

Short communication

# Bio-degradation of bioactive glass ceramics containing natural calcium phosphates

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## Abstract

Body fluids (similar to the blood plasma) enhance apatite formation on the surface of calcium phosphate based bioceramic implants, which in turn improve the bone-binding strength of such devices. A systematic investigation of hydroxyapatite formation on the surface of bioactive glass ceramics immersed into simulated body fluids (SBF) was therefore conducted on glass ceramic samples produced from pre-treated animal bones. Significant changes of phase composition, microstructure as well as microhardness of surface prior to and after SBF-treatment were observed. Open porosity correlates with other results. Dissolved constituents (Ca and P) of bioactive glass ceramics were quantified by X-ray fluorescence analysis of SBF. Optimum conditions were determined by using microhardness, porosity, and dissolved Ca as independent variables.

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## 1. Introduction

Hydroxyapatite, (HA),  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$  is the most widely used synthetic calcium phosphate for the implant fabrication because is the most similar material from the structural and chemical point of view, to the mineral component of bones [1]. It amounts to 65% of the total bone mass, with the remaining mass formed by organic matter and water [2]. Manufacture of hydroxyapatite from animal bones is described elsewhere [3].

Hench et al. [4] first showed that some glasses in the  $\text{Na}_2\text{O}$ – $\text{CaO}$ – $\text{SiO}_2$ – $\text{P}_2\text{O}_5$  system, called Bioglass<sup>®</sup>, spontaneously bond to living bone without the formation of fibrous tissue on their surfaces. Since then, various kinds of synthetic materials, such as sintered HA, sintered  $\beta$ -tricalcium phosphate, biphasic ceramics of HA and tricalcium phosphate (TCP) as well as apatite–wollastonite (A–W) glass–ceramics have also been found to bond to living bone. These are now considered to be bioactive materials and are clinically used as important bone substitutes [4].

When bioactive ceramics such as bioglass, apatite–wollastonite glass ceramics or HA/ $\beta$ -TCP biphasic calcium phosphate

are implanted in bone tissue, the examination of the implant site reveals the presence of nanocrystalline calcium-deficient carbonate apatite at the bonding interface [5]. This intermediate apatite layer is similar to biological apatites in terms of calcium deficiency and carbonate substitutions, and it was believed that it would interact with osteoblast in a similar manner as biological apatites do. On the other side, when the so-called bioactive ceramics are soaked in artificial or simulated physiological fluids, the surface analysis evidence the setting off of chemical reactions at the material surface, such as dissolution, precipitation, ionic exchange, etc. together with biological material adsorption [6,7].

Later, it was found by Kitsugi et al. that A–W glass–ceramics, containing crystalline apatite and wollastonite in a glassy matrix, also form a calcium phosphate layer on its surface in the living body and bonds to bone through this calcium phosphate layer [8]. This was identified by X-ray diffraction as crystalline apatite [9]. Kokubo et al. showed that this in vivo apatite formation can be reproduced in an organic substance-free, non cellular simulated body fluid with ion concentrations nearly equal to those of human blood plasma [10,11].

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SBF is a metastable aqueous solution with a pH of about 7.4, supersaturated with respect to the solubility product of HA. This solution only contains inorganic ions in concentration almost equal to the human plasma (Table 1). The main difference between SBF and the inorganic part of the biological plasma is the bicarbonate ( $\text{HCO}_3^-$ ) concentration, which is significantly lower in SBF (4.2 mM instead of 27 mM in plasma). SBF has been widely used for in vitro bioactivity assessment of artificial materials by examining their apatite-forming ability in the fluid [12–14].

All the bone-bonding materials except beta calcium phosphate [15] and calcite [16] were also reported to form a layer of apatite or calcium-phosphate on their surfaces in SBF in the references given after the materials described above. Some glasses in the  $\text{Na}_2\text{O}$ – $\text{CaO}$ – $\text{SiO}_2$  system also bond to living bone through an apatite layer formed on their surfaces in the living body. The rates of bone formation around the glass particles decreases with increasing  $\text{SiO}_2$  content of the glasses from 50 to 70 mol% with a constant molar ratio of  $\text{Na}_2\text{O}$  to  $\text{CaO}$  of 1 [6]. The times required for the glasses to be fully covered with apatite in SBF increase with increasing  $\text{SiO}_2$  content [17].

Various types of simple metal oxide gels were treated in SBF. As a result, it was found that gels of  $\text{SiO}_2$  [18],  $\text{TiO}_2$  [19],  $\text{ZrO}_2$  [20],  $\text{Ta}_2\text{O}_5$  [21] and  $\text{Nb}_2\text{O}_5$  [22] form apatite on

their surfaces, but  $\text{Al}_2\text{O}_3$  did not [18]. This indicates that functional groups such as  $\text{Si-OH}$ ,  $\text{Ti-OH}$ ,  $\text{Zr-OH}$ ,  $\text{Nb-OH}$  and  $\text{Ta-OH}$  on these gels are effective for apatite nucleation. Tanahashi and Matsuda examined the apatite-forming ability in SBF of self-assembled monolayers with various functional groups and showed that functional groups such as  $\text{COOH}$  and  $\text{PO}_4\text{H}_2$  are also effective for apatite nucleation [23].

