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Effect of air plasma treatment on mechanical properties of bioactive composites for medical application: Composite preparation and characterization

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A B S T R A C T

The aim of this study was to fabricate unidirectional bio-glass fibers (BGF) (13-93)-reinforced polylactic acid (PLA) composites with improved mechanical properties for a bone plate to heal the weight-bearing long-bone fractures. Various exposure times of air plasma (30, 60, 90 and 120 s) on BGF fibers were studied, and the most suitable conditions were determined for the fabrication of the BGF/PLA composite. Mechanical properties, microscopic characteristics, *in vitro* degradation and the bioactivity of the BGF/PLA composites were evaluated. Fatigue life of 30 s plasma treated composites completed 1 million cycles when actual loading conditions (10–20% body weight) were used. 30 s plasma treated specimens showed increase in tensile strength, flexural strength and interlaminar shear strength as 31%, 13.5% and 33%, respectively. The SEM image of fractured surface of 30 s plasma treated composite suggested that the failure was shifted to the PLA which is evidence of superior bonding between fiber and PLA matrix. Moreover, a bone-like calcium-phosphate layer was precipitated on the surface of degraded composite that is essential for bone healing.

1. Introduction

Over the last few decades, biodegradable materials have been of great interest for use in orthopedic devices such as pins, bone plates, intramedullary rods, screws, and so on. Polylactic acid (PLA) alone shows poor mechanical properties for weight-bearing fixation devices. Therefore, reinforcements such as magnesium alloys, ceramics, and glass have been mixed with the PLA to obtain sufficient mechanical properties [1,2]. Phosphate glass fiber (PGF)-reinforced polymer composites offer excellent mechanical and biological properties for the repair of bone fractures [3]. It was reported that 18% random and 14% unidirectional (UD) PGFs by volume provided 110–120 MPa of flexural strength and 6–9 GPa of flexural modulus [4], which is not enough for bone plates used in weight bearing long bones fractures [5]. To analyze the performance of various types of fracture fixation devices, finite element models are extensively used to estimate the healing process of bone fractures [6–11].

Scientists have shortlisted the most effective chemical treatments which improve the interfacial mechanical properties of glass fibers and polymer matrices, which include 3-aminopropyltriethoxy saline (APS), hexamethylene diisocyanate (HDI), and sorbital-ended PLA oligomers (SPLA) [12]. These chemicals effectively improve the interfacial properties of composites; however, they also induce poor mechanical interlocking between the glass fiber and the polymers. To improve the mechanical interlocking in PGF/polymer composites, magnesium, which is a bioresorbable metal, has been coated on the surfaces of glass fibers and then fabricated into a composite material of magnesium-coated PGF with PCL and PLA matrices. Magnesium-coated PGF/PCL composite showed good strength; however, PLA did not show good strength with magnesium-coated PGFs [13,14]. The interfacial properties of the PGF/PLA composites were not improved using a magnesium coating on the PGFs. Kharazi et al. developed resorbable bioactive composite for bone plates using BG (13-93)/PLLA with different chemical treatments and their degradation properties were evaluated [15]. Plasma treatments are widely used to enhance the interfacial adhesion of composite materials [16], however, no study was found that considered the plasma treatment on BGF/PLA composites.

The aim of this study is to fabricate the BGF/PLA composites treated with air plasma to evaluate their mechanical and biological properties for the purpose of the design of bone plates used in

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long-bone fractures. Atmospheric air plasma treatment of different durations (30, 60, 90, and 120 s) was done on the surface of BGF to improve the bonding characteristics with the PLA matrix and the optimal duration of air plasma treatment was determined. Changes in mechanical properties, fracture mode, *in vitro* degradation, microscopic characteristics including SEM and EDS, and bioactivity of the BGF/PLA composites were evaluated. Finite element analysis was used to determine the stresses in a bone plate in working condition and fatigue life of the BGF/PLA composite was experimentally estimated accordingly.

2. Materials and specimen preparation

2.1. Fabrication of unidirectional fibers mats

Continuous unidirectional (UD) bio-glass fibers (BG 13-93) with diameters of 20–40 µm were prepared (MO-SCI HealthCare, USA). The composition of BGF was SiO_2 53% wt, Na_2O 6% wt, K_2O 12% wt, CaO 20% wt, P_2O_5 4% wt and MgO 5% wt. The fibers were cut into 130 mm lengths and manually aligned on a steel plate covered with a Teflon film. A Cellosize™ (hydroxyethyl cellulose) (SIGMA–ALDRICH) solution was prepared by dissolving 2.75 g of powder in 1.0 L of deionized water and stirred at 650 rpm for 30 min. The solution was then sieved through a fine mesh in order to remove any residuals. The Cellosize™ solution was sprayed on the fibers using a pump and sonicating sprayer (Qsonica LLC, USA) to bind the fibers together, as shown in Fig. 1a. The fiber mats were then dried in a drying oven at 50 °C for 1 h.

