Ibn Al-Haitham Journal for Pure and Applied Science | مجلة إبن الهيثم للعلوم الصرفة و التطبيقية
No. 3 Vol. 25 Year 2012 | 2012 السنة 25 المجلد 3 العدد

Preparation of Fine Hydroxyapatite (HA) Powder And Its Characterization As Ceramic Biomaterial

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Received in: 27 May 2009 Accepted in: 21 January 2012

Abstract

Biomimatic basic calcium phosphate ,known as Hydroxy apatite(HA), was synthesised using calcium acetate and disodium phosphate dilute hot (boiling) solution of both salts were added slowly to each other. A colloidal suspension formed was aged for few days, then boiled for one hour to induced setting and coagulation of preapatite, and left to cool until they reached room temperature.

The precipitate was filtered and washed with distilled water, then ethanol and dried.

The dried sample was calcined at 1100^{o}C.The obtained solid lumps were then crushed reground fine powder and analyzed for calcium to phosphorous ratio. X-ray analysis was carried out to examine the solid sample. Chemical analysis for phosphorus and calcium had been carried out using standard method.

The results proved that the calcined sample used in this investigation where Hydroxyapatite .
Key word : Hydroxy apatite synthesis.camical anaylysis

Introduction

Calcium phosphate materials have received attention in reconstructive surgery because they are biocompatable and capable of forming tight bonds with surrounding bones.

X-ray studies established that the major constituent of all mineralized tissue was some form of calcium phosphate called “Hydroxyapatite, HA”.

The inorganic portion of mineral tissue contains ions other than that found in the hydroxyapatite formula, $Ca_{10}(PO_4)_6(OH)_2$, where calcium and phosphorous are the main elements beside that sodium , potassium, aluminum, citrate, sulphate, bicarbonate, chloride and fluoride are also found.

Hydoxyapatite has received special interest in the field of biomedical engineering [1]. Synthetic HA 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.

HA has attracted much attention as a suitable material for damaged teeth over the past several decades. Also it has potential to become the “coating of choice” for metal surgical implants (most often made of titanium and its alloys, or stainless steel) and it’s an attractive alternative to the polymer coating.

The submicroscopic crystal of calcium phosphate in bone resembles the crystal structure of synthetic hydroxyapatite.

Porous hydroxyapatites have become highly regarded biomaterials in clinical applications, but it has inadequate mechanical properties, low mechanical strength and low toughness, sofar that is now used as a matrix for transplant atom of osteoprogenitor cells.

Hydroxyapatite has long been investigated and used as bone implant material due to the properties of bioactivity, biocompatibility and osteoconductivity[2].

Method of Preparation

In this investigation basic hydroxyapatite was prepared as ultra fine powder. This is only possible when the reaction is carried out at high temperature and form dilute solutions. A hot solution of phosphate as disodium hydrogen phosphate was added drop wise to a boiling solution of calcium acetate.

The resulting colloidal solution was left to settle and age for at least three days, better in a thermo stated incubator.

The solution, then, boiled for one hour and filtered. The precipitate was washed with hot water and ethanol, and then dried in on oven at 120^{o} C.

Few grams can be prepared in this way, thus the procedure was repeated several times to collect about twenty gram of fine precipitate the dried sample was calcined at 1100^{o}C to get calcined apatite.

A sample was taken for chemical analysis to check phosphorus to calcium ratio and impurities.

Results and Discussion

Apatite Solubility

Extensive solubility studies on a well characterised synthetic hydroxyapatite have shown that this material seems to depart from the laws of solubility for difficulty soluble compounds.

It was found that the solubility of this compound was different when determined from the under saturated equilibria and that determined from the saturated equilibria.

Spontaneous crystallization when mixing solutions of calcium and phosphate was determined as a function of pH.

