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Preparation and Physico-Chemical Characterization Studies of Fluoride Doped Bioceramics (P_2O_5-CaO-CaF_2-Na_2O) for Bone Regeneration

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Abstract

Fluoride doped calcium-phosphate bioceramics (P_2O_5-CaO-CaF_2-Na_2O) containing different proportions of calcium fluoride (X= 0.00, 1.25, 2.50, 3.75 and 5.00 mol %) were prepared by melt quenching technique. These bioceramics were characterized for their structural, physico-chemical, and morphological properties as per the standards set for orthopedic implants. The FTIR of these materials were recorded to assess the presence of key functionalities. The density, ultrasonic velocity, elastic constants and Poisson's ratio measurements were made to understand the effect of concentration of fluoride doping in the bioceramics. The morphological aspects of the chosen bioceramics were studied using SEM and EDS. The crystallization, melting and thermal stability of fluoride doped bioceramics were evaluated using DSC and TG studies. From these characterizations, concentration of fluoride in the mole percentage of 5 is more ideal for further clinical application studies in bone tissue engineering.

Keywords: Bone tissue engineering • Melt quenching •Fluoride doped calcium-phosphate bioceramics

Introduction

Biomaterials promote healing and enhance bone regeneration by creating natural living tissue in internal milleu. The crucial property of the biomaterials is to create chemical interaction with the host tissue without affecting the structural integrity of the host site. Biomaterials are classified as metals, ceramics, natural or synthetic polymers and composites. Among these, ceramics have more versatile properties such as biocompatibility, high density and low fracture strength [1-3]. In 1969,Hench and Clark made an attempt to prepare bioceramics using inorganic minerals with the composition such as 45.0% SiO_2, 24.5% Na_2O, 24.4% CaO and6.0 % $P_2O_5$and later it is simply denoted as 45S5 bioglass. The 45S5 bioglass has an excellent biocompatibility and bone bonding property which plays a vital role in small bone replacement of middle ear or ossicle replacement and dental application till now. Though 45S5 bio glass gets compatible with the natural bone, it has failed due to its slow dissolution rate, brittleness and so inability to be used in load bearing application of bone [4]. To overcome these pitfalls, researchers focused on borate based bioceramics and metal oxide doped bioceramics [5].

The focus of the present study is to prepare fluoride doped calcium phosphate bioceramics with the low elastic moduli, high mechanical strength, high bioconversion and controlled rate of dissolution similar to the rate of formation of the natural bone inorganic minerals such as hydroxyapatite (HA), fluorapatite and other calcium phosphates. Fluoride ions promote bone formation, is a factor known to clinical experts.Mostly, the fluoride is used for root canal filling for tooth remineralization and avoid demineralization which prevents enamel erosion and bacterial infection [6]. In the present study fluoride is incorporated in the form of calcium fluoride and the concentration range is 0.0-5.0 mol %. These fluoride doped bioceramics were characterized for their structural, physico-chemical, mechanical and morphological properties and the results are presented and discussed.

Review of Literature

Ultrasonic measurement

The measurement system consists of an ultrasonic process control system (Model FUII050; Fallon Ultrasonics Inc. Ltd., ON, Canada), a 100-MHz digital storage oscilloscope (Model 54600B; Hewlett Packard, Palo Alto, CA) and a computer. The measurements were carried out by generating longitudinal and shear waves using X- and Y- cut transducers operated at a fundamental frequency of 5 MHz. Ultrasonic velocities (UL, longitudinal and US, shear) and attenuations (UL, longitudinal and US, shear) measurements were done using pulse echo method and cross- correlation technique.

X-ray photoelectron spectroscopy

An elemental composition of the prepared bioceramics was analyzed by X-ray photo electron spectroscope (Model AXIS Ultra DLD, Kratos, Kyoto, Japan) with Al Kα assource at an operating condition of 210 W.A survey spectrum was recorded in the range of 0–1200 eV and highresolution spectra for C1s and N1s bands were obtained. In XPS measurements, X-ray was used as an excitation radiation.Thespectra were observed in a fixed retarding ratio mode with bandpass energy of about 10 eV.

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Scanning electron microscopy

The morphological characteristics of the prepared bioceramics were examined using sputtering technique. Scanning Electron Microscope (Model Ultra 55; Zeiss, Oberkochen, Germany) was used for the present study.

Energy dispersive X-ray spectroscopy

Energy dispersive X-ray spectrograph (EDS) was taken for obtaining semi quantitative elemental information of the surface of samples using EDS (Model: Oxford Xmax50 EDS; Oxford instrument England).

Discussion

The density of the bioceramics is affected by the chemical composition of the material.The variation in bioceramics density with respect to calcium fluoride (network modifier) content has been presented. Initially, the addition of CaF_2 decreases the density from 2707 to 2630 kgm-3 of the material formed and further increase in the addition of CaF_2 increases the density slightly. The decrease in the density with increasing CaF_2 content probably leads to the breakage of the P-O-P bonds. While increasing the mol percentage of CaF_2, ionic cross-linking was formed between the non-bridging oxygen of two different phosphate chains. These results showed that calcium fluoride act as network modifier. The increase in the density at higher quantities of incorporation of CaF_2 is due to change in the crystallinity, structural reorganization of the atom and ionic cross-linking between non-bridging oxygen of the phosphate groups of the sample.

The present investigation discloses the bioceramics preparation (P_2O_5-Ca_2O-Ca_2F-Na_2O) by melt quenching method. The FTIR studies showed absorption bands corresponding to metaphosphate (1107cm^{-1}) and phosphorofluoridate (750 cm^{-1}) moieties. The increasing mol percentage of the calcium fluoride from 0.0 to 5.0 affects the density of the bioceramic formed. Usually the measured longitudinal, bulk, Young's and shear moduli for the ceramics follow the same trend of variation as that of density. The obtained density, longitudinal, bulk, Young's and shears moduli results for the material FP3 are 2628 kgm^{-3}, 68.63 GPa, 44.42 GPa,
47.94 and 18.16GPa respectively. The correlation of the elastic properties with density explains the structural integrity of the bioceramic. The X-ray photoelectron spectroscopic study validates the existence of sodium metaphosphates (binding energy 1071KeV) and calcium fluorophosphates (binding energy 347KeV) which are considered as key components for bio network and hydroxyapatite formation. By clinical experts, the calcium and phosphate with fluoride is identified to enhance the osteo-induction progression. The surface morphology and the energy dispersive spectra exhibit the appearance and the composition of inorganic minerals for bone formation. The crystallization exotherm and the melting endotherm of the bioceramics are noted at 504 and 734°C respectively. The thermal studies for the material (FP3) show the purity and the thermal stability of the bioceramics. The doping of 5 mol % of fluoride molecule in P_2O_5-CaO-Na_2O-CaF_2 system is expected to meet the requirements for the orthopedic applications in terms of physical, elastic and morphological properties.

In summary, we are approaching seemingly to a logical direction to demonstrate any influence of melanocytes might have on neural cells for its differentiation, dopamine production and releasing abilities of neural factors, like BDNF and GDNF. Production of BDNF /GDNF are vital for neural cells survival. In brief, our hypothesis, therefore, may open up a modification tool for upgrading neural cells before AD cell-replacement therapy.

Conflict of Interest

Nil

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