index	**1.63**	**biocompatibility**	**Bioactive, osteoconductive**
Solubility constant K_s at 25 ºC	**$6.62*10^{-126}$**	**Crystal structure**	**Hexagonal a=b=9.432, c=6.881**
synonyms	**Tricalcium phosphate, tricalcium orthophosphate tribasic calcium phosphate, bone ash.**		

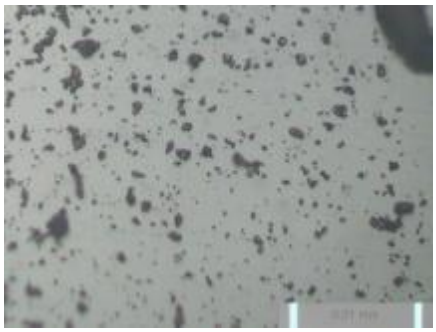
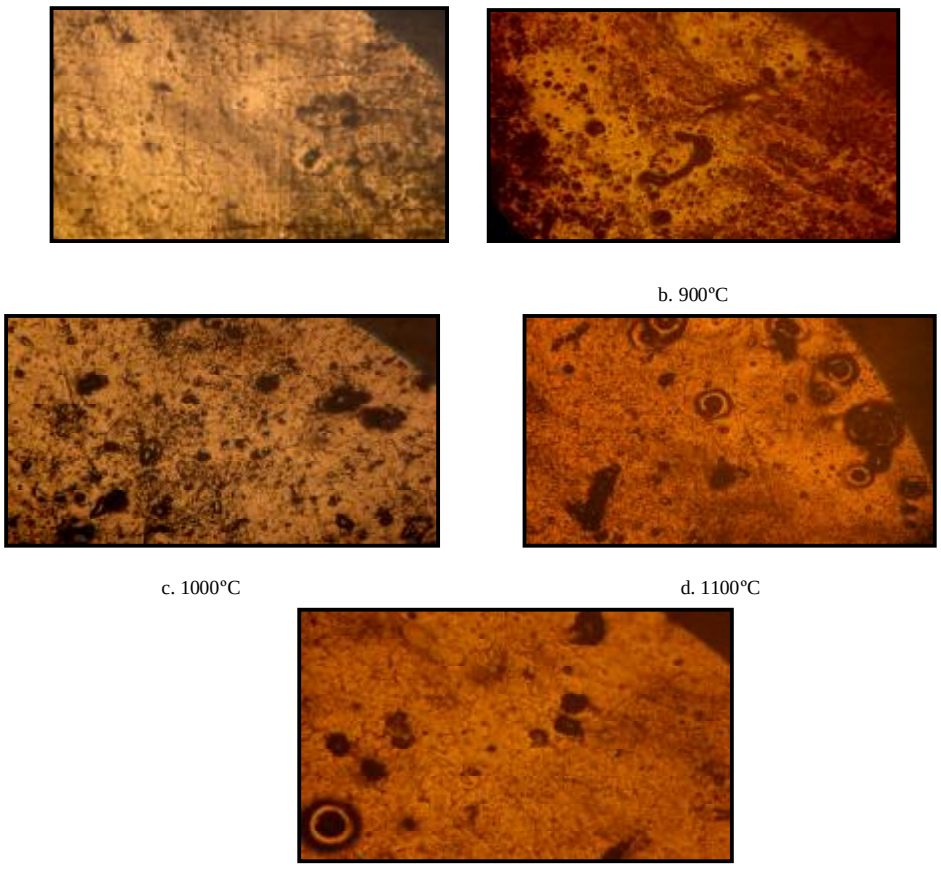


Figure (1) shows the particle shape of fabricated HA



b. 900°C

c. 1000°C

d. 1100°C

Figure (2) EM pictures of sintered HA powder compact at different Sintering temperatures and at a magnification of (X63) .

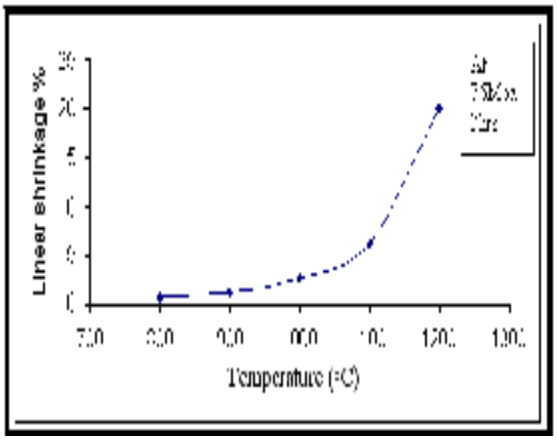


Figure (3) shows the effect of sintering temperature on linear shrinkage of sintered (HA) powder compac