The solid formed had a molar ratio for Ca/P equals to unity, this solid is stable below a pH of 6.9 ; but at higher pH values, it rapidly hydrolyses to hydroxyapatite , giving Ca/P molar ratio equals to 1.6 .The solutions in which the hydroxyapatite was suspended had varying solubility.

The presence of ion such as carbonate, magnesium and sodium may explain the increase in calcium and phosphate level oven the solubility of apatite. This leads to format ion of calcium deficient apatite [3,4].

X-Ray Analysis

The prepared powder was calcined at 1100^{o}C for at least one hour. X-ray diffraction analysis was carried out for dried sample and calcined sample as shown in figures (1, 2, and 3).

XRD results reveal that major characteristic peaks of HA appear in the region of approximately 25, 27, 30, 32, 34, 46, 53, 49, 120 degree. HA (a,b=9.418Å ,c=6.884 Å) are well agreed with lattice parameters of prepared samples (a,b=9.44 Å ,c=6.87 Å). X-ray analysis was preformed by SHIMADZU model chemical analysis was preformed to confirm the (Ca/P)molar ratio(1.6) of the synthesized powder.

X-ray diffraction analysis identified the hydroxyapatite products, air dried and oven dried powder, as poorly crystallied powder continuing water and carbonate impurities as shown in figures (1,2,3).

The X-ray diffraction pattern of the calcined samples shows distinct reflection lines of well defined crystalline compound as shown in figure(3) [5].

X-ray diffraction studies established that the major constituents of all mineralized tissue were some form of hydroxyapatite .The inorganic portion of mineral tissue contains ions other than those found in the hydroxyapatite formula.

Some secondary phases are formed during HA sintering process there are related to its:Original ca/p ratio, chemical composion and sintering temperature .

XRF analysis showed the elements Ca, p and K as major elements as shown in figure (4) for hydroxyapatite powder.

It is necessary to decide whether the sodium, magnesium, potassium, chloride, carbonate and water found in the mineral were substituted in a single apatite molecule or admixed in some manner. Thus complete chemical analysis of these ions is essential pant of the work.

References

1. Ttistan, B. and Owen, S. (2001), Materials for Biomedical Engineering, J. Mater. Sci. And Eng. UNSW, 1-15.
2. Parshant, N. ; Charles S. ; Dong H. ; Ana O. and Daiwan, C. (2005) , Nanostruuture Ured – Calcium Phosphates for Biomedical Application,Act Biomaterial, 1, 65-83.
3. Caross, K. and Bernadt, C. (2002), Mineralogical Society of America, 48, 634 – 672.
4. Zhang, B. ;Fukne ,Y. ;Wang, Y. and Shipu, L. (2002), Morphology and Formation Mechanism of Hydroxy apatite Whiskers from Moderately Acid Solution, Mat. Research, 6, 8 – 25.
5. Prabakaran, K.; Balamurugan, A. and Rajeswari, S. (2005), Develompent of Calcium Phosphate Based Apatite from Hens Eggshell, Bull . Mater. Sci., 28, 115-119.

