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Effect of polyethylene glycol and acetic acid on morphology of nanoparticle Hydroxyapatite synthesized by sol-gel

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Abstract: Hydroxyapatite is a ceramic with wide application in medicine and dentistry due to most important feature is bioactivity. So, researchers have tried to customize its properties such as bioactivity, mechanical strength, solubility and sinterability by controlling its composition, morphology and particle size. In this research the morphological changes of nanoparticle hydroxyaparite in presence of polyethylene glycol and acetic acid synthesized by sol-gel technique is presented. Nanocrystalline powders of hydroxyapatite (HA) were prepared from $Ca(NO_3)_2{\cdot}4H_2O$ and $PO(OC_2H_5)_3$ as calcium and phosphorus precursors, respectively. Polyethylene glycol and acid acetic were added as modifier. The obtained samples were characterized through X-ray diffraction (XRD), scanning electronic microscopy (SEM) and TEM analysis. Their morphology of hydroxyapatite depend on the kind of modifier, polyethylene glycol create preferential growth of particles in result morphology become the needle-shaped, while acetic acid does not affect on the shape of particles but it create agglomerate.

Keywords: Hydroxyapatite, Polyethylene glycol, SEM, Needle shape

Introduction

Hydroxyapatite is the most ubiquitous calcium phosphate that has assumed substantial interestand importance because of its chemical similarity to the natural calcium phosphate mineral present in biological hard tissues. Different clinical applications involve repair of bone defects,bone augmentation, as well as coatings for metallic implants [1,2]. The important requiremen to famaterial designed for bone substitution and/or repair,is the ability to create a bond with the host living bone [3]. Hence,researchers have tried to customize its properties such as bioactivity,mechanical strength,solubility and sinterability by controlling its composition,morphology and particle size [4,5]. The chemical,structural and morphological properties of synthetic hydroxyapatite can be modulated by varying the method and conditions of synthesis.There are several different synthetic methods used to generate HAP as reported in the literature including chemical precipitation [6], hydrothermal techniques [7], sol–gel [8], solid state and mechano-chemical methods [9,10]. Among the alternative methods, sol–gel approaches have attracted much attention recently because of its well known advantages, which include homogeneous molecular mixing, low processing temperature, the ability to generate nano-sized particles and the tremendous flexibility to generate nanocrystalline powders, bulk amorphous monolithic solid sand thin films [11]. In the morphology-controlled synthesis of hydroxyapatite nano fibers [12], plates [13], nanoparticulates [14] and nanorods [15], several organic modifiers are used such as ethyleneglycol [13], cetyltrimethyl ammoniumbromide(CTAB) [12,16], poly vinyl alcohol [16], citric acid [17] and ethylene diamminetetra acetic acid (EDTA), [18] etc. The present work carried out elaborates on morphology controlled synthesis of nano structured hydroxyapatite by sol–gel method using calcium nitrate tetra hydrate and thrietyle phosphate as starting materials with the aid of polyethylene glycol and acid acetic as modifier.

Experimental

triethyl phosphate ((C2H5O)3PO, TEP,) diluted in ethanol and was added the appropriate amount of water and was stried for 24 h to be hydrolyzed. This solution was added to solution Ca(NO3)2·4H2O .the mixture was stried for 24 h and was kept 48 at room temperature. four sample were prepare that their difference is at addeding modifier.so this deitel is shown in table 1. The products in four tests were dried at 80 for 24 in dry oven then calcined at 600 tempereture.

Table 1.type of synthesis sample

Sample code	modifier
a	No any modifier
b	Polyethylene glycol
c	Acid acetic
d	Polyethylene glycol+ Acid acetic

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In order to study Phase identification of the calcined gels was performed using the X-ray diffractometer (XRD, Philips, X'pert Pro, Cu K) at a scanning speed of 1◦ 2θ min−1 from 10◦ to 60◦. Scanning electron microscopy (SEM, S 360, Oxford-England) was used for

charachterise of morphology and elemental analysis of Ca and P.

Results and Discussion

XRD analysis

The XRD patterns of produced sample that don't use any modifier and sample produced that using polyethylene glycol and acid acetic as modifier, were shown in figure 1. The patterns indicate the presence of hydroxyapatite.in both sample. No characteristic peaks of impurities, such as calcium hydroxide and calcium phosphates were observed, meaning that phase pure HAP was prepared under the present experimental conditions. The mean crystallite size (D) of the particles was calculated from the XRD line-broadening measurement from the Scherer equation. The crystallite size was 40-50 nano meter.

