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Characterization of Structural Properties of Tri-calcium phosphate at different Temperatures

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Abstract
In this research, calcium phosphate beta phase was prepared under special conditions, where catalyst calcium carbonate prepared from cow bones and treated with phosphoric acid at a concentration of 87% was used. Wet chemical precipitation method was used in the preparation process, where the acidity the scalar=11, and the particle and granule formation process of the compound was controlled. By the amount of phosphoric acid added. Structural examinations were performed using X-ray Diffri techniques and SEM, in addition to the X-ray energy dispersive spectroscopy technique. The preparation of the compound was adopted employing various heat treatments to show a variety of patterns and shapes of nanoparticles.

Key words: Calcium Phosphate, Catalyzed Calcium Carbonate, Cow Bones, Chemical Precipitation, Structural Tests

Introduction

At the present time, the use of bone grafts is the preferred method for treating bone defects resulting from disease, trauma, or tumor resection, and this method is associated with mechanical problems such as the patient's condition and age .and the possibility of transferring the disease from a living donor, a bone scaffold is used with stem cells of the body (1-2-3-4), and although these results achieved indicate that it is a safe recovery of bone defects it has not been achieved ,among these defects is the decrease in the production of bone stimulatory cells and the lack of amulets among the progenitor cells. In light of this, Many option have been proposed put forth . By combining stem cell culture and cell encapsulation in microspheres with a scaffold material,this issue can be resolved. (5-6-7)It has high quality properties as the scaffolding material is biologically active, biodegradable, moldable, easy to apply, and has a certain porosity .Suitable for the body to allow tissue growth and fixation. It is a recently developed calcium phosphate cement. It was found to meet the likethusRequirements, because it is good for bone growth, works as a good conductor, and is biodegradable due to the possibility of reshaping the bone. It can also be injected and includes microspheres with the possibility of manufacturing it.In large and precise pores, in order to allow the penetration of the nutrients needed for good bone growth and the removal of waste products that damage the bone, different types of caps were used, suchas Di-calcium phosphate, pyrophosphate, tetra-calcium phosphate, and tri-"calcium phosphate" (TCP).Which turns into hydro-xyapatite and it is amorphous at the start of the preparation, but calcium – phosphate during the transition, ions may be released that could be damaging tocell culture, as it is noticeableNo problem has been observed for cement materials in vivo because toxic substances during the initial Stage of healing,are transported away.Additionally ,thisphenomenon explain the contradiction of studies, that have been conducted in stem cell culture[8-9-10].

Experimental Method

Calcium hydroxide (0.3779 mole, 28 gram) was dissolved in (300 mL) of deionized water and stirred for one hour to produce a white turbid an over saturated solution. Concentrated phosphoric acid (0.1326 mole, 13 mL) was added drop wise to the resulted calcium hydroxide aqueous solution and stirred for another six hours. The pH was tested during the addition of the concentrated acid using pH meter type (PHS-3C) and showed pH 12.19 at the laboratory temperature. Furthermore, the resulted solution was left stand overnight to produce soluble and insoluble phases. A simple filtration process was applied to

separate the desired product. Finally, the white fine powder was dried at 100 °C for three hours to produce (24 gram, 85.71 %) of $Ca_3 (PO_4)2$ pure sample.

