

Preparation and Characterization Bioactive Properties of Bioceramic Composite (Zinc Oxide/Hydroxyapatite)

Elham Abd Al-Majeed and Ola Saleh Mahdi

Department of Ceramic Engineering and Building Materials, College of Materials Engineering,
University of Babylon, Hillah, Iraq

Abstract: Zinc oxide offers many benefits for growth of bones and an antimicrobial action for implants, promotes bone development inhibits bone resorption, offers antimicrobial resistance. The effect of Zinc Oxide (ZnO) on the bio behavior of Hydroxyapatite (HA) was estimated. Extraordinary pureness zinc oxide was supplementary in proportions of (10, 30 and 50%) by weight. ZnO/HA displayed In vitro tests samples soaking for 14 days in Simulated Body Fluid (SBF) appropriate to approximation all the alteration in the several elemental investigation of the (HA) after soaking in (SBF) such as Ca, P, O, ..., etc. and their values were determined by using Energy Dispersive Spectroscopic (EDS), weight loss and pH. The HA composite reinforced with 50 wt.% ZnO showed high bioactivities and lowest weight loss.

Key words: Zinc oxide, Hydroxyapatite (HA), degradation, EDX, SBF, antimicrobial

INTRODUCTION

An alternative way to prepare hydroxyapatite without need to excess in energy and heat which leads to many problems in materials before and after preparation like structure, microstructure, thermal stability, also, physical and mechanical properties (Jin *et al.*, 2011). Mechano-chemical offers all of these parameters, moreover, it is reflect perfect methods to avoid many problem due to excess in heat. The milling and chemical reaction are the main factors to prepare any materials (Siriphannon *et al.*, 2002). Therefore, it must give attention to the milling ingredients like time of milling and ball to powder ratio and ball size. To get flawless and suitable result, we need to use many materials help to, complete the reaction (Elliot, 1994) and the crushing and milling process such as use wet materials and in the same time reduce friction and heat that produce in reaction and blending (Silva *et al.*, 2003). In thirsty mechanic-chemical, blend powders short of any flush are conservative creased. The reactions dearth extraordinary temperatures in this handling will occur in a ball mill at lesser temperatures deprived of approximately necessary for external warming.

Biomaterial science considers hydroxyapatite one of the most materials used in bone and teeth injured auxiliary shares of a animated union or to determination in close communication with animated materials (Deptula *et al.*, 1992; Hattori *et al.*, 1989). Material is present in

substantial mineralized numbers in the tissue of vertebrates, (Ito *et al.*, 2000). The 65-75% of the mineral portion of the bone humanoid (Miyaji *et al.*, 2005). The mechanical belongings of hydroxyapatite are unspoiled enough to be useful as a implant in cargo behavior sites, comparable false fangs or frames (Miyaji *et al.*, 2005). One track to scatter this worry is to seam it with a firm stage, making a multiple and recover its mechanical limits by consolidation it by fitting substantial (Bigi *et al.*, 1995). In this process a material realized with sensitive things, starved of damage to its biocompatibility (Gomes *et al.*, 2012). Zinc oxide exhibition more than a few supports for bone progress and an no microbial movement for scions, arouses evolution of bone averts bone renewal appearance antimicrobial hostility (Hu *et al.*, 2012). The handiness of zinc arranges the routine and this can be cautious by the crystal structure, crystal extent, form and crystallinity, of zinc (Mondal *et al.*, 2012). Private drills associate zinc, since, a marine in crystal apatite and fortitude quite a few hitches. Zinc expressively drops the inert growth volume over adsorbing to the exterior (Rey *et al.*, 2008). Combination of hydroxyapatite with a mechanic-chemical development was inspected in this examination labor. Primarily, the neutral was to make HA nonstop shorn of the requirement for a dual-step course wherever mechanical instigation meant for 6 h, was tailed via. a hotness handling method at 1100°C (De Aza, 2006). The grinding strictures were thoroughly inspected and these

involved the pounding extent, the sorts of powdering radio and globe in the direction of precipitate percentage. The practice of granulating broad casting, i.e., the droplets and the pots was circumscribed to the changed important as equated to divergent supplies which had been informed in the nonfiction (Park, 2008). The current research focuses on creation bio composite of hydroxyapatite-zinc oxide and displays the bioactive properties of these biomaterials by changed the expanse of zinc oxide.

