Short Communication

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Poly (butylene succinate) scaffolds prepared by leaching

Abstract

In recent years there was an increase in the development of new materials for use in bioengineering mainly related to bone tissue substitutes. A myriad of natural or synthetic materials can be used, such as polymers, ceramics, metals, and composites. In this work, the functionality of poly (butylene succinate), PBS, in producing scaffolds by leaching was studied. PBS is an aliphatic biopolyester, which presents excellent mechanical and osteoconductive properties. PBS is biocompatible, can allow cell adhesion and proliferation in vivo and in vitro as well. Production of scaffolds was performed using a circular die of 1 mm thickness and 8.5 mm diameter, using a hydraulic hot press. The porosity of the scaffolds was reached by the preparation of mixtures with different concentrations of PBS and NaCl. The samples were characterized aiming to observe the formation of the pores. Besides that, the material behavior depending on temperature was also performed. The obtained results allowed inferring that leaching is a fast, little expensive and efficient way to prepare PBS scaffolds.

Keywords: scaffolds, PBS, leaching, NaCl

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Abbreviations: PBS, poly (butylene succinate); Tg, glass temperature; Tm, melting temperature; PLA, polylactic acid; PCL, poly caprolactone; PGA, poly glycolic acid; PHB, poly hydroxy butyrate acid; NaCl, sodium chloride; FTIR, fourier transform infrared spectroscopy; STA, simultaneous thermal analysis; SEM, scanning electron microscopy; µCT, computerized micro tomography; PHB, poly hydroxy butyrate acid

Introduction

Biomaterials are the main focuses of scientists' efforts to develop new materials to replace or repair bone tissues.[1] Biomaterials are natural or synthetic materials which can interface with biological systems allowing the increase, repair or even the replacement of tissues and organs of the human body.[2] Within biomaterials, the polymers can be natural or synthetic as well. The main polymers used to repair bone tissue are the polyglycolic acid (PGA), polylactic acid (PLA), polyhydroxybutyrate acid (PHB) and polycaprolactone (PCL).[3–5] These polymers are useful to the "scaffolds" development in the bone tissue engineering field, acting as templates for the formation of new tissues. For this, these materials must present specific requirements such as adequated internal architecture and surface properties, besides mechanical resistance and porosity.[6,7] These biodegradable templates must be porous to the highly efficient propagation of cells and nutrients, and also for supplying oxygen.[8] Besides that, these pores should be three-dimensionally interlinked, allowing the proliferation of new bone tissue and facilitating fluid transport through the material.[9] A wide variety of natural and synthetic materials are being investigated for the design and construction of scaffolds for bone tissue engineering. Among them, the most remarkable are the natural polymers, such as gelatin, agar, fibrin or collagen; the synthetic bioresorbable polymers, such as polylactic acid (PLA), polycaprolactone (PCL); and, finally, the porous ceramics, such as bioglass and calcium phosphate structures as well as the natural ceramics, such as coral.[10–12] In this context, the poly (butylene succinate), PBS is a biodegradable aliphatic biopolyester. When compared to other similar polymers, PBS presents improved mechanical and processing properties. PBS also presents biocompatibility, being an osteoconductive material which allows cell adhesion. Literature shows PBS associated with other polymers as an efficient matrix to the preparation of scaffold.[13–16] Mostly, expensive techniques, such as electro-spinning, are used to the preparation of these materials.[16] Thus, PBS was used here to prepare scaffolds by leaching using NaCl as a cheap porogenic agent. The obtained materials were characterized using several techniques, and the results allowed inferring that leaching is a fast, little expensive and efficient way to prepare PBS scaffolds.

Materials and methods

Materials

Poly (1,4-butylene succinate), Lot#MKBQ7938V and sodium chloride were purchased from Sigma-Aldrich. All materials were used as received.

