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Mechanical, moisture absorption, and biodegradation behaviours of bacterial cellulose fibre-reinforced starch biocomposites

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ABSTRACT

In this work, bacterial cellulose (BC) nanofibres were used as the biodegradable reinforcement. The BC nanofibres were incorporated in the starch plasticised with glycerol via a solution impregnation method. Tensile properties of the BC/starch biocomposites were tested and compared with those of the unreinforced starch. Moisture absorption of the biocomposites under 75% RH at 25 °C was analysed. The kinetics of sorption–diffusion process was investigated and typical kinetic parameters D, k, and M_∞ were determined. Additionally, the BC/starch biocomposite (15.1 wt.% BC) and starch were submitted to biodegradation by soil burial experiments in perforated boxes. Tensile strength after exposure to moisture and microorganism attacks was measured. It is found that the moisture sorption mechanism in the BC/starch biocomposites follows a Fickan diffusion mode. The presence of BC nanofibres improves the tensile properties and the resistance to moisture and microorganism attacks. The mechanisms behind these phenomena are discussed in this paper.

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1. Introduction

In recent years, natural fibres have attracted much attention as reinforcements for both thermoplastic and thermosetting polymer composites. It should be pointed out that a lot of previous work focused on developing partially biodegradable composite materials including natural fibres reinforced non-biodegradable matrices [1,2] or non-biodegradable fillers such as carbon nanotubes reinforced biodegradable matrices [3,4]. Currently, more and more researchers are developing fully biodegradable composites, the so-called 'green' composites, ecocomposites, or biocomposites which are composed of natural fibres and natural matrices [5] or synthetic biodegradable matrices such as poly (lactic acid) (PLA) [6]. To date, numerous natural fibres such as cotton, hemp, sisal, jute, flax, ramie, coir, and cellulose have been explored [7–9] and a lot of 'green' matrices such as starch and cellulose have been employed [10,11].

Cellulose, the most abundant natural homopolymer, is considered to be one of the most promising renewable resources and an environmentally friendly alternative to products derived from the petrochemical industry. Plant derived cellulose has been widely used as either reinforcement [2,7,10,12] or matrix [10] and even as the sole component to prepare all-cellulose composites [11]. Besides cellulose from plants, cellulose is also secreted extracellularly as synthesized cellulose fibres by some bacterial species, which is called bacterial cellulose (BC) or microbial cellulose (MC). Though identical in chemical composition, the mechanical properties and structure of BC differ from those of plant cellulose. It has high mechanical properties like tensile strength and modulus. Compared with cellulose from plants, BC possesses higher water holding capacity, higher crystallinity, and a finer web-like network. BC has been widely used in foods, in acoustic diaphragms for audio speakers or headphones, for making unusually strong paper, and has medical applications as wound dressings and artificial skins, artificial blood vessels, and tissue engineering scaffolds [13–16].

Recently, BC has been used as reinforcements for various composites due to its excellent mechanical performance [17–20]. For instance, Gindl and Keckes prepared cellulose acetate butyrate-based composites reinforced with BC sheets [18]. Yano et al. demonstrated the production of nanocomposites made with BC nanofibres and acrylic resin [17]. High-strength BC/phenolic resin composites were produced by Nakagaito et al. [20]. Compared to those aforementioned matrices, starch shows its uniqueness. It is fully biodegradable in a wide variety of environmental conditions. As a result, various starch-based composites have been prepared [12]. However, there is little literature regarding the combination of BC and starch [21]. So far no information on mechanical properties, moisture absorption and environmental biodegradability of the BC/starch composites has been reported. To this end, biocomposites based on starch and BC fibres were developed by a solution impregnation method in this work. The main objective of the present study is to characterise the mechanical, moisture absorption,

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biodegradation properties of the BC/starch composites. These basic data are necessary for the design and use of the resultant biocomposites.