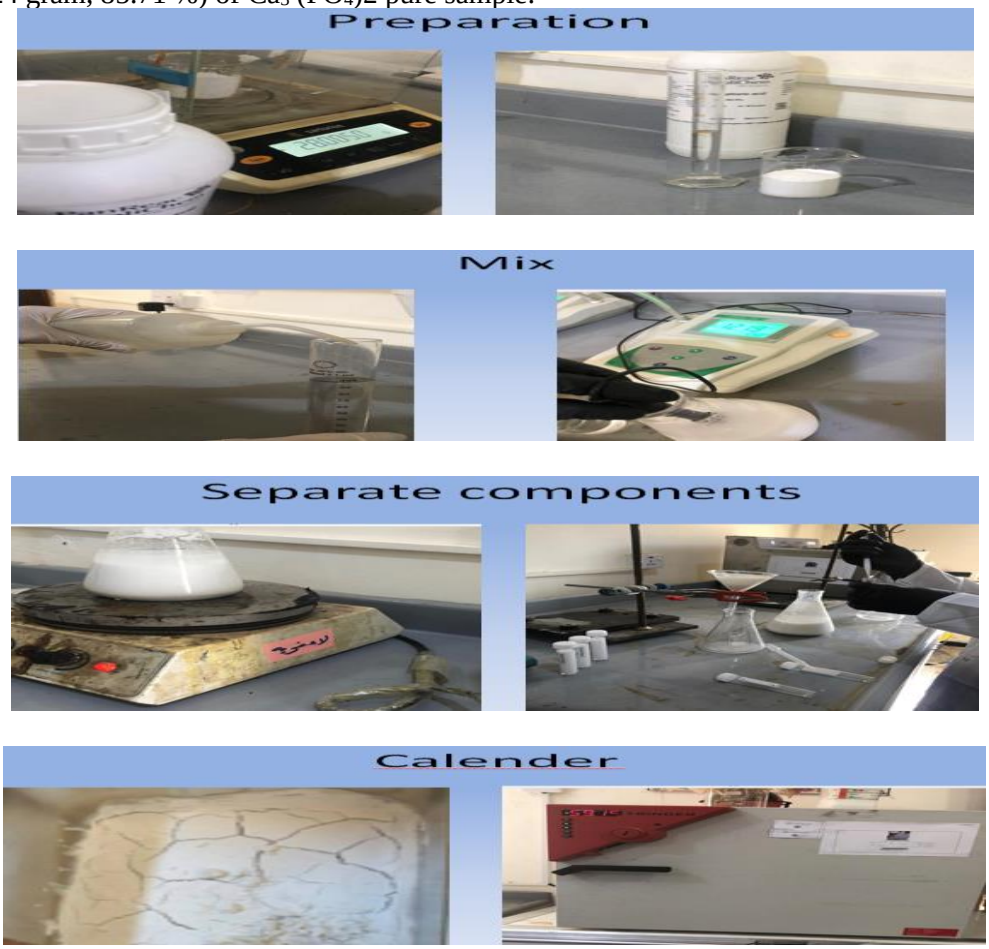
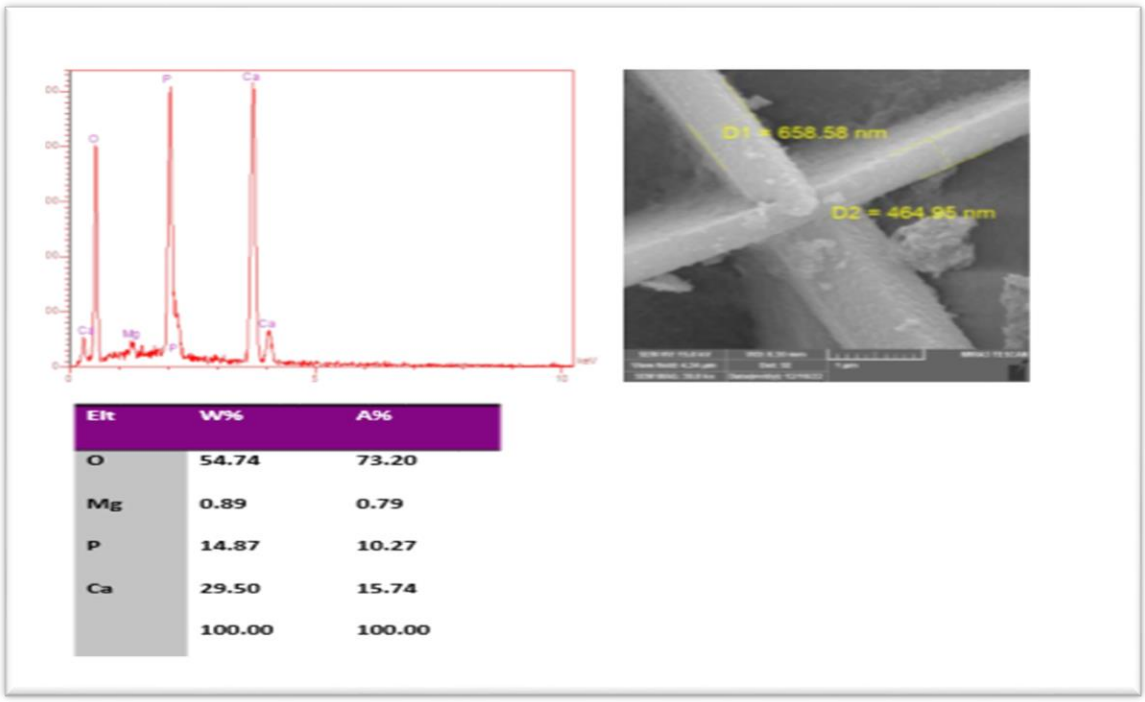