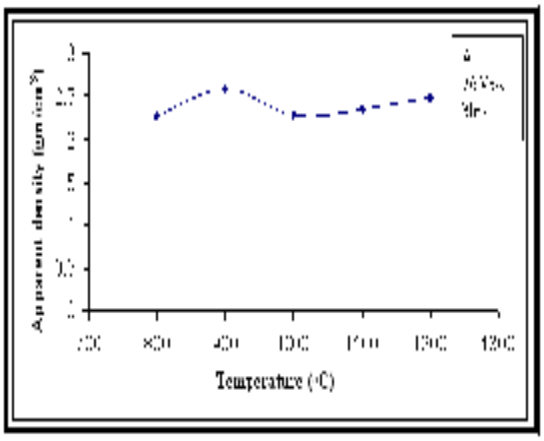


Figure (4) shows the effect of sintering temperature on apparent density

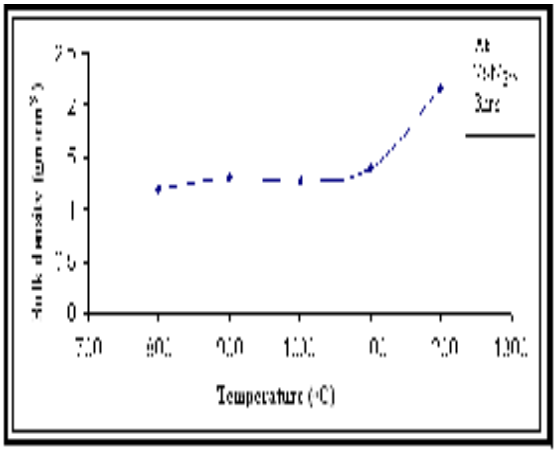


Figure (5) shows the effect of sintering temperature on Bulk density of sintered (HA) powder compact.

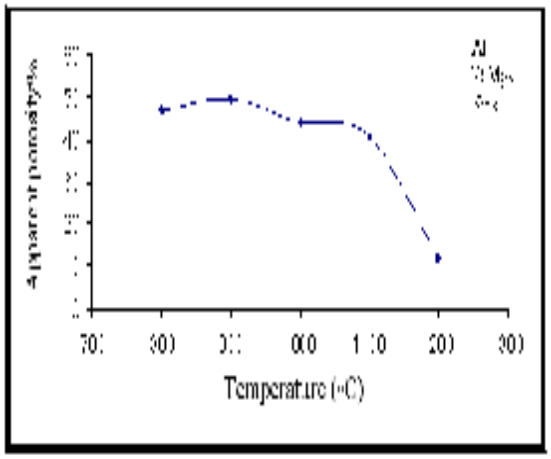


Figure (6) shows the effect of sintering temperature on apparent porosity% of sintered (HA) powder compact.

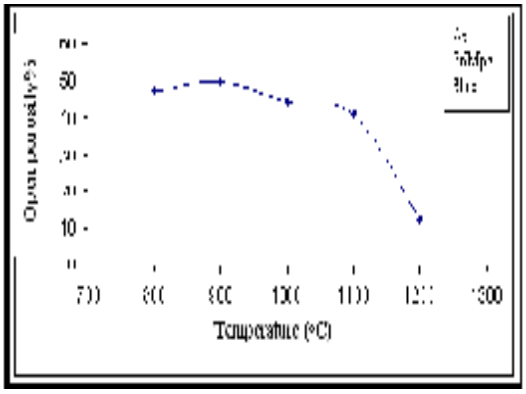


Figure (7) shows the effect of sintering temperature On open porosity% of sintered (HA) powder compact.

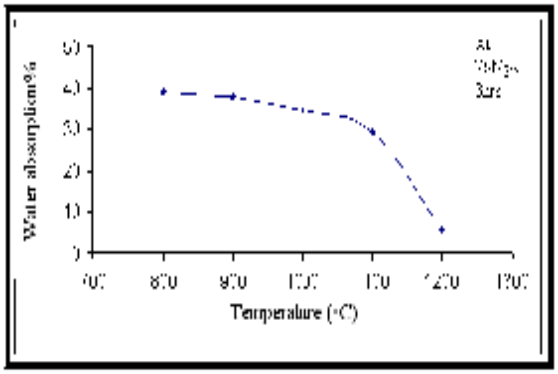


Figure (8) shows the effect of sintering temperature on water absorption of sintered (HA) powder compact.

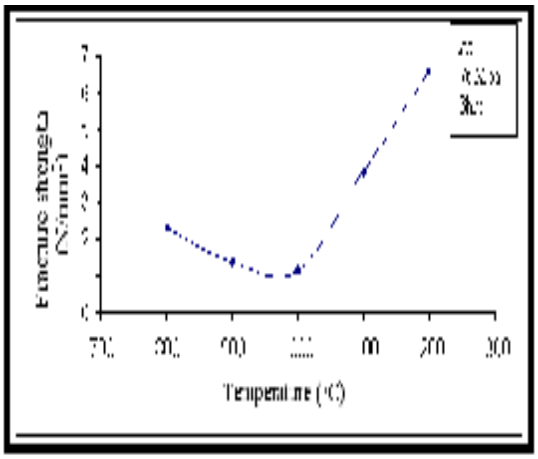


Figure (9) shows the effect of sintering temperature on Fracture strength of sintered (HA) powder compact.

