



Simultaneous structural and surface modifications of nanophase hydroxyapatite for improving its dissolution and bioactivity

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Abstract

The aim of this study was to improve the dissolution rate and bioactivity of nanophase hydroxyapatite (HA). For this purpose, simultaneous structural and surface modifications of nanophase HA was made by incorporating SiO_4^{4-} into the HA structure and creating a silica layer on its surface via the sol–gel method. Characterization of the prepared nanopowders was performed by transmission electron microscopy, X-ray diffractometer, Fourier transformed infrared spectroscopy, and acid dissolution testing. An atomic absorption spectrometer was used to determine calcium ions concentration in physiological saline solution. Results revealed that the simultaneous structural and surface modifications of nanophase HA had a considerable increasing effect on calcium ion release from nanophase HA, and on surface reprecipitation rate of calcium ions, respectively. The obtained results suggest that this modified nanophase HA could be used as a new bioceramic for bone repair.

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1. Introduction

Hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$; HA) has been demonstrated to be suitable for biomedical applications, such as bone repair [1]. However, slow dissolution rate of nanophase HA [2], and its limited bioactivity [1], are weaknesses for its clinical applications which must be improved.

According to literature, the osteoconductive properties of HA are related to its dissolution behavior in the human body environment [3]. Suitable structural change is one way for increasing the dissolution rate of HA [4]. Moreover, it has been shown that negatively charged surfaces on

bioceramics have a vital role in their bioactivity [5]. For example, surface silanol groups (Si–OH) could have an accelerating effect on the nucleation and growth of apatite (bioactivity) [6].

The aim of the present work was to improve the dissolution rate and bioactivity of nanophase HA by means of its simultaneous structural and surface modifications.

2. Materials and methods

2.1. Synthesis of nanopowders

Calcium nitrate tetrahydrate (1.67 mol L^{-1}) and phosphoric pentoxide (0.5 mol L^{-1}) as HA precursors were dissolved in separate beakers containing absolute ethanol. Both of the prepared solutions were mixed at a Ca/P molar ratio of 5:3. The resultant solution was then stirred for $\sim 3 \text{ h}$ at $30 \text{ }^\circ\text{C}$, and

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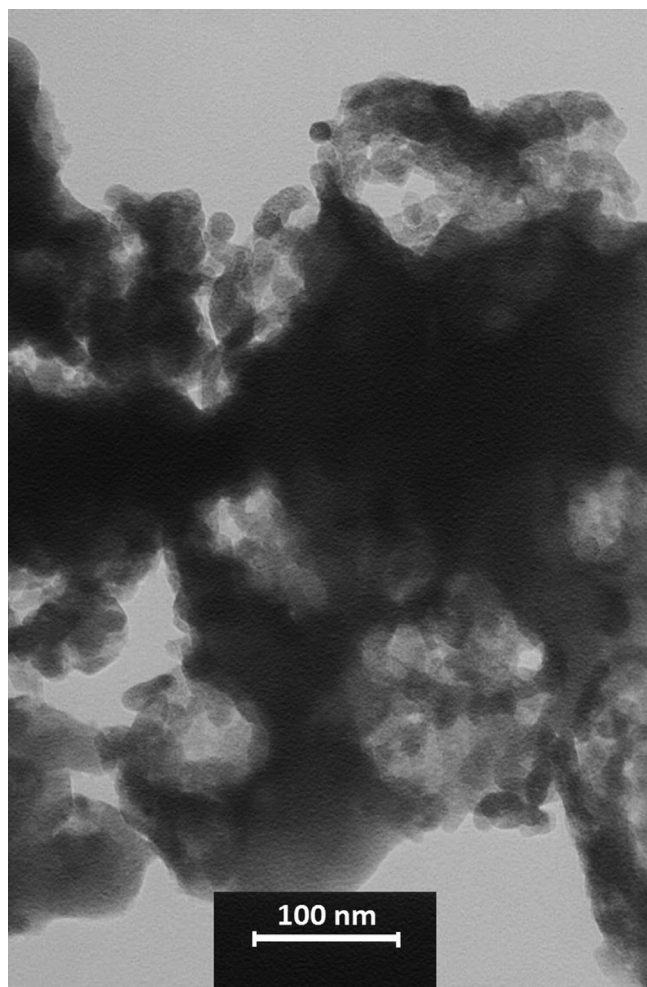