Fig. 1: Photographic of Experimental of Synthesis Ca \ P Nano powder.

RESULTS AND DISCUSSION

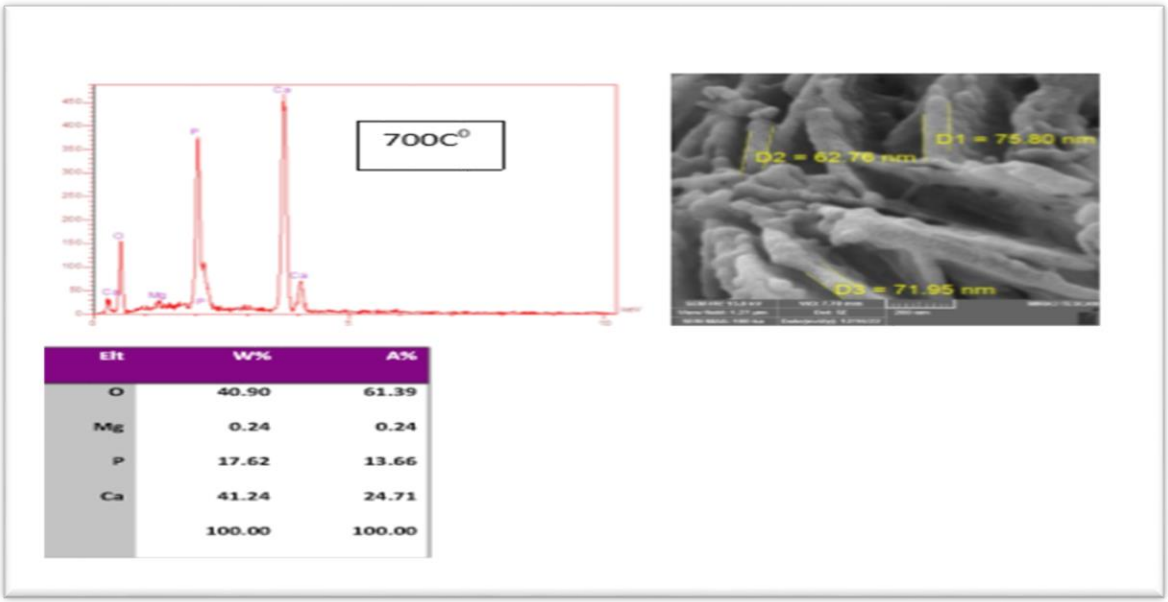
Fig.2. Shows the micrograph of hydroxyapatite obtained by SEM.The material's range of particle sizes is (D1=658.58, D2=464.95)nma high particle size dispersion being indicated. The elemental composition of the final synthesized white powders was determined to be as follows, according to the visual findings of the semi-quantitative elemental chemical analysis produced by the EDS method,bein6000^{0}C(O 54.74 wt%, P 14.87 wt% and Ca 29.50wt%,Mg0.89wt%). These results reveal high purity of the calcium phosphate obtained by the continuous precipitation method since no other chemical element was detected by the EDS technique when hydroxyapatite was analyzed in different zones of the sample.