2.2. Fabrication of PLA films

PLA (3251-D) pellets as a matrix were prepared (average molecular weight M_w = 90,000–120,000, melting temperature T_m = 170.9 °C, and glass transition temperature T_g = 61.3 °C). PLA pellets were dried in a drying oven at 50 °C for 24 h prior to use. PLA films with average thicknesses of 0.25 mm and 0.31 mm were fabricated

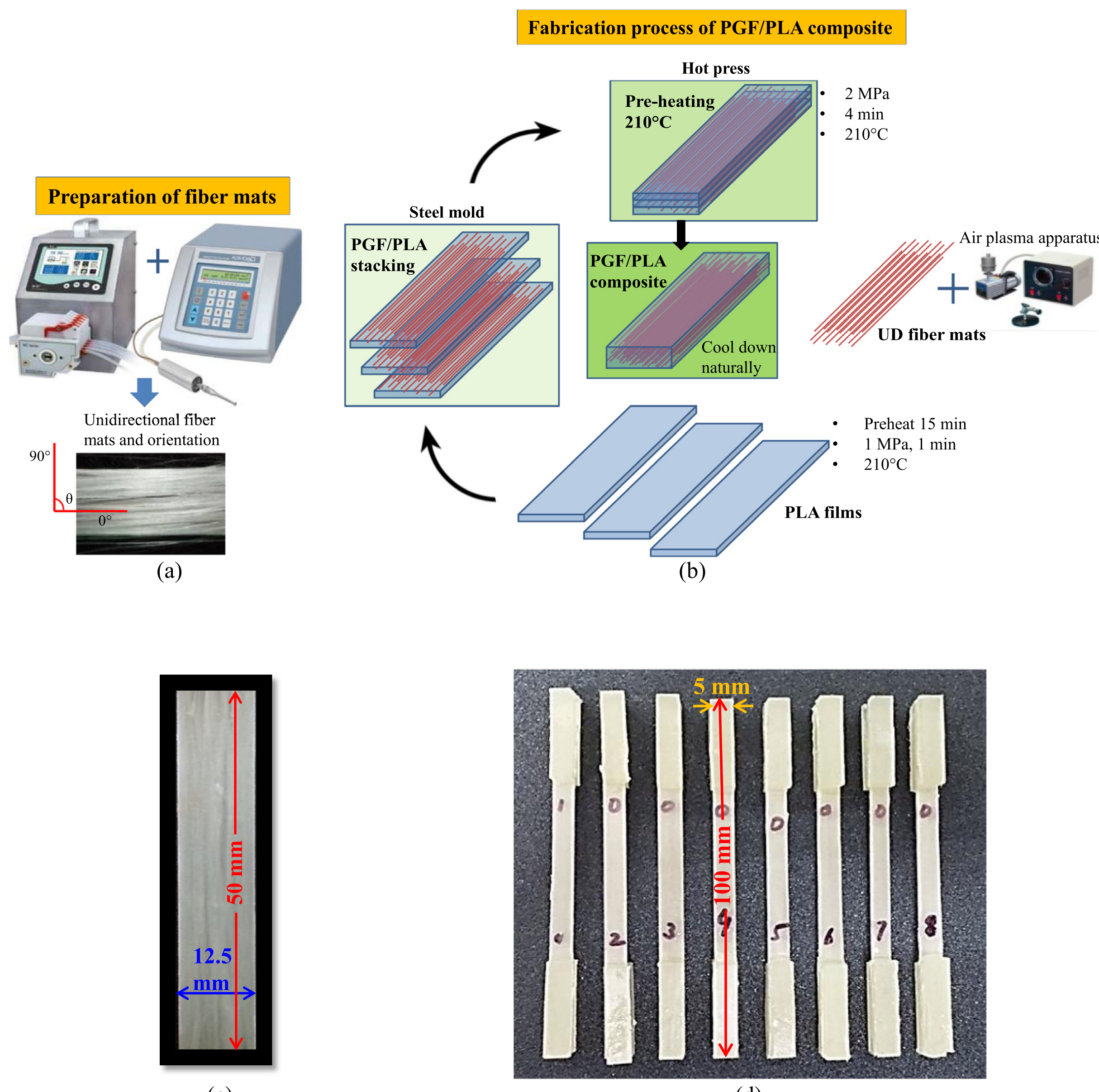

Fig. 1. Preparation of PGF/PLA composite for mechanical testing: (a) pump and sprayer for the preparation of unidirectional PGF mats, (b) stepwise fabrication process of BGF/PLA composite, (c) PGF/PLA composite specimen for bending test, and (d) PGF/PLA composite specimen for tensile and fatigue tests.