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Featured Letter

# Improved biocompatibility of polyurethane film by association with bioactive glass through ultrasonic implantation

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ABSTRACT

Synthetic biodegradable polymers are of great interest in the biomedical field; however, their biocompatibility is not always adequate for tissue engineering applications. Surface treatment and secondary bioactive phase incorporation have been reported as means of resolving such issues. In this work, biodegradable polyurethane (PU) film biocompatibility was improved with the incorporation of bioactive glass via ultrasonic method. Structural analysis revealed successful PU-glass composite formation, and MTT cytotoxic testing reported increased bioactivity via bioactive glass incorporation. The synthesised films furthermore exhibited great flexibility, with up to 889.3% elongation and increased maximum stress at failure. Ultrasonic implantation is therefore a promising potential method to obtain superior polyurethane films for biomedical applications.



## 1. Introduction

Polyurethanes (PU) are potential materials for biomedical applications owing to their wide range of physical and mechanical properties and easy processability [1]; however, their biocompatibility and bioactivity is not always adequate, which could cause toxic or unwanted immune effects after their implantation into the body. One strategy to overcome this issue is the association of PU with bioceramics to obtain composites with improved properties [2]. Previous works have shown that association of PU with bioactive glass (BG), a bioceramic with known osteoconductive and osteoinductive properties [3], resulted in materials with improved bioactivity and biocompatibility [1]. For example, PU coated with BG demonstrated great fixation of the glass into the PU surface in a biocompatible and bioresorbable composite [4], and materials for potential application in bone tissue engineering were developed by the association of PU and 85S bioactive glass [5]. Nonetheless, in spite of the extensive works on the association of biopolymers and bioceramics [3], only a few research groups reported PU-BG composites [6].

In a previous work [9], we synthesized PU films for biomedical applications with high deformation at break, hydrolytic degradation capability, and hydrogel behavior. Nonetheless, these materials produced a moderate cytotoxic effect. In this work, sol-gel-derived BG was incorporated on PU films under ultrasonic energy; in this process, the polymer is immersed in a solution capable of swelling and softening its surface, and by the application of ultrasonic energy, the bioceramic particles present in the solution are fixed into the softened material, resulting in a macroscopic composite with improved biocompatibility and bioactivity [7,8]. The physicochemical properties and cytotoxicity of these materials were evaluated. This study shows that a simple and fast process can be applied to improving the PU properties.

## 2. Materials and methods

### 2.1. Synthesis of PU films

Polycaprolactone triol (PCL Triol 900, *Sigma-Aldrich*), polyethylene glycol (PEG 600, *Sigma-Aldrich*), hexamethylene diisocyanate (HDI, *Sigma-Aldrich*) and glycerol (*Synth Brazil*) were used to obtain PU films as previously described [9]. The HDI and glycerol contents were maintained at 34% and 5% (wt%), respectively, while the PCL Triol 900 and PEG 600 contents were varied: PU3 (3% PCL Triol 900 and 58% PEG 600) and PU12 (12% PCL Triol 900 and 49% PEG 600).

### 2.2. Ultrasonic process

The Stöber process was used to obtain the BG in a $SiO_2$–CaO–$P_2O_5$ system, as previously described [10]. PU films and BG (10% BG/90% polymer, wt%) were immersed in a 40% acetone and 60%

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ethyl alcohol (volume) solution mixture contained in Falcon tubes; these solvents have the ability to swell and soften the surface of the polymer substrate. The tubes were sealed and placed in an ultrasonic bath (1 $dm^3$, 40 W) for 30 min. Then, the PU films containing BG were removed from the solution, washed with distilled water, and dried at 60 °C for 24 h. The composite samples were named PU3BG and PU12BG, using PU3 and PU12 associated with BG, respectively.

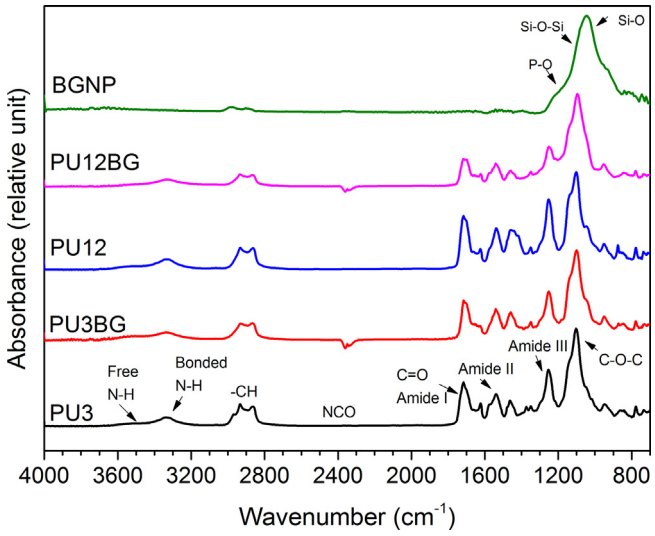


**Fig. 1.** FTIR spectra of samples.

### 2.3. Materials characterization

Fourier transform infrared (FTIR) spectra were recorded on a Thermo Scientific Nicolet 6700 OMNI-Smart Accessory Spectrum FTIR spectrometer (64 scans per spectrum; 4 $cm^{-1}$ resolution). The morphology was observed using a FEI INSPECT S50 scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectroscope (EDS-EDAX Genesis) at 15 kV. The mechanical behavior was evaluated according to ASTM D882-12 using an EMIC-DL300 universal testing machine under a 50-N load cell at a crosshead speed of 20 mm/min in ambient temperature, using five test specimens in each test.

