

Synthesis of bovine hydroxyapatite-iron composite via dry mechanochemical process for biodegradable bone scaffolds

JA Nordin¹, NM Daud¹, DH Prajitno², H Nur³, H Hermawan¹

¹ [Faculty of Biosciences and Medical Engineering, University Teknologi Malaysia \(UTM\), Malaysia.](#) ² [Indonesian Nuclear Research Agency \(PTNBR-BATAN\), Indonesia.](#) ³ [Ibnu Sina Institute for Fundamental Studies, UTM, Malaysia.](#)

INTRODUCTION: Hydroxyapatite (HAp) is widely used in orthopedic and dental surgeries [1]. This study aims to synthesize a Fe-reinforced natural HAp composite via dry mechanochemical process without chemical addition. An improvement to the mechanical strength and cytocompatibility is expected with the presence of Fe ions in the HAp structure.

METHODS: Samples of a bovine cortical bone were cut, washed and boiled for 24 h to remove impurities. They were deproteinized at 160°C for 48 h and calcinated at 750°C for 6 h to obtain bovine HAp powders (HAp(B)). A composite of HAp-30wt% Fe (HAp(B)+Fe) was prepared by mixing HAp(B) and pure Fe powders (Goodfellow, UK) using high energy milling for 3 h in a Teflon vial with Zirconia balls. The charge to ball ratio and rotational speed were 1:10 and 1200 rpm, respectively. Pellets ($\varnothing = 8$ mm, thickness = 5 mm) were compacted at 129 kg.cm⁻² and sintered at 900°C for 2 h. Characterization was done using XRD, FTIR, and SEM, while a hardness Vickers test was carried out using a 10 kgf-load. The cytotoxicity test was carried out using human mesenchymal stem cell (HMSC) following the ISO 10993-12. The MTT assay was used to measure the cell viability after 24 h.

RESULTS: Figure 1 shows the FTIR spectra identifying the substitution of Fe in the HAp(B).

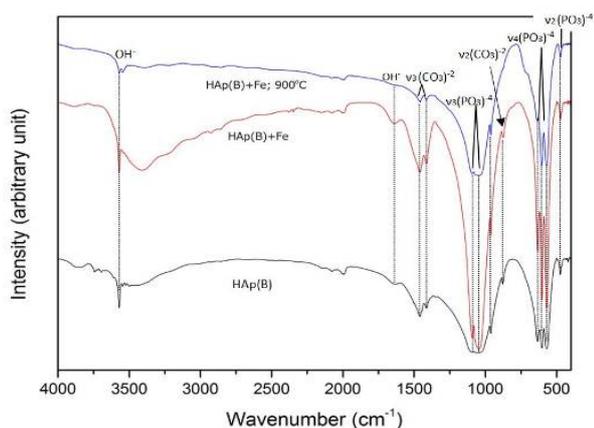


Fig. 1: FTIR spectra of the HAp(B) and the HAp(B)+Fe composite.

The hardness of the HAp(B) pellets was 54.15 Hv, while the HAp(B)+Fe was 108 Hv. The XRD pattern identified the presence of HAp (JCPDS 9-0432), crystalline Fe (JCPDS 6-0696) and Fe₂O₃ (JCPDS 033-0664) on the HAp(B)+Fe but no change on that of the HAp(B). The MTT result showed the percentages of cell viability of 88% and 87% for HAp(B) and HAp(B)+Fe, respectively. The viability of HAp(B)+Fe was 80% higher than the control.

DISCUSSION & CONCLUSIONS: The FTIR peaks at 1415 and 1459 cm⁻¹ correspond to ν_3 , and a band at 871 cm⁻¹ corresponds to ν_2 , indicating the stretching vibration of carbonate groups. A sharp peak was defined after the HAp(B) was milled with the Fe addition. The sintering at 900°C caused a disappearance of hydroxyl peak at 1638 cm⁻¹. Additional 3 peaks observed at 633, 602 and 568 cm⁻¹ corresponds to ν_4 of phosphate [2]. The addition of Fe increased the hardness of HAp(B) by two fold due to the presence of Fe³⁺ in the HAp lattice structure that increases its crystallinity [3]. The MTT assay demonstrated that the presence of Fe in the composite was not toxic to HSMC and the HAp(B) gave higher percentage of the viability compared to the synthetic HAp. Other work found that bovine HAp increased osseointegration at 12 weeks compared to the synthetic one [4]. It was also observed that the released Fe ions into the medium accelerated the precipitation of Ca²⁺ and PO₄³⁻ on the surface of the composite. The addition of 30wt% Fe into bovine HAp has improved its hardness and cytocompatibility.

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