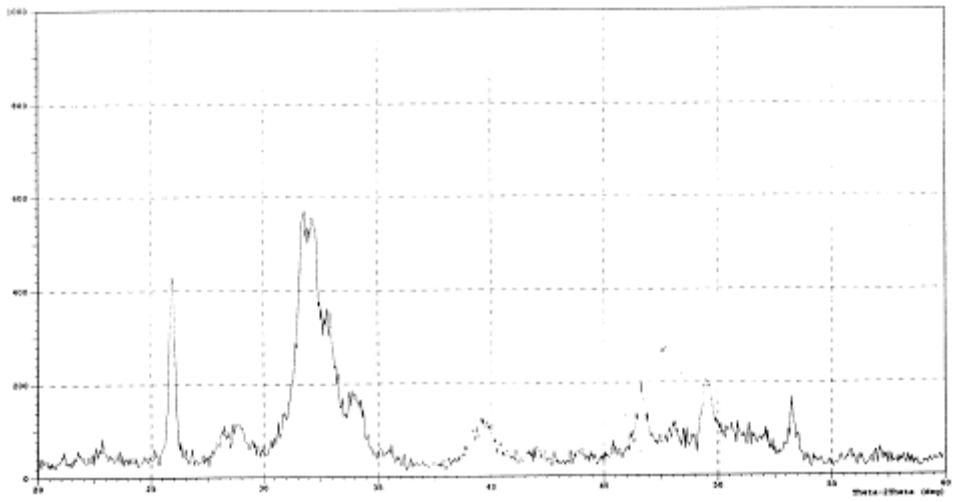

Fig. (1): Dried hydroxyapitite powder, prepared with molar ratio (1:1)

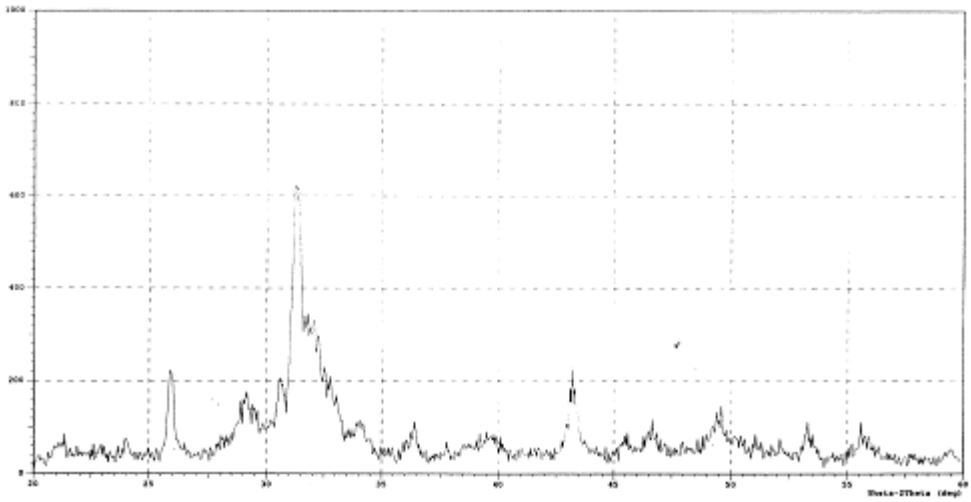

Fig. (2): Dried hydroxyapitite powder, prepared with molar ratio (2:2)

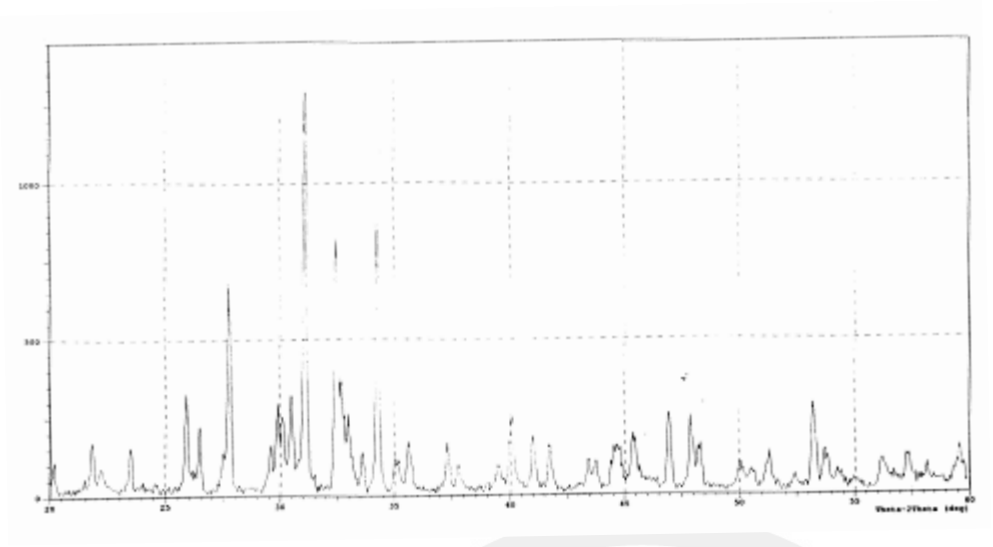

Fig. (3): The calcined Hydroxyapitate powder in 1100^{o}C, prepared with molar ratio (1:1)