Degradation assay was evaluated from the weight loss of samples in a simulated body fluid (SBF) [11], in which the samples were immersed for different periods up to 90 days at 37 °C and 75 $mg.ml^{-1}$ ratio. The samples were weighed before immersion and after removal and drying.

Toxicity assay was conducted by MTT as previously described [9], using human osteoblast (SAOS) cells provided by Prof. Goes (UFMG, Brazil). ISO standard 10993-5:1999 was used as a reference. Controls using cell and DMEM medium (10%) were created; Triton X-100 (1% v/v, *Sigma Aldrich*) was used as positive control and chips of sterile polypropylene (1 $mg.ml^{-1}$, *Eppendorf*) as negative control.

## 3. Results and discussion

Typical PU and BG absorption bands can be observed in the FTIR spectra (Fig. 1), and previous reports were used as reference [10,12]. The absence of isocyanate stretching in the range of

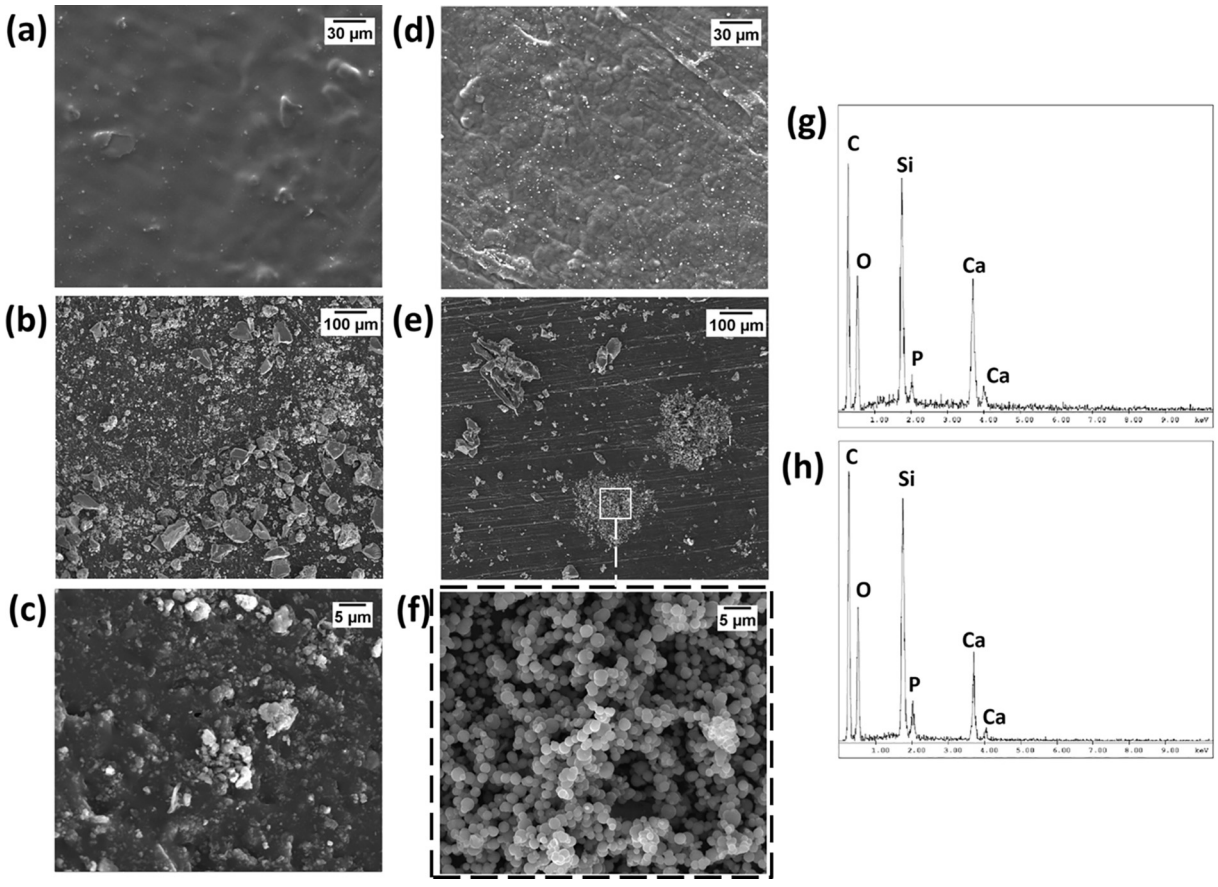


**Fig. 2.** SEM micrographs of (a) PU3, (b) and (c) PU3BG, (d) PU12, (e) and (f) PU12BG and EDS maps of (g) PU3BG and (h) PU12BG.