MATERIALS AND METHODS

Materials used: Marketability hydroxyapatite, zinc oxide of 98 and 96%) (Fluka) purity were used as bio-composite elements.

(ZnO)-(HA) production: The 10, 30 and 50 wt% of (ZnO) mixed with (HA) powders, The mixtures were entirely homogenous drawer four hours complete sphere grinding. Arranged tablets (13*26 mm) ended by cold pressing in a die steel by compression device at force of 100 MPa. The bits were fired at 1100°C for 2 h, through an regular warming degree of 5°C/min.

Characterization

(XRD) diffraction: X-ray rebound pulse (Shimadzu, 6000) at apartment temperature by Cu $k\alpha$ energy ($\lambda = 1.5405 \text{ \AA}$) and 40 kV/30 mA was practiced as the focal critical device.

EDX: Models next 14 days immersing in the (SBF) solution, EDX for external of tasters was completed to identify organic arrangement of crystal-like current segments.

Biological responses: The bioactivity In vitro of combined ZnO-HA estimation was achieved in a Body Fluid (SBF) media of pH 7.4 at 37°C. Dissimilarities in SBF intermediate pH all day planned at dated of time gaps by a pH meter. Energy Dispersive Spectroscopic (EDS) was used to recognize chemical composition of crystalline present phases after immersion in medium of SBF for 14 days. Models weight loss determined by the formulation as given below:

$$\text{Weight loss (\%)} = \frac{W1 - W2}{W1} \times 100$$

Where:

W1 = The sample initial weight then

W2 = The sample final weight after soaking
(Quinn *et al.*, 2009)

RESULTS AND DISCUSSION

Phase analysis: Figure 1 shows XRD outline of models contained 10 and 50 wt.% of the (ZnO) and 100% (HAP) powder. The deflection angle (2θ) from 10-60°, pattern in the peak positions corresponds to the HA compared with (JCPDS) Card No. (09-0432). Phase constancy for the preserved apatites quiet expressions at advanced temperatures sintering short of disintegration and changed quantities of ZnO phase when matching its peaks with (JCPDS) Card No. (13-0311).

Bioactivity evaluation: Figure 2a-c shows the EDX spectra of the (ZnO-HA) samples of (10, 30 and 50%) after soaked in (SBF) solution for 14 days. The EDX reveal such precipitate apatite, the main of Ca, P and O presence. As (ZnO) percent weight was increased the intensity of peaks also increased means that the (ZnO-HA) samples react with the surrounding (SBF) solution to form Ca-rich layer. Figure 3 shows the concentration of Ca, P, O on the (ZnO-HA) samples surface with the (ZnO) percent weight. It is evident that the increasing in he (ZnO) percent weight will cause increment in calcium and phosphate content. It will be interesting to know that the dissolution of hydroxyapatite crystals and high release of Ca^{2+} , PO_4^{3-} . Attentiveness of calcium and phosphorus rise in the nearby fluids and this super saturation induces precipitation of apatite crystals at the exteriors samples (Roy, 2010).

Figure 4 displays the (pH) of the (SBF) solution change in the data were collected for 14 days with periodic interval of 3 days. The initial (pH) value of the solution was 7.4. Up to 3 days the (pH) values for all the samples vary in between 7.8 and 9.2 due to energetic nature of finish course which really effects in increase the exchange of $\text{Ca}^{2+}/\text{H}^+$ ion concentration and responsible for the increase in the (pH). Piercing decline in (pH) value of the solution after 8 days, irrespective of samples is noticed values fluctuate in between 7.5 and 7.2 till 14 days due to precipitation of apatite into the samples surface happens by the Ca^{2+} , P^+ and OH^- ions consuming in the (SBF) solution (Uchino and Toda, 2013).

