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Mechanical stability of highly porous hydroxyapatite scaffolds during different stages of *in vitro* studies

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ABSTRACT

Degradation properties of bone scaffolds are strongly related to cell growth, host response and tissue regeneration. However, bone formation is a very slow process and, in the first few weeks after implantation, the mechanical stability of the scaffold plays a key role for the success of the regeneration progression. In the present study, *in vitro* properties of highly porous hydroxyapatite (HA) scaffolds were analysed in terms of bioactivity and biodegradation by observing their microstructure, measuring the weight loss and evaluating their mechanical stability. HA scaffolds fabricated by sponge replica method have been incubated for different periods of time in Simulated Body Fluid and in tris-HCL buffer. The morphological and mechanical characterizations performed on the scaffolds before and after immersion demonstrated the scaffolds being bioactive and almost not biodegradable in four weeks in terms of weight loss and mechanical resistance.

1. Introduction

Degradation behaviour of bone scaffolds has a crucial importance in tissue engineering being strictly linked to cell growth, host response and tissue regeneration [1]. Ideally, scaffolds should have a degradation rate matching the regeneration rate of new bone tissue. However, bone formation is a slow process and, in the first weeks after implantation, mechanical stability of the scaffold assumes a primary importance for the regeneration procedure success. Actually, although a faster remodelling process of the graft could be theoretically advantageous, from a clinical point of view, it did not appear to provide any beneficial effect in terms of failure or complications [2]. Ceramic scaffold are typically characterized *in vitro* in terms of bioactivity, degradation, cell proliferation, differentiation and matrix deposition [3]. Instead, compressive mechanical characteristics of ceramic scaffolds are usually evaluated before their permanence *in vitro* [4–6]. Though, the mechanical stability of the scaffold can be strongly affected by its microstructure, chemical and physical properties as well as environmental conditions [7,8]. Few studies investigated the effects of SBF incubation on mechanical properties of bioglass-based scaffolds produced by thermally induced phase separation [9], indirect selective laser sintering [10] or sponge replication method [11]. Recently, Arahira and Todo [12] reported the mechanical behaviour of β-TCP scaffolds at different time points of *in vitro* cell culture test. The tested scaffolds exhibited structural instability with the collapse of the porous structure when immersed in culture medium. In our previous studies, we developed macroporous HA scaffolds by sponge replica method that showed superior mechanical properties with respect to scaffolds made with a commercial powder [13]. However, the developed scaffolds were mechanically characterized before their permanence in aqueous solution. In the present study, we aim at investigating the *in vitro* biological properties of the highly porous HA scaffolds in terms of bioactivity and biodegradation by observing their microstructure, measuring the weight loss and evaluating their mechanical stability. HA scaffolds have been fabricated by sponge replica method and incubated for different periods of time in SBF and in Tris-HCL buffer. Morphological and mechanical characterizations were performed on scaffolds before and after immersion to evaluate the influence of the permanence in the two solutions on scaffold microstructure and mechanical properties.

2. Materials and methods

2.1. Scaffold preparation

HA porous scaffolds were obtained by polyurethane sponge replica method using an in lab powder synthesised by a

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precipitation method [13,14]. HA powder (70 wt%) was added to a binder solution of 2%wt polyvinyl alcohol (PVA); an organic deflocculating agent (Dolapix CE-64, 0.5%wt with respect to ceramic powder) was used to achieve the suitable viscosity of the ceramic suspension. The polyurethane sponges were cut in to cubes of 10 mm length, impregnated with HA slurry, gently squeezed to remove the exceeding suspension and dried at room temperature for 24 h. Finally the dried samples were sintered at 1300 °C to obtain the final HA scaffold [13,14].