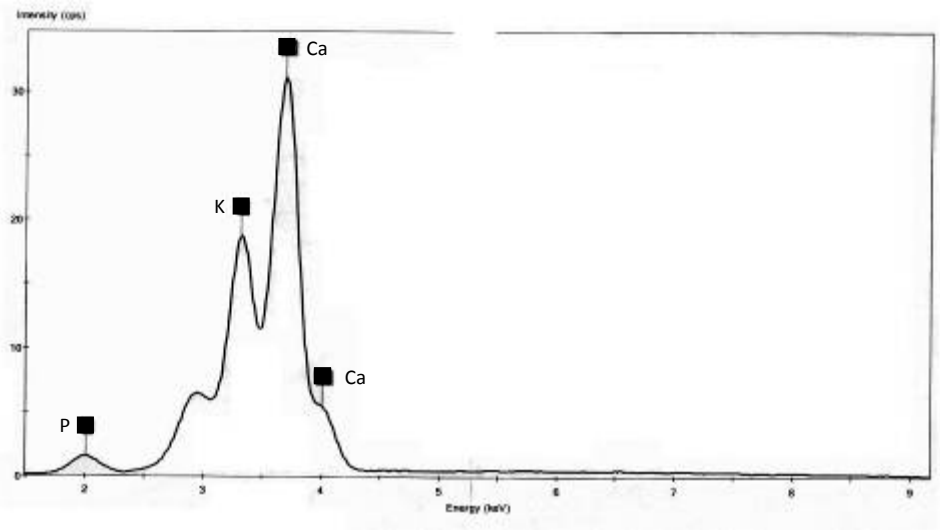

Fig. (4): XRF analysis of wet HA sample prepared with molar ratio (1:1)

تحضير مسحوق الهيدروكسي ابتات الناعم وتوصيفه مادة سيراميكية حياتية

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استلم البحث في 27ايار 2009، قبل البحث في 12كانون الثاني 2012

الخلاصـة

فوسفات الكالسيوم القاعدية الاحيائية المعروفة بهيدروكسيد ابتايت (HA) حضرت مختبرياً باستعمال محاليل مسخنة الى درجة الغليان ومخففة لكل من املاح خلات الكالسيوم وفوسفات الصوديوم، اذ اضيف محلول احد الملحين الى الاخر قطرة بعد قطرة وضمن مدة زمنية تعتمد على حجم المحلولين .نتج من هذه الاضافة محلول غروي عالق، وجرى تعتيقه لبضعة ايام، ولغرض ترشيحه تم تسخينه حتى الغليان لساعة واحدة لاجل حث عملية التخثروالترسيب ثم ترك المحلول بعد ذلك ليبرد الى درجة حرارة الغرفة. فصل الراسب عن المحلول بالترشيح وجرت عملية غسل الراسب بالماء المقطر والكحول الاثيلي وترك ليجف. الانموذج المجفف حرق بدرجة 1100 ^{o}C ثم طحن يدويا ليصبح مسحوقا ناعما.

أجريت عملية تحليل كيمائي لـ (Ca/P) وحسب الطرائق القياسية للانموذج الجاف والانموذج المحروق، كذلك تم تحليل الانموذجين بطريقة حيود الاشعة السينية.

اظهرت نتائج الفحص والتحليل تكون الهيدروكسيد ابتايت في الانموذج المحروق سواء في التحليل الكيميائي او بتحليل حيود الاشعة السينية.

الكلمات المفتاحية: هيدروكسي ابتايت ،تحضير،تحليلات كيميائية