Figure 5 expressions the relative among the compact models with varied weight percentage of ZnO and weight loss after 14 days soaking in SBF. A trend in the increase in weight loss is observed as the ZnO content in the models increases with respect to pure HA. This shows that the composite samples were more biodegradable thru reverence to HA pure. This variance in bio-degradation and bioactivity due to the differences in the surface area, structure and the current phases in the materials (Kutz, 2002).

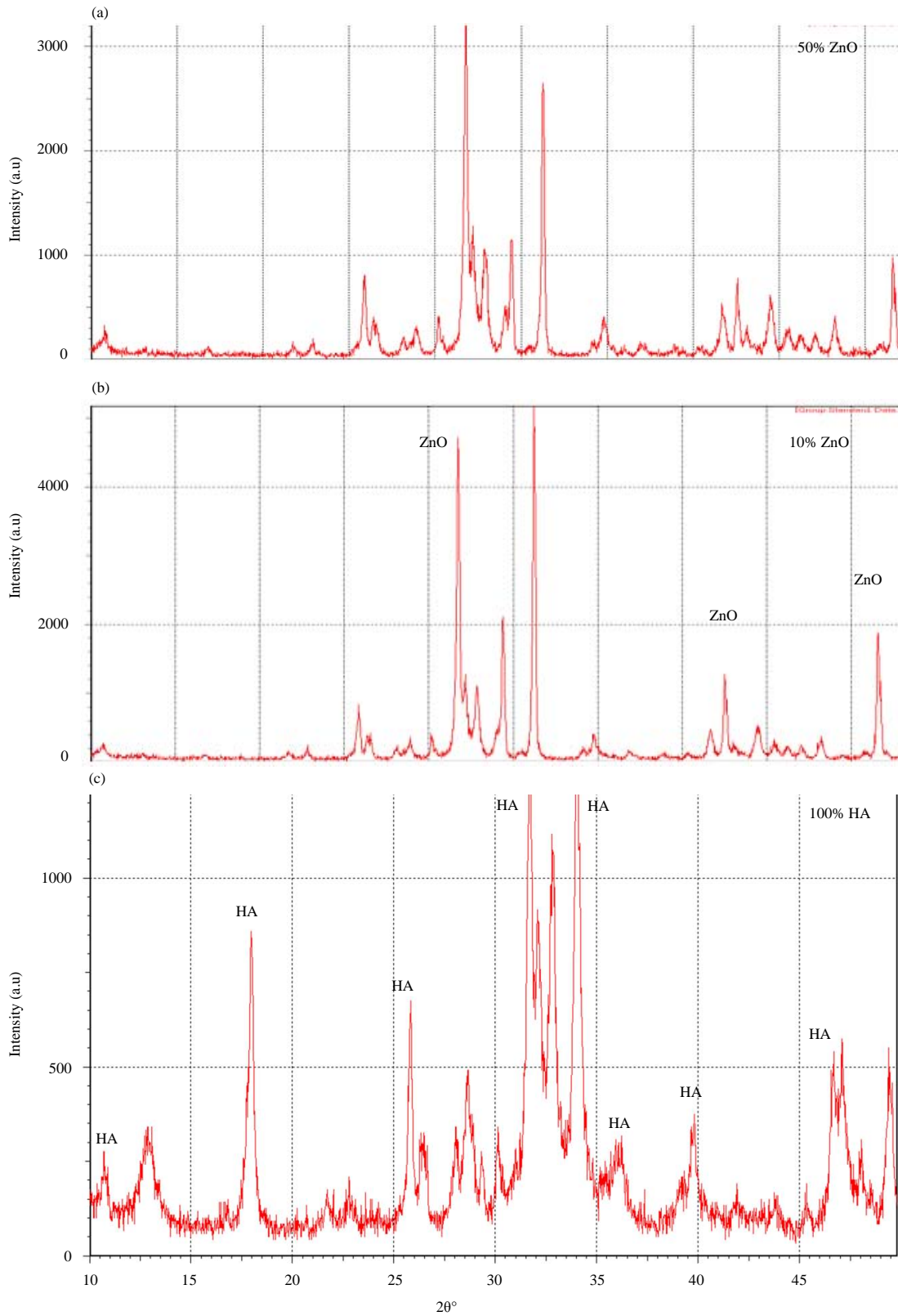


Fig. 1: XRD conformations for (HA/ZnO) models of A (30%), B (50%) of (ZnO), (100% HA) sintered at 1100°C

