REFORMULATION OF HAP WITH ZnO

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Abstract

The effect of zinc on the stimulation of osteogenesis has become a major focus in research work on bone formation. Based on this fundamental work, a base HAP body (HA1) was reformulated by adding 10 wt% ZnO. The physical properties (i.e. bulk density and porosity), phase analysis (XRD) and surface fracture analysis (SEM) were determined after sintering at five different temperatures (i.e. 1150, 1200, 1250, 1300 and 1350°C). Addition of ZnO shows that the bulk density is decreased (2.30 – 2.70 g cm⁻³) and porosity is increased (5.0 -27.0%) compared to a pure HAP (HA1) at these five different temperatures. The XRD pattern of the HAP + ZnO (HA1Z) indicated that the reaction of HAP and ZnO to form CaZn₅(PO₄)₂ occurred at 1250°C.

Introduction

Surgical implant materials officially approved for hard-tissue replacement including titanium, alumina, hydroxyapatite (HAP), tricalcium phosphate (β-TCP) and glass ceramics are all biocompatible, showing bone-bonding ability to the materials directly or with thin interfacial fibrous connective tissues (1). The slow release of zinc incorporated into an implant material could promote bone formation around the implant and accelerate recovery of a patient. Zinc is an essential trace element having stimulatory effect on bone formation in vitro and in vivo (2). Based on this factor, zinc oxide was added to HAP. Narasaraju, (1996) reported that zinc would react and replace calcium in this composite.

Experimental Procedure

The fabrication of HAP + ZnO was effected by a wet mixing process of 90 wt% + 10 wt % ZnO for 16 hours. Two polymeric binders (2% PVA + 2% PEG) were then added whilst mixing for 2 hours. After mixing, the aqueous slurry was dried in an oven at 56°C for another 24 hours. The dried cake was crushed into powder form, which was passed through a 100 μm-mesh sieve. The powder was granulated with 8 weight % water being before being sieved through a 250 μm mesh. The granules were then pressed uniaxially at room temperature into bar shaped specimens (15 x 50 x 5 mm). The pressure was maintained for 1 minute at 51 MPa before the specimens were ejected. The compacted specimens were then sintered at a heating rate of 2°C/min and soaked for 2 hours at 5 different temperatures (1150, 1200, 1250, 1300 and 1350° C). After sintering, the physical properties (bulk density and porosity) of specimens were measured, and XRD and SEM analyses were carried out.
Results and Discussions

Physical Properties

Figure 1: The bulk density of HA1 and HA1Z plotted against sintering temperature.

Figure 2: The porosity of HA1 and HA1Z plotted against sintering temperature.

A significant difference can be observed in the bulk density values of sintered specimens of HA1 and HA1Z (Figure 1). HA1 shows bulk density values that are more than 2.85 g/cm³ and HA1Z less than 2.70 g/cm³. Thus means that ZnO has changed the bulk density value and the microstructure of HA1 (Figure 3). Similarly, it can be seen from Figure 2 that ZnO has changed the porosity value of specimen HA1. The porosity value of HA1 is below than 2.5% whereas HA1Z more than 8.5%. It is believed that ZnO particle were distributed between the HA1 particles and created a distance between the HA1 particles. ZnO can also act as filler in the HA1 specimen.

SEM Analysis

Figure 3 shows the micrograph of the fracture surface of specimen HA1 and HA1Z at five different temperatures. It can be seen that the effect of ZnO on the HA1. At lower temperature, ZnO contributes to a more porous structure and it decreases with increasing sintered temperature.

XRD Analysis

Figure 4 and Figure 5 show the XRD pattern of HA1 and HA1Z that have been sintered at 1150°C−1350°C. Figure 4 indicates the formation of β-TCP at a lower sintering temperature (1150°C) until the highest temperature (1350°C). Specimen HA1Z (Figure 5) shows the formation of CaZn₅(PO₄)₂ phase starts at 1250°C.
Figure 3: SEM micrograph of the fracture surface of HA1 and HA1Z after sintering at five different temperatures
Figure 4: X-ray diffraction patterns of sintered HA1 at 1150-1350°C (β-TCP, ICDD File card No. 9-169).

Figure 5: X-ray diffraction patterns of sintered HA1Z at 1150-1350°C (β-TCP, ICDD File card No. 9-169; zincite, ICDD File No. 36-1451; CaZn$_2$(PO$_4$)$_2$, ICDD File No. 20-250).

Conclusions

The ZnO that was added to the HA1 body appears to decrease the bulk density and increase the porosity. SEM analyses show the effect of ZnO on the porosity of HA1 specimen. The phase in HA1 changes to β-TCP at a lower temperature (1150°C) and this phase will react with ZnO to form CaZn$_2$(PO$_4$)$_2$.

References