Elt	W%	A%
O	54.74	73.20
Mg	0.89	0.79
P	14.87	10.27
Ca	29.50	15.74
	100.00	100.00

Fig. 2: EDX and SEM– 600 ^{O}Cdata of Ca \ P Nano powder

Fig. 3. Possesses a sphyrical-crystalin similar to that of simpetals,and agree with[11]. Additionally, there is a needle structure that is connected to spinal development. Figure 2 from the EDX study displays the weight percentage of the mean chemicals. (40.90wt% O, 0.24wt% Mg , 17.62wt% p,41.24wt%Ca)and it can be shown that the preparation procedure is accurate in accordance with the stereo-chemical shown in fig 2. The mean grain particle size is (D1=75.80, D2=62.76, D3=71.95)nm, is fusiform is high homogeneity in the anyling and sinering at 1000^{o}C.

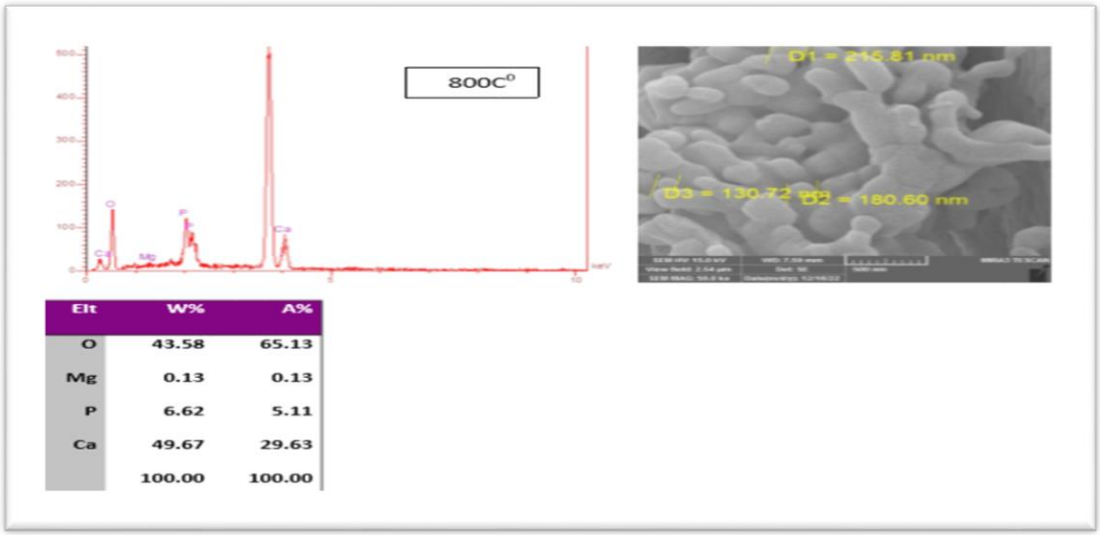


Elt	W%	A%
O	40.90	61.39
Mg	0.24	0.24
P	17.62	13.66
Ca	41.24	24.71
	100.00	100.00

Fig. 3: EDX and SEM– data 700 ^{O}C of Ca \ P Nano powder.

Figure. 4. Represent FESEM of specimen (TCP), It is possible to see the homogeneity of the structure, the uniform distribution of the particles, and the grain size (D1=215.81,D2=180.60,D3=130.72)nmin