2.2. Bioactivity and biodegradation in vitro tests

In order to evaluate the bioactivity and the biodegradability of the HA scaffolds, two groups of samples were soaked in SBF and

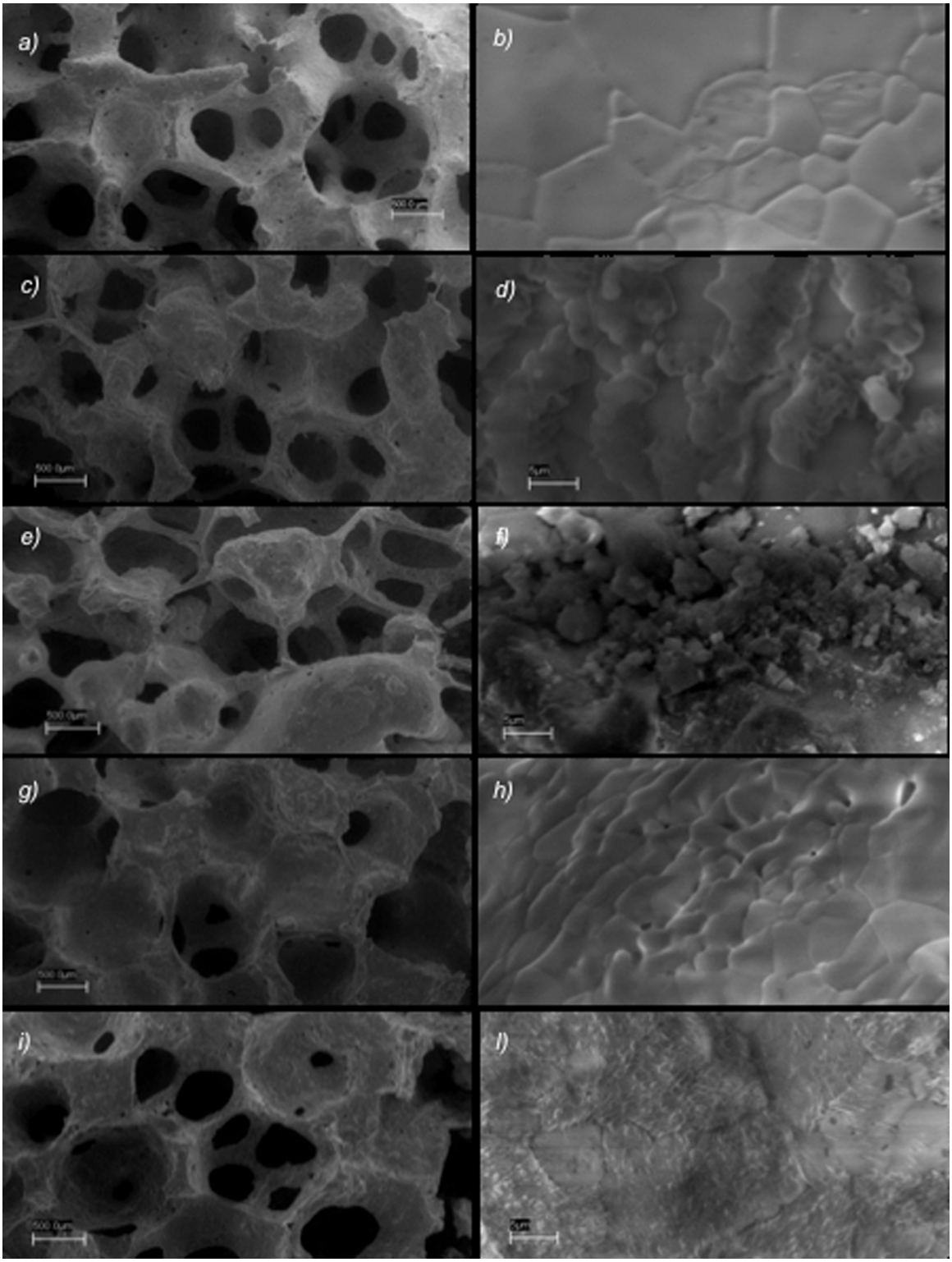

Fig. 1. SEM images of the HA scaffolds before soaking *(a, b)*; after 7 days of immersion in SBF *(c, d)*; after 28 days of immersion in SBF *(e, f)*; after 7 days of immersion in Tris-HCl buffer *(g, h)*; after 28 days of immersion in Tris-HCl buffer *(i, l). Scale bar (a,c,e,g,i): 500 μm; Scale bar (b,d,f,h,l): 5 μm.*