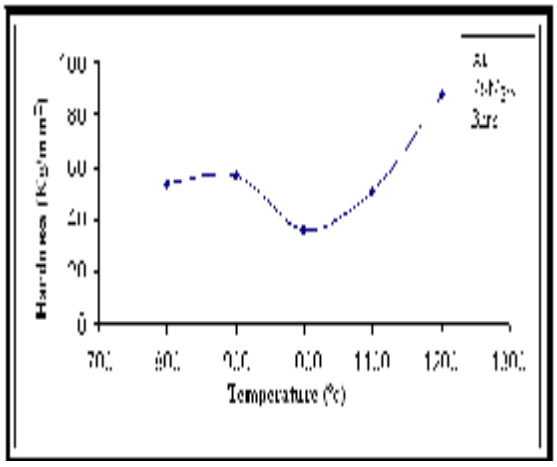
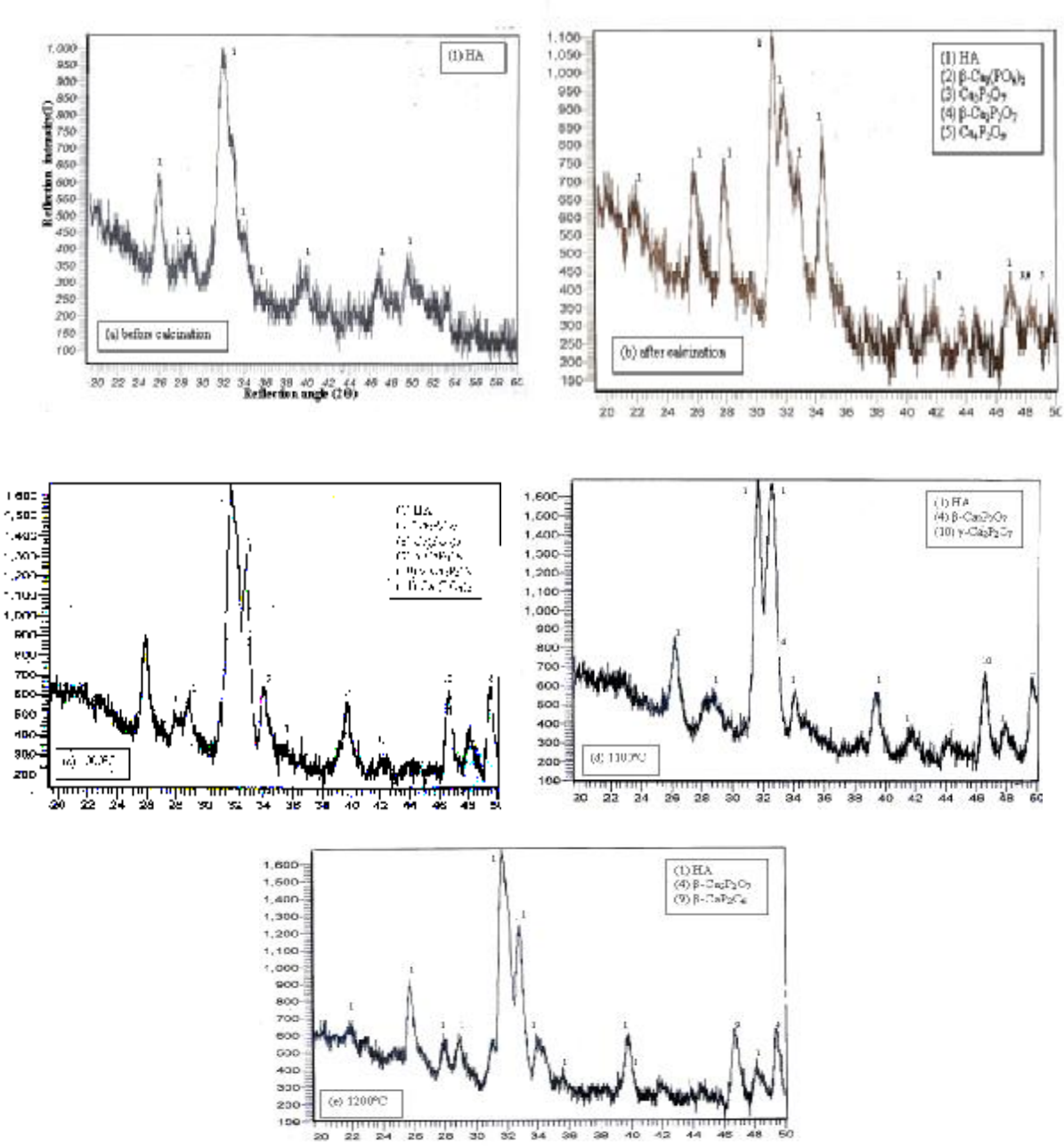


Figure (10) shows the effect of sintering temperature on Hardness of sintered (HA) powder compact.



Appendix (2-a to e) shows x-ray patterns for Hydroxyapatite powder compact samples at temperature range (800-1200)°C .