Apatite formation on a  $\text{Na}_2\text{O}$ – $\text{SiO}_2$  glass having  $\text{Si-OH}$  groups is described in papers by Takadama et al [24,25].

Mechanism of apatite formation (Fig. 1):

- The sintered HA is initially negatively charged on its surface and combines with positively charged  $\text{Ca}^{2+}$  ions in the surrounding fluid.
- Ca-rich amorphous calcium phosphate is formed on the sintered HA. As the  $\text{Ca}^{2+}$  ions accumulate, the sintered HA becomes positively charged on its surface and reacts with negatively charged phosphate ions.
- Ca-poor amorphous calcium-phosphate (ACP) is formed.
- This ACP is eventually transformed into more stable crystalline bone-like apatite [26].

This mechanism is also essentially the same in SBF containing protein [27].

As explained above the aim of research is to prepare HA from animal bones. Besides of converting a hazardous waste into valuable high-tech ceramics the elimination of expensive chemicals necessary for HA synthesis is also a key factor in scaling-up production to pilot scale and further industrialization. Animal bone derived HA shall have good mechanical and dissolution properties. Work described here below aims to confirm the above statement.

## 2. Materials and methods

### 2.1. Materials

The pretreated bovine bones were obtained from Alfalox Ltd., Hungary. Powdered bones with grain sizes between 63

Table 1

Ion concentrations of SBFs and human blood plasma (mM).

	$\text{Na}^+$	$\text{K}^+$	$\text{Ca}^{2+}$	$\text{Mg}^{2+}$	$\text{HCO}_3^-$	$\text{Cl}^-$	$\text{HPO}_4^{2-}$	$\text{SO}_4^{2-}$
<b>Human plasma (total)</b>	142.0	5.0	2.5	1.5	27.0	103.0	1.0	0.5
<b>Human plasma (dissociated)</b>	142.0	5.0	1.3	1.0	27.0	103.0	1.0	0.5
<b>SBF</b>	142.0	5.0	2.5	1.5	4.2	148.0	1.0	0.5
<b>i-SBF</b>	142.0	5.0	1.6	1.0	27.0	103.0	1.0	0.5
<b>m-SBF</b>	142.0	5.0	2.5	1.5	10.0	103.0	1.0	0.5
<b>r-SBF</b>	142.0	5.0	2.5	1.5	27.0	103.0	1.0	0.5
<b>n-SBF</b>	142.0	5.0	2.5	1.5	4.2	103.0	1.0	0.5

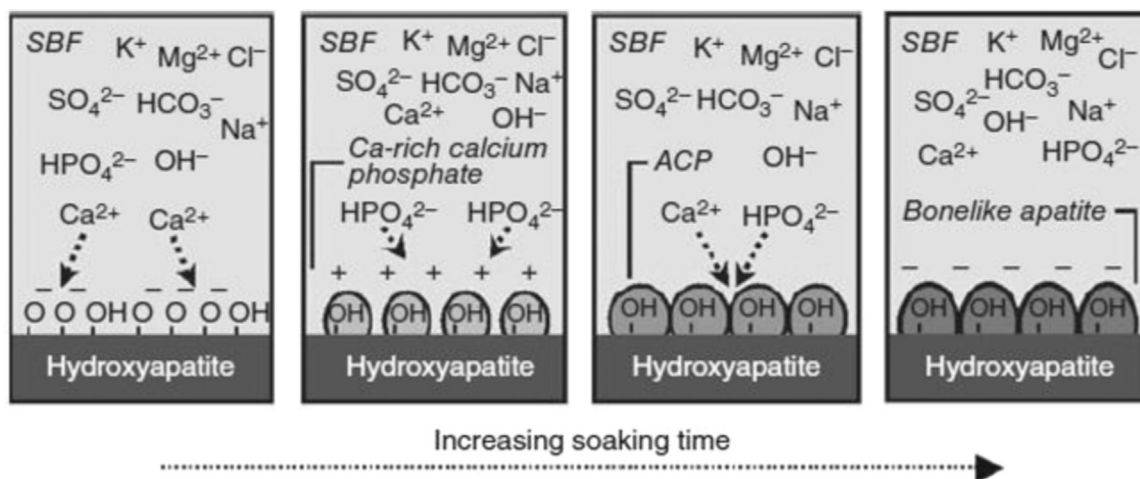


Fig. 1. Schematic representation of mechanism of apatite formation on the sintered HA in SBF [26].

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