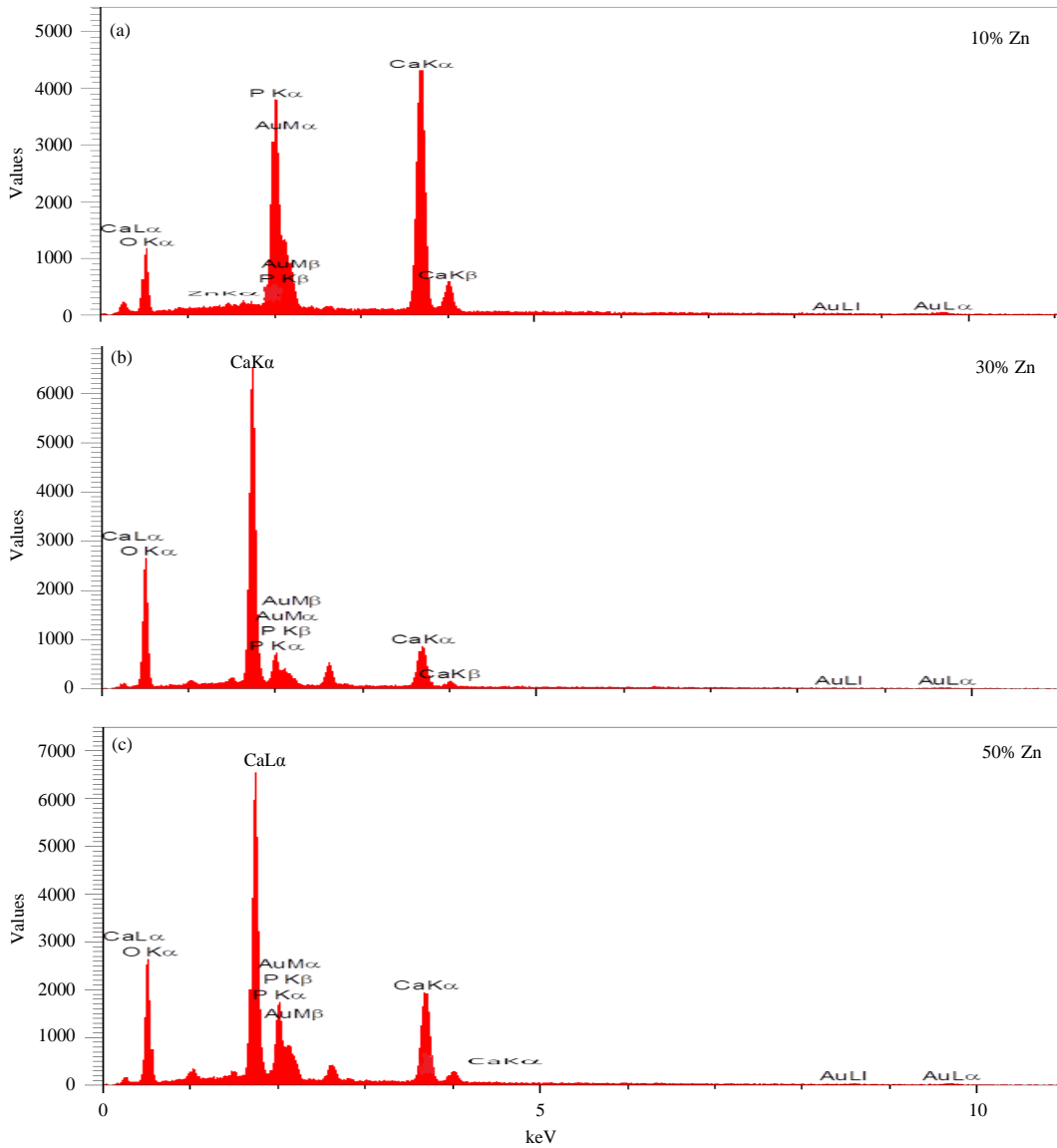


Fig. 2: EDX profile taken from the (ZnO/HA) surfaces: (a) 10% ZnO, (b) 30% ZnO and (c) 50% ZnO after soaking for 14 days

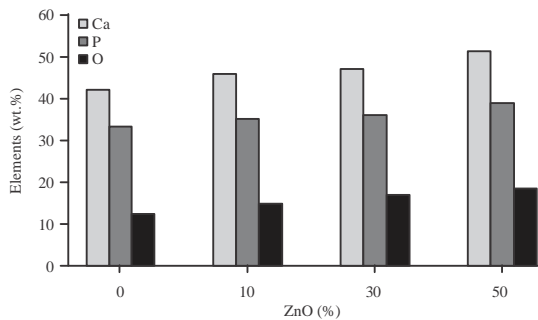


Fig. 3: Variations of elemental concentration (wt.%) on the (ZnO/HA) samples surfaces at various percent weight of ZnO in the (SBF) solution

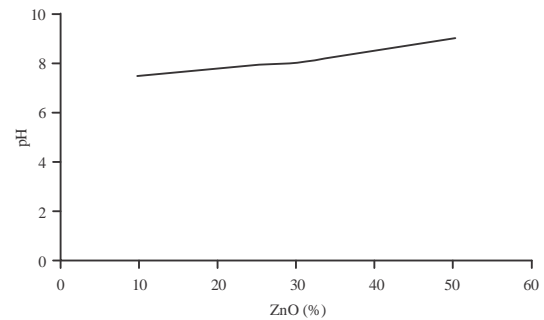


Fig. 4: Variation of (pH) solution values for (ZnO/HA) samples with percent weight of ZnO in the (SBF) solution

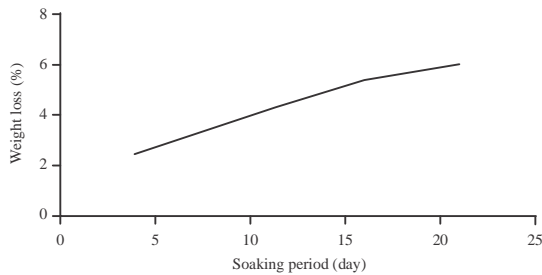


Fig. 5: Variation of weight loss value for (ZnO/HA) samples with percent weight of ZnO in the (SBF) solution

CONCLUSION

Additional proportion of (10, 30, 50 wt.%) Zinc Oxide (ZnO) to Hydroxyapatite (HA) powder to prepare bio-active composite. It was instigate that the (ZnO) growth in the hydroxyapatite matrix prime to growth the biological belongings (bioactivity and degradation) afterward 14 days immersed in (SBF) solution. Which revelations the capacity of hydroxyapatite to technique Ca-P-rich layer on the surface.

Directed that competence of (HA) models later soaking in the (SBF) and foremost existence of P, Ca and O peaks with high intensity of Ca peak. The (pH) of simulated body liquefied clarify the skilled to the alike performance to form Ca-P-rich layer on the surfaces. By maximum weight loss shows degradation level characterized after soaking days in (SBF) solution for the produced hydroxyapatite powder.

REFERENCES

Bigi, A., E. Foresti, M. Gandolfi, M. Gazzano and N. Roveri, 1995. Inhibiting effect of Zinc on hydroxylapatite crystallization. *J. Inorg. Biochem.*, 58: 49-58.

