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Bioactivity Evaluation of Ti-Nb-$CaSiO_3$ in Hank's Balanced Salt Solution

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Abstract

Titanium (Ti) is the most often used metallic biomaterial in orthopedic implants due to its remarkable properties such as high strength and good corrosion resistance. Nonetheless, the analysis must be carried out for Ti-based material in order to ensure their quality for use as biomaterial. The initial test that needs to be performed include bioactivity in Hank's Balanced Salt Solution (HBSS) to determine whether the material can form an apatite layer. The present work investigates the bioactivity evaluation of Ti-Nb-$CaSiO_3$ composite in HBSS. Ti, Nb and $CaSiO_3$ powders were mixed in planetary mill at 200 rpm for 2 h, followed by compaction under 500 MPa and sintering at 800, 1000 and 1200 °C. Upon immersion test in HBSS for 30 days, apatite deposition corresponding to the Ca and P layer was formed on the surface of all composites. At a sintering temperature of 1200 °C, the composite displayed greater apatite coverage, exhibiting an amorphous structure, which reveals its potential as a bioactive material.

Keywords: bone implant, apatite, bioactivity, sintering, HBSS.

1. Introduction

Biomaterials are becoming more important in developing medical and dentistry applications, especially in recent years. Titanium-based alloys, in particular, have received substantial interest as they have great mechanical properties, good biocompatibility, and excellent corrosion resistance [1]. It must be noted that titanium (Ti) has poor osseointegration, being unable to properly integrate itself into surrounding tissues and bones [2]. Therefore, in order to improve the bioactivity of Ti, wollastonite ($CaSiO_3$) is added due to its similar composition with bone [3], [4].

Bioactivity refers to the capacity of a material to interact with the surrounding biological environment, facilitating desired cellular responses such as cell adhesion, proliferation, and development of new tissue. Bioactivity evaluation is essential to determine the viability of the biomaterial in clinical applications. In this study, Hank's Balanced Salt Solution (HBSS) is used as the medium to simulate the ionic content and pH of human bodily fluids for *in-vitro* evaluation of Ti-Nb-$CaSiO_3$.

2. Materials and Method

2.1 Materials and Preparation of $CaSiO_3$

Commercially pure Ti powder (cp-Ti), niobium (Nb) and wollastonite ($CaSiO_3$) were used as shown in Table 1. $CaSiO_3$ was synthesized through the wet milling method by mixing calcium oxide (CaO) and silicon dioxide (SiO_2) in a ratio of 55:45, followed by sintering at 950 °C for 3 h. A composite comprising Ti, Nb and $CaSiO_3$ was fabricated by mechanical alloying (MA). The planetary mill machine of the type Fritsch Pulverisette, P-5 was used to perform the MA of the starting powders. A grinding jar with a capacity of 250 ml and stainless-steel balls with a diameter of 10 mm were used. The mixed elemental powders underwent MA for 2 h with a speed of 200 rpm and BPR (ball-to-powder ratio) of 10:1. The MA was accomplished in n-heptane to avoid cold welding and oxidization of the mechanically alloyed (MAed) powders. The composite powders were compacted under 500

MPa, and pellets with a diameter of 10 mm were obtained. The compacted pellets were sintered in a Lenton tube furnace at different temperatures (800, 1000, and 1200 °C) under flowing argon.

