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Material properties

Mechanical and thermo-mechanical behaviors of sizing-treated corn fiber/polylactide composites

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ABSTRACT

Sizing treatment of glass fiber has been shown to effectively improve fiber-matrix interfacial adhesion whereas few studies have been conducted on the sizing treatment of natural fibers. In this work, corn fiber was subjected to alkali and sizing treatments. The treated corn fibers were used to prepare corn fiber reinforced polylactide (CF/PLA) composites through mechanical mixing and injection molding. Fiber surface, tensile fracture surface, mechanical properties and thermo-mechanical behavior of various CF/PLA composites were characterized. SEM observation indicates better interfacial adhesion between sizing-treated corn fiber and PLA, as compared to untreated and alkali-treated corn fibers, due to the interfacial reactions at fiber-matrix interfaces. Sizing-treated CF/PLA composites demonstrate improved mechanical properties and thermo-mechanical behavior as compared to PLA composites containing untreated and alkali-treated corn fibers. It is demonstrated that sizing treatment of corn fiber could be a potential and promising method to produce high performance biocomposites.

1. Introduction

The use of fibers from agricultural byproducts or agro-waste materials as reinforcements in green composites offers a low cost and environmentally friendly solution to waste disposal [1] and can alleviate the resource shortage of wood, particularly in countries that have little or no wood resources left [2]. Among all agricultural byproducts, including stalks of most cereal crops, rice husks, coconut fibers, bagasse, maize cobs and peanut shells, corn fiber stands out with some favorable characteristics such as lower cost (which is about 90% cheaper than agricultural fibers [3]) and more abundance than other natural fibers. Furthermore, rather than cultivating those natural fibers (such as sisal, jute, bamboo, ramie, flax and hemp) by occupying large areas of land, replacing them with agricultural byproducts like corn fiber favors sustainable development. As a result, making full use of corn fiber should be the more attractive approach. However, less progress has been made on the development of corn fiber green composites as compared to other natural fibers composites, probably owing to the relatively poor mechanical properties of corn fiber composites [4-6].

It is well documented that one of the most remarkable disadvantages of natural fibers is their poor compatibility with the matrix materials due to their hydrophilic surfaces. Therefore, surface modification is a mandatory step in the

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preparation of natural fiber-reinforced polymer composites. To date, numerous methods such as alkali treatment, silane treatment, enzyme treatment, benzoylation treatment and maleated coupling have been reported [7-10], among which alkali treatment and silane treatment are the most widely used and effective methods [11,12]. Unlike the aforementioned methods, surface treatment with sizing agents can not only improve handling properties of fibers, protect the fibers from fuzzing and fragmenting during composite processing, but also enhance fiber wetting by the matrix and, in particular, improve fiber-matrix adhesion due to the presence of functional groups on sized fibers that can react with the matrix [13-15]. Although extensive research has been carried out regarding sizing treatment of conventional glass and carbon fibers [16-20], few investigation has been carried out on the sizing treatment of natural fibers [21] and no report can be found on the sizing treatment of corn fiber.

The present paper describes the preparation of corn fiber reinforced polylactide (abbreviated as CF/PLA) green composites by simple and scalable mechanical mixing followed by injection molding. The selection of PLA is based on the fact that it is the most widely commercialized and readily available bio-based thermoplastic polyester that is derived from renewable resources such as corn, beet and sugar. For comparison, CF/PLA composites with untreated, alkali-treated and sizing-treated corn fibers were fabricated and their mechanical and thermo-mechanical properties were assessed. In addition, the effect of sizing concentration was studied. The emphasis was placed on the effect of sizing treatment on the properties of CF/PLA composites.

2. Experimental

2.1. Materials

The PLA as matrix material was supplied by Shenzhen Guanghua Weiye Industrial Co. Ltd, China, in the form of pellets with a melt flow index of 12 g/10 min. Corn stalks were obtained from a farm in Hebei Province, China. The sizing material used in this work was an isocyanate modified epoxy emulsion. Both the sizing material and silane coupling agent KH550 (γ-aminopropyl triethoxysilane, molecular structure $H_2NCH_2CH_2CH_2Si(OC_2H_5)_3$) were purchased from Nanjing Chemical Industry Group Co. Ltd, China. Ethanol, analytical grade, was provided by Tianjin Chemical Reagent Co., Ltd, Tianjin, China.

2.2. Sample preparation

2.2.1. Preparation of corn fiber

Corn stalks were cleaned and dried and straw skins were obtained by a skin separator (PRFII-0.2, Jilin Fenghe Botanical Development Co. Ltd, China) from corn stalks. The skins were smashed to pieces using a JWF-250 Cutting Mill (Hongle Machinery Plant, Xingyang, Henan, China). The corn husks and debris generated in the process of crushing were removed with a 40-mesh sieve and finally corn fiber samples with an approximate length of 3 mm were obtained.

2.2.2. Preparation of sizing agent solutions

Typically, silane coupling agent KH550 was hydrolyzed in an ethanol aqueous solution (deionized water to ethanol ratio = 3:1 by weight) for 20 min at room temperature, and the percentage of silane agent was kept at 1.0 wt%. The sizing material was added to the solution of KH550 with stirring at a rate of 250 rpm until a homogeneous solution was formed. The concentrations of sizing agent were set at 8, 12 and 16 wt%, respectively, in the final sizing solution.

2.2.3. Sizing treatments of corn fiber

Corn fiber was first treated with alkali prior to sizing treatment. The process of alkali treatment was similar to that described by Asumani et al. [22]. Typically, corn fiber was soaked in sodium hydroxide (NaOH) solution (at an optimal concentration of 8 wt%) at room temperature for 4 h, followed by rinsing with water and then 1% acetic acid until pH reached neutrality. The alkali-treated corn fiber was oven-dried at 95 °C followed by immersion in various sizing solutions with concentrations of 8, 12 and 16 wt% for 30 min. Subsequently, the sizing-treated corn fiber samples were removed from the solutions and dried in an oven at 80 °C for 48 h.

2.2.4. Preparation of CF/PLA composites

PLA and corn fibers with varying surface conditions were blended at ambient temperature for 8 min at 60 rpm using a SLH-0.01 internal mixer (Shanghai Dongqiu Mixing Machine Co. Ltd., Shanghai, China). The mixed materials were then dried at 105 °C for 4 h. Finally, test specimens were injection molded at 190 °C and an injection pressure of 70 MPa. The fiber volume fraction in all composite samples prepared in this work was kept at 20%. Untreated, alkali-treated and sizing-treated corn fibers were used to reinforce PLA, and the resultant composites are denoted as UCF/PLA, ACF/PLA and SCF/PLA, respectively, as listed in Table 1.

2.3. Mechanical testing

Injection-molded specimens were tested following ISO 527-1 for tensile properties and ISO 178 for flexural properties. Tensile and flexural tests were performed using a CMT-4304 universal testing machine (Shenzhen Suns Co. Ltd., China). The crosshead speed for tensile and flexural tests was 5 and 1.4 mm/min, respectively. Notched Izod impact tests were conducted using a XCJD-50 impact tester (Chengde Puhui Testing Instrument Co. Ltd, Hebei, China) as per ISO 179-1:2010. All mechanical tests were performed

Table 1
Composite samples prepared in this work and their designations.

Sample designation		Volume content of corn fiber (%)	Concentration of NaOH (wt%)	Concentration of sizing agent (wt%)
UCF/PLA		20	–	–
ACF/PLA		20	8	–
SCF/PLA	SCF8/PLA	20	8	8
	SCF12/PLA	20	8	12
	SCF16/PLA	20	8	16