De Aza, P.N., 2006. Progress in Bioceramic Materials. In: *Ceramics and Composite Materials: New Research*, Caruta, B.M. (Ed.). Nova Science Inc, New York, USA., ISBN:9781594543708, pp: 101-132.

Deptula, A., W. Lada, T. Olczak, A. Borello and C. Alvani *et al.*, 1992. Preparation of spherical powders of hydroxyapatite by sol-gel process. *J. Non Cryst. Solids*, 147: 537-541.

Elliot, J.C., 1994. Structure and Chemistry of the Apatites and other Calcium Orthophosphates. Elsevier, Amsterdam, Netherlands, ISBN:9781493302468, Pages: 389.

Gomes, S., J.M. Nedelec and G. Renaudin, 2012. On the effect of temperature on the insertion of Zinc into hydroxyapatite. *Acta Biomater.*, 8: 1180-1189.

Hattori, T., Y. Iwadate and T. Kato, 1989. Hydrothermal synthesis of hydroxyapatite from calcium pyrophosphate. *J. Mater. Sci. Lett.*, 8: 305-306.

Hu, W., J. Ma, J. Wang and S. Zhang, 2012. Fine structure study on low concentration Zinc substituted hydroxyapatite nanoparticles. *Mater. Sci. Eng. C.*, 32: 2404-2410.

Ito, A., K. Ojima, H. Naito, N. Ichinose and T. Tateishi, 2000. Preparation, solubility and cytocompatibility of Zinc-releasing calcium phosphate ceramics. *J. Biomed. Mater. Res.*, 50: 178-183.

Jin, H.B., F.N. Oktar, S. Dorozhkin and S. Agathopoulos, 2011. Sintering behavior and properties of reinforced hydroxyapatite/TCP biphasic bioceramics with ZnO-whiskers. *J. Compos. Mater.*, 45: 1435-1445.

Kutz, M., 2002. Handbook of Materials Selection. John Wiley & Sons, Hoboken, New Jersey, USA., ISBN:9780471359241, Pages: 1520.

Miyaji, F., Y. Kono and Y. Suyama, 2005. Formation and structure of Zinc-substituted calcium hydroxyapatite. *Mater. Res. Bull.*, 40: 209-213.

Mondal, S., B. Mondal, A. Dey and S.S. Mukhopadhyay, 2012. Studies on processing and characterization of Hydroxyapatite biomaterials from different bio wastes. *J. Miner. Mater. Charact. Eng.*, 11: 55-67.

Park, J., 2008. Bioceramics: Properties, Characterizations and Applications. Springer, New York, USA., ISBN:9780387095448, Pages: 363.

Quinn, G.D., B.T. Sparenberg, P. Koshy, L.K. Ives and S. Jahanmir *et al.*, 2009. Flexural strength of ceramic and glass rods. *J. Test. Eval.*, 37: 222-244.

Rey, C., C. Combes and C. Drouet, 2008. Tricalcium Phosphate-Based Ceramics. In: *Bioceramics and their Clinical Applications*, Kokubo, T. (Ed.). Woodhead Publishing, Cambridge, England, ISBN: 9781845692049, pp: 326-366.

Roy, M., 2010. Vickers hardness measurements: Practices and Calibration check. Master Thesis, Department of Materials Engineering, The University of British Columbia, Vancouver, Canada.

Silva, C.C., A.G. Pinheiro, M.A.R. Miranda, J.C. Goes and A.S.B. Sombra, 2003. Structural properties of hydroxyapatite obtained by mechanosynthesis. *Solid State Sci.*, 5: 553-558.

Siriphannon, P., Y. Kameshima, A. Yasumori, K. Okada and S. Hayashi, 2002. Formation of hydroxyapatite on CaSiO₃ powders in simulated body fluid. *J. Eur. Ceram. Soc.*, 22: 511-520.

Uchino, T. and K. Toda, 2013. Synthesis of Zinc-containing amorphous calcium phosphate. *Key Eng. Mater.*, 529: 119-122.