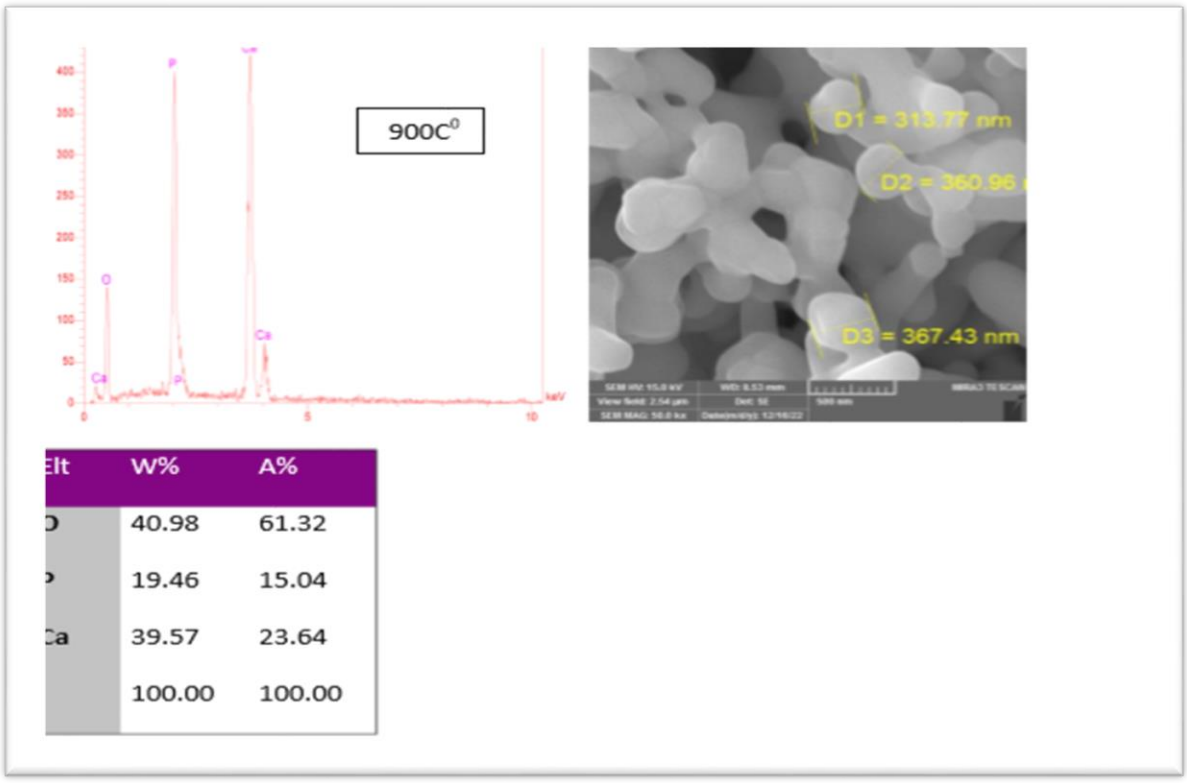
figure 3, shows the level of concordance between(theo. and Experm.)demonstrating the effectiveness of the preparation and EDX the Sample has Hexo-gonalcrystaline with agree[12,13].



Elt	W%	A%
O	43.58	65.13
Mg	0.13	0.13
P	6.62	5.11
Ca	49.67	29.63
	100.00	100.00

Fig. 4: EDX and SEM– data 800^{O}C of Ca \ P Nano powder.

Figure. 5. Shows the specimen's SEM microstructure and EDX after it has been polished and sintered at 1000 ^{0}Cfor 5 hours. It is evident from the microstructures that there are both smaller and bigger grains. Larger grains accumulated atop smaller seed. the size of porosity and quantity depending on sintering temperature, because in the liquid phase sintering the densification process achieve in three stages rearrangement of the particles, second stage dissolution of the particles and precipitation and third stage solid state anything particles. has circular crystal structure, comparable to round petals, and this form was identical shown by [11]. The Energy Dissipationanalitic fig. 4.Explain the percenaget of the compo-unds(40.98wt% O, 19.46wt% P and 39.57wt% Ca)and it can be shown that the preparation procedure is accurate in accordance with the stereochemical calculation as shown in fig.4. Grain is (D=1313.77,D2=360.96,D3=367.43) nm,



Elt	W%	A%
O	40.98	61.32
P	19.46	15.04
Ca	39.57	23.64
	100.00	100.00

Fig. 4: EDX and SEM– data 900 ^{O}C of Ca \ P Nano powder.

The system (TCP) is depicted in figure 4 with the development of homogenous cordierite phases and grain sizes nano particles, as shown by the EDX test in fig. 4.with the highest peak intensity for (O) element, followed by (p,ca) sequentially. Specemine has hexa.crys. of the same form as agree with[12,13].