Methods

Scaffolds preparation: Scaffolds were prepared as disks of 1mm thick and 8.5mm diameter by hot molting under pressure (130°C@5MPa). For assure the formation of the pores, different amounts of NaCl were mixed to the PBS. The used proportions between PBS and NaCl were equal to 1:0.10, 1:0.25 and 1:0.50. These proportions allowed preparing PBS filled with 9.1, 20.0 and 33.3wt% of NaCl, respectively. The leaching was performed in deionized water. Drying took place at room temperature for 24 hours.

Characterization: Fourier transform infrared spectroscopy using attenuated total reflectance (FTIR-ATR) analyses were performed in a Perkin-Elmer 1720X Fourier transform spectrometer. The FTIR spectra were obtained using ATR (diamond crystal) in an inert atmosphere, with a resolution of 4cm^{-1} in the range 4000-675cm^{-1}. Stored results were averages of 124 scans. Simultaneous Thermal Analysis (STA) was performed using a PerkinElmer STA 6000. Measurements were carried out under nitrogen at a heating rate of 20°C/min up to 700°C with a gas flow rate of 20mL/min. Scanning electron microscopy (SEM) experiments were performed with a JEOL JSM-5610 LV microscope, using acceleration voltage of 15kV. Samples were coated with gold to study the morphology of the prepared materials. Computerized Microtomography (μCT) scaffolds were analyzed by μCT (SKYSCAN), with a detection array of 2240x2240 pixels, Voltage 40kV, Current 160μA, Aluminum Filter 1mm, Rotation Degree 0.6°, Pixel Size 7, 12μm, Acquisition time 1h and 50 minutes.

Result and discussion

PBS spectra of PBS and PBS: NaCl composites are shown in Figure 1. The dublets at 1331 and 1317cm^{-1} are attributed to the symmetric and asymmetric deformational vibrations of CH_2 groups in the main chain of PBS, respectively. The bands around 1155 and 1243cm^{-1} correspond to stretching of the -COC- bonds in the ester group.[17] The band at 1714cm^{-1} is related to stretching vibrations of the ester group in PBS, specifically in the C=O bonds of the carbonyl. The peaks in the 1044cm^{-1} region are related to the stretching vibrations of O-C-C bonds in this polymer.[18] It is also possible to notice a band at 917cm^{-1} corresponding to vinyl groups. The occurrence of terminal vinyl groups is attributed to the degradation reaction of the PBS chain.[19,20] Composites, in comparison with pure PBS, do not present changes in their FTIR spectra simply because NaCl does not absorb in the IR region.

The Table 1 shows the density of the prepared materials after leaching. The density analysis demonstrated that the use of increasing NaCl amounts produced a decrease of the final density and the increase of the pores in the materials. The thermal analyzes results are also shown in Table 1. The TGA and DSC were used to determine the effect of the pores on the thermal properties of the materials. The scaffolds presented melting and degradation temperatures equal to (119±2)°C and (400±5)°C, respectively. Besides that, data shown in Table 1 allows inferring correlations between density and melting and degradation enthalpies equal to 0.87 and 0.81, respectively. Thus, as the pores volume increase, the density decreases as well as the energy necessary to the melting and degradation.

Table 1 Apparent density and main thermal properties of the scaffolds

Sample	Density (g/cm³)	Melting enthalpy (J/g)	Degradation enthalpy (J/g)
PBS	1.1617±0.0108	53.9±0.7	397.1±5.4
PBS: NaCl 1:0.10	1.1483±0.0259	46.3±0.6	341.0±4.6
PBS: NaCl 1:0.25	1.1089±0.4374	40.8±0.5	337.2±4.5
PBS: NaCl 1:0.50	1.0232±0.0243	37.4±0.5	309.8±4.2