Fig. 1. TEM micrograph of M-HA nanopowder.

was then aged for ~ 6 h at 30°C under static conditions. Following this process, tetraethylorthosilicate (2.74 ml) was added to the aged mixture under continuous stirring to form a final product. After ~ 1 h of stirring, 0.1 N HNO_3 and acetic acid were added as the catalyst [7]. The molar ratio of deionized $\text{H}_2\text{O}/\text{TEOS}$ and deionized $\text{H}_2\text{O}/\text{acetic acid}$ was 2:1 and 7:1, respectively. The final mixture was constantly stirred for ~ 3 h at 30°C and was then aged for 24 h at room temperature under static conditions. The resulting gel was dried at 80°C for 24 h. Finally, the calcination of the crushed dry powder was carried out at 600°C for 30 min, after which, the prepared powder was kept at room temperature to be cooled. The sol-gel derived powder is referred as M-HA nanopowder. For comparison purposes, HA nanopowder was also prepared. All of the raw materials were obtained from Merck (Merck, Darmstadt, Germany).

2.2. Characterization of nanopowders

The morphology of M-HA nanopowder was observed by transmission electron microscopy (TEM, Philips CM120). The crystalline phases of the prepared nanopowders were determined by an X-ray diffractometer (XRD, Philips X'Pert-MPD)

using a $\text{Cu K}\alpha$ radiation ($\lambda=0.15418$ nm, 40 kV and 30 mA) over the range of $10\text{--}70^\circ$ with a step size of 0.05° s^{-1} . Also, the lattice parameters (a and c) and unit cell volume (V) of the prepared nanopowders were determined using the cell parameters refinement software (Celref V.3).

Fourier transformed infrared (FTIR, Bomem MB 100) spectroscopy analysis was performed to identify functional groups of the prepared nanopowders. The spectra were recorded in the $4000\text{--}400$ cm^{-1} region with 4 cm^{-1} resolution.

In order to confirm surface modification of nanophase HA by silica, an acid dissolution test [7–9] was performed by dispersing the powder samples with a ratio of 75 mg/ml in 2 M HCl and subjecting them to sonication for 30 min in an ultrasonic bath.

In order to evaluate dissolution behavior of the prepared nanopowders, powder samples were compressed using a hydraulic press at room temperature. The prepared disks were immersed in physiological saline solution (0.9 wt% NaCl) for up to 4 days at 37°C in a water bath. The surface area (mm^2) of the disk sample to physiological saline solution (ml) ratio was fixed at 10 to 1. At pre-determined time periods, the disk samples were removed and the concentration of dissolved calcium ions in the filtered solutions was measured using an atomic absorption spectrometer (AAS, Perkin-Elmer 2380).

3. Results and discussion

3.1. TEM analysis

Fig. 1 shows the TEM micrograph of M-HA nanopowder. As can be seen, components of M-HA nanopowder have an elliptical shape with an average size of less than 30 nm.

3.2. XRD analysis

Fig. 2 shows the XRD patterns of HA and M-HA nanopowders. As can be seen in Fig. 2B, no peak related to silica (amorphous phase) is recorded in the XRD pattern of M-HA nanopowder. However, the presence of silica reduced the intensity of the HA peaks in the XRD pattern (Fig. 2B). The lattice parameters and the unit cell volume for HA nanopowder and HA in M-HA nanopowder were $a=9.4093$ Å, $c=6.8862$ Å, $V=527.98$ Å³ and $a=9.4345$ Å, $c=6.8905$ Å, $V=531.16$ Å³, respectively. In agreement with other researchers, the higher lattice parameters and unit cell volume of HA in M-HA nanopowder rather than HA nanopowder can be attributed to partial substitution of silicate (SiO_4^{4-}) groups for phosphate (PO_4^{3-}) groups in the HA lattice [1,10]. In fact, the higher lattice parameters of HA in M-HA nanopowder rather than HA nanopowder is due to the larger Si^{+4} ionic radius than P^{+5} ionic radius and also the higher average Si–O bond length than the P–O one [11].

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