Fig.6.X-ray diffraction are performed at an angle range from 10-80o by using LabX XRD-6000 X-ray with speed scanning 5°/min from Shimadzu, operated at 40 keV and 30 mA. CuKα radiation tube λ=1.54A ^{0}.

The data of X- Ray was plotted by matched with JCPDS software to identify the phase changing. The grain size has been calculated (Dhkl) by using corrected Debye-Scherrer formula:

$$G \cdot S = \frac{k\lambda}{\beta coscos\,\theta}$$

Where

β = FHM (full width at half maximum)

K = 0.9

θ =(angle of diffriection)

λ = 1.54A^{0}

shows the XRD pattern of the obtained powders: the examination of the figure indicates that the synthesized material was only a crystalline single phase. The This synthetic material has a hexagonal hydroxyapatite structure, as shown by indexation calculations, with unit cell parameters determined the XRD of "Ca10(PO4)6(OH)2" (a=b=9.385A^{0}),(c=6.870A^{0}) acquired using the method given by [14,15]. The CP that was produced had all nanocrystalline grains with an average size of around 75 nm, in accordance with the Sherrer equation. The 2Θangle range between 20^{0} and 60^{o} shows the sharpest and most vivid lines. These lines correlate with the hydro-xyapatite-related XRD lines that were reported in the JCPDS 9-0432 file.

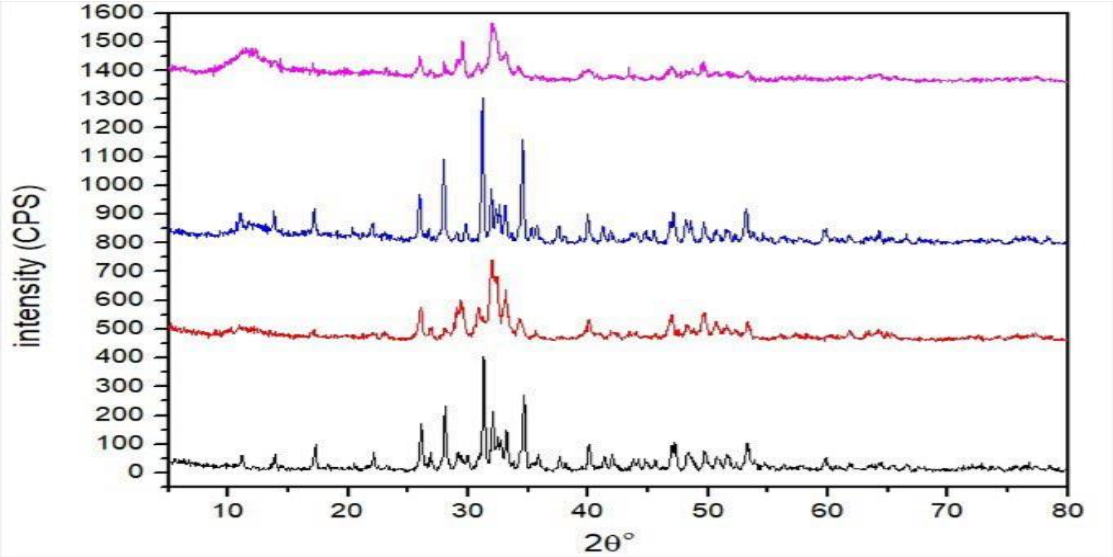


Fig .6. X-ray Diffraction Examinations

Conclusions

Increasing the samples' calcination temperature increases the crystal stability and purity. Therefore, a temperature of 1000°C was adopted in preparing Ceramics nanocomposites.

X-ray diffractionthe growth of a hydroxyapatite layer on the samples' surfaces. It was noticed by the diffraction pattern peaks and the difference in intensity before and after immersion.

The results of surface morphology and the shapes of the nanoparticles, nanoparticle size, and the surface area of their distribution were Verification by scanning electron microscope (FE-SEM), where the (FESEM) images show or has a dendritic form, MgO has a spherical shape and Al2O3 have cluster shape, and the Nanocomposite have uniform aggregation.

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