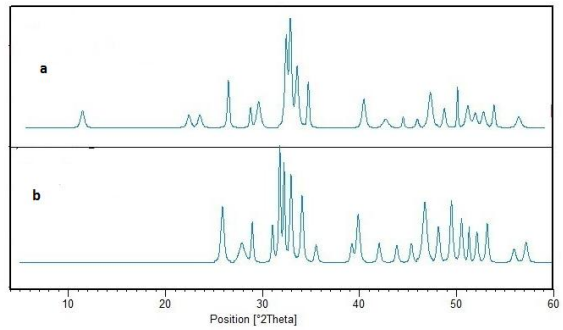
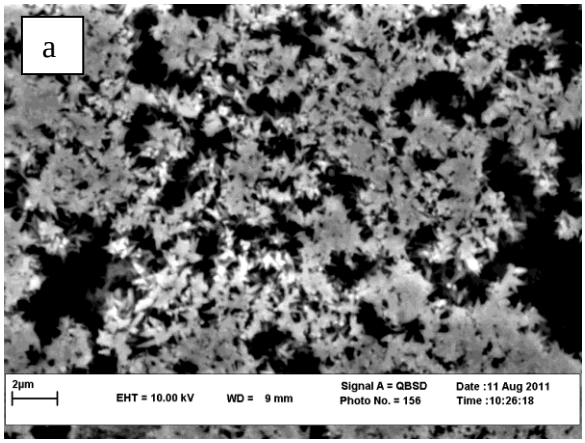
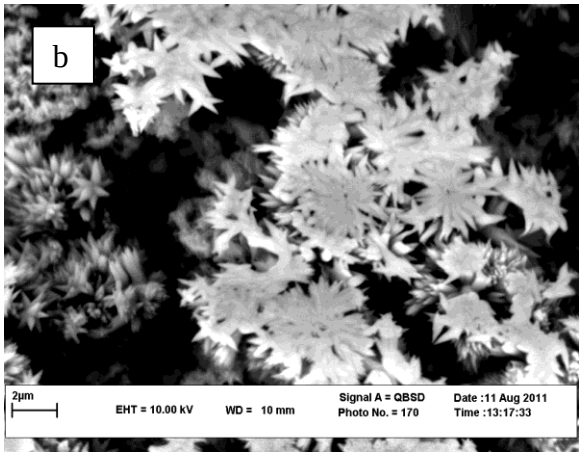


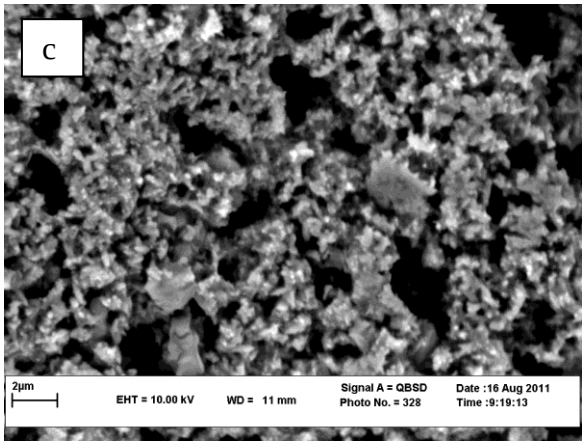
Fig1: XRD patterns of HA powder (a) no using modifier (b) using modifier

Morphology and particle size:

The using of modifier was important factor influenced the morphology of HAP. As shown in Fig. 7. HAP particles prepared by using modifier and not modifier had different morphologies, using poly ethylene glycol create needle-like morphology that aggregation of a lot of particle make a flower shape (fig.b) but using acid acetic create cauliflower morphology.(fig.c) also contemporary using acid acetic and poly-ethylene glycol create complex morphology ,some particles are needle like but another are spherical and very agglomerated, also some another are plate shape.(fig.d).







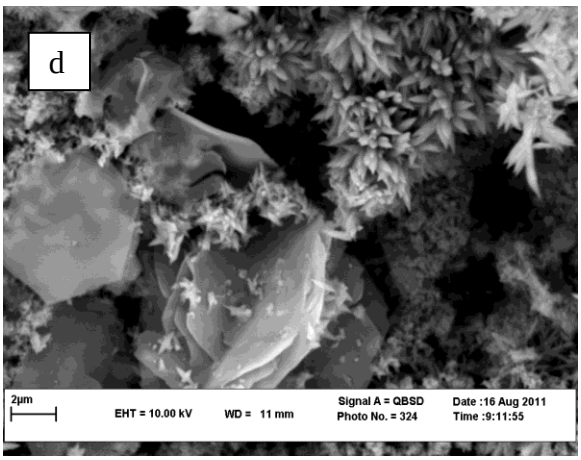


Fig 2. SEM image of calcined HA powders (a) no using modifier (b) poly ethylene glycol modifier (c) acid acetic modifier (d) poly ethylene glycol and acid acetic modifier.

Conclusions

Nano particle hydroxyapatite were produced by sol-gel method and. effects of polyethylene glycol and acid acetic were investigated on the morphology. Using polyethylene glycol create needle shape hydroxyapatite but using acid acetic create more agglomerate that particles don't have regular shape and their shape are similar cauliflower. But using together polyethylene glycol and acid acetic create a complex morphology In result type of modifier have important effect on morphology of hydroxyapatite.

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