Table 1. Composition of the produced Ti-Nb-$CaSiO_3$ composite

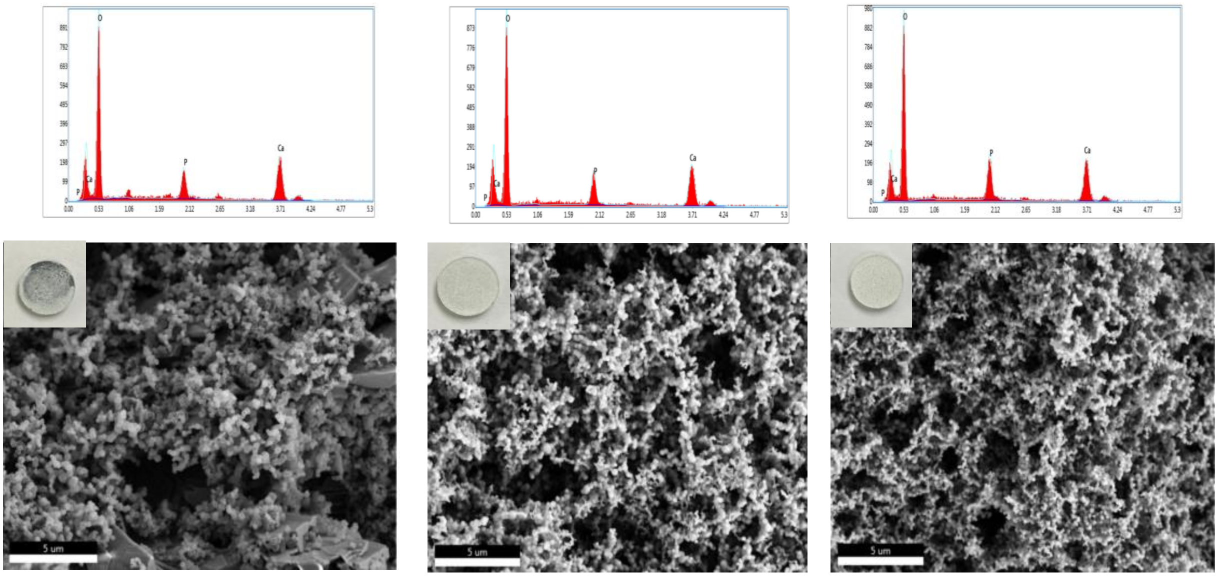
Materials	Composition (wt.%)	Brand	Purity (%)
Titanium (Ti)	50	Strem	99.70
Niobium (Nb)	40	Strem	99.80
Wollastonite ($CaSiO_3$ which consist of CaO: SiO_2)	10	CaO purchased from R&M Chemicals	96.00
		SiO_2 purchased from R&M Chemicals	96.00

2.2 *In-vitro* bioactivity evaluation of Ti-Nb-$CaSiO_3$

The *in-vitro* bioactivity of Ti-Nb-$CaSiO_3$ composite was evaluated in HBSS (Gibco, Life Technologies) (14175-095). As-sintered Ti-Nb-$CaSiO_3$ composite was first weighted using an electronic balance (Sartorious), and the initial weight was recorded. The HBSS solution was filled into Falcon tubes, and its temperature was raised to and maintained at 37 °C using a water bath (TWB-30D). Once the set temperature was achieved, the composites were placed into the Falcon tubes, and the tubes were capped to prevent evaporation of the HBSS solution. The composites were left immersed in the HBSS for 30 days. After that, the composites were removed from the HBSS, gently rinsed with distilled water, and dried in a desiccator without heating for 72 h. In order to assess the bioactivity of the composites, field emission scanning electron microscopy (FESEM, Zeiss Supra 35VP) was utilized to examine the formation of the apatite layer. Additionally, an energy dispersive X-ray (EDX) was conducted, which was attached to FESEM, to detect the presence of calcium (Ca) and phosphorus (P), which are essential components of apatite.

3. Result and Discussion

The FESEM micrographs of Ti-Nb-$CaSiO_3$ composites after immersion in HBSS for 30 days are presented in Fig. 1. A whitish deposit, thought to be the apatite, was observed on the surface of all composites as shown in the FESEM image insets. At 800 °C, a randomly distributed rhombohedral structure co-existed with spherical globules. As the temperature increased to 1000 °C and 1200 °C, the surface morphology exhibited a homogenous distribution of spherical globules throughout the composite. These spherical globular structures, composed of aggregated smaller entities known as apatite layer, were confirmed to be calcium phosphate (CaP) through EDX analysis. The presence of Ca and P provided evidence for the formation of apatite due to the physiological reaction during HBSS immersion. Similar structures were previously observed by A. Adawy and W.I. Abdel-Fattah [5] when the biomaterials were immersed in a physiological solution, closely resembling the structure of biological apatite on the material surface. The ability of Ti-Nb-$CaSiO_3$ composite to form apatite due to the presence of CaP on its surface signifies its potential to form apatite in human bone. Composites exhibiting greater bioactivity demonstrate higher apatite coverage on their surface. When comparing the micrographs shown in Fig. 1, Ti-Nb-$CaSiO_3$ composite sintered at 1200 °C showed higher apatite coverage, leading to a more bioactive surface that facilitates direct bonding with the surrounding bone tissues. Consequently, this can promote better osseointegration and contribute to the improved long-term stability of the implant.



(a) (b) (c)

Fig. 1. Representative EDX spectra showing peaks for Ca and P and FESEM micrographs on the surface of Ti-Nb-$CaSiO_3$ composite at different sintering temperatures : (a) 800 °C, (b) 1000 °C, and (c) 1200 °C, after immersion in HBSS solution for 30 days (5000x magnification)

4. Conclusion

The EDX analysis revealed that the precipitate layer contains Ca and P, confirming the apatite formation on the surfaces of the Ti-Nb-$CaSiO_3$ composite sintered at 800, 1000, and 1200 °C. This study gives important insight into the potential of Ti-Nb-$CaSiO_3$ composite in orthopedic applications.

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