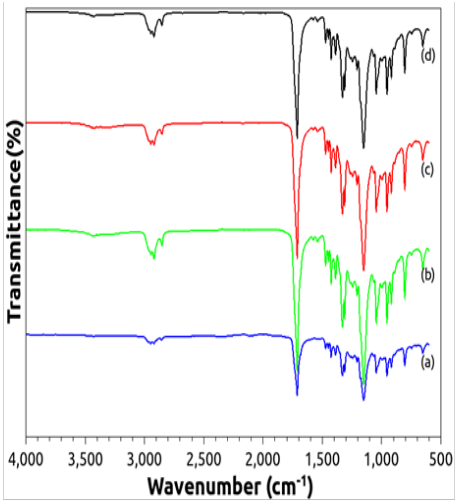

Figure 1 (A) FTIR spectra of PBS (B) PBS: NaCl 1:0.10 (C) 1:0.25 and (D) 1:0.50.

Scanning Electron Microscopy (SEM) images, shown in Figure 2, allowed inferring the increase of the pores number as more and more NaCl was used in the composites. Besides that, the diameter of pores was calculated. Samples filled with 10, 25 and 50 phr of NaCl presented average surface diameter of pores equal to 136±58μm, 232±33μm, and 231±84μm, respectively. Thus, the increase of used NaCl also produced an increase of the pore size and distribution. In turn, the microtomography of the scaffolds of PBS with 50 phr of NaCl after leaching is shown in Figure 3. The average pore diameter was also calculated. The smaller set of pores presented an average diameter equal to 329±94μm. This result is statistically equal to the one obtained from SEM. On the other hand, the microtomography also allowed inferring the presence of macropores, which presented an average diameter equal to 1831±515μm. Despite microtomography image shown non-homogeneous distribution of pores in certain parts of the scaffold, it proves the presence of pores (in blue) inside the scaffolds and the interconnectivity among them.

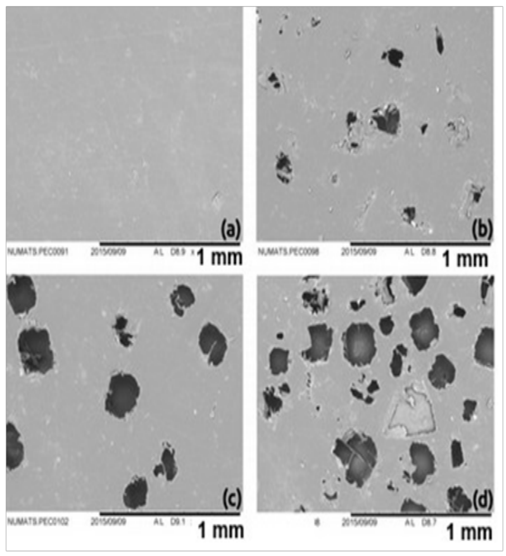

Figure 2 (A) Scanning Electron Microscopy of PBS (B) PBS: NaCl 1:0.10 (C) 1:0.25 and (D)1:0.50.

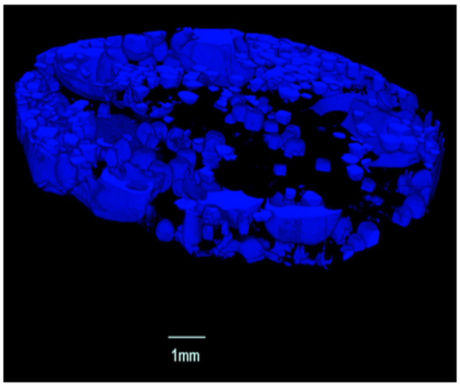

Figure 3 Computerized microtomography (µCT) of the scaffolds of PBS: NaCl 1:0.50.

Conclusion

This is a preliminary study on PBS based scaffolds, further detailed investigations are in progress and will be reported later. Thus, this work allowed proving that the production of scaffolds based on PBS by the leaching method is efficient. Therefore, this is another application for PBS, which is a platform polymer and will be more and more used as its price falls making this polymer even more present in our lives.

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Conflict of interest

The author declares no conflict of interest.

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