2. Experimental procedures

2.1. Materials

The starch used in this work was commercial industrial-grade wheat starch, supplied by Kewei Chemicals, Tianjin, China. The glycerol (99.5% purity), the plasticizer, was purchased from Aldrich and used as received.

The preparation procedures were reported elsewhere [16]. Briefly, the bacterial strain, *Acetobacter xylinum* X-2, was incubated for 7 days in a static culture containing 0.3% (w/w) green tea powder and 5% (w/w) sucrose. The obtained gel-like BC pellicles about 10-mm thick were purified by immersion in deionized water at 90 °C for 2 h and then boiled in a 0.5 M aqueous solution of NaOH for 15 min. The BC pellicles were then washed with deionized water several times and soaked in 1% NaOH for 2 days. Finally, the BC pellicles were washed free of alkali.

2.2. Fabrication of BC/starch biocomposites

The BC/starch biocomposite samples were prepared by the solution impregnation method. Starch and glycerin, 30% w/w of glycerin to starch, were mixed prior to the addition of water. Solutions containing starch and glycerol were stirred at 80 °C for 30 min. The pH of the resulting solutions was kept at 3–4 and the concentration varied from 10 to 20 (w/v)%. Then air-dried BC sheets were immersed in the solutions. The immersed mats were maintained in reduced pressure at 0.03 MPa for 12 h and kept under ambient pressure at 25 °C over 96 h. The impregnated sheets were taken out of the solutions, cut into smaller pieces of 50 mm by 30 mm, air dried at 37 °C. The obtained composite sheets had a thickness of 0.5 mm and varying fibre loadings of 7.8, 15.1, and 22.0 wt.%. The amount of BC fibre in the composites was calculated as a percentage of the total dry weight of starch plus glycerin.

2.3. Mechanical testing

Test specimens with a length of 30 mm and a width of 10 mm were cut from composite sheets. All specimens were equilibrated in a chamber kept at 20 °C and 35% relative humidity for 24 h before testing. The tensile strength of the plain matrix and all biocomposites was measured by employing a M350 universal tensile machine following ASTM D 5937-1996. The crosshead speed was set at 5 mm/min. All these tests were conducted at ambient temperature and an average value of five repeated tests was taken for each material.

Samples after moisture absorption (see Section 2.4) or biodegradation (see Section 2.5) were also tested to determine the tensile strength. Tensile strength retention was determined as

$$\text{Tensile strength retention} = \frac{\tau_t}{\tau_0} \times 100 \quad (1)$$

where τ_t and τ_0 are, respectively, the tensile strength after moisture absorption or after soil burial degradation and the initial tensile strength.

2.4. Moisture absorption experiments

Moisture absorption measurements were performed under 75% RH (relative humidity) at 25 °C. All specimens for moisture absorption experiments with dimensions of 30 mm by 10 mm were cut from composite sheets. Prior to the absorption experiments, all specimens were thoroughly washed and then vacuum dried until a constant weight was attained. At predetermined intervals, specimens were taken out from the chambers and weighed using a TG328-A model analytical balance (with a precision of 0.1 mg). The moisture uptake at any time points as a result of moisture sorption was determined by

$$\text{Moisture uptake} = \frac{W_h - W_0}{W_0} \times 100 \quad (2)$$

where W_h and W_0 denote, respectively, the weight of humid specimens and the original dry value (the initial weight of materials prior to exposure to moisture absorption). All data from three repeated tests were averaged.

2.5. Soil burial degradation experiments

Soil burial degradation experiments were carried out at ambient temperature under moisture controlled conditions. Triplicate specimens of each composite were placed in a series of perforated boxes containing moisturized soil. The specimens (30 × 10 mm) were buried 100 mm beneath the surface of soil which was regularly moistened with distilled water. The samples were removed at predetermined time points, carefully washed with distilled water several times in order to ensure the stop of the degradation, dried at room temperature to a constant weight and then were stored in darkness until testing. The specimens were weighed on the TG328-A model analytical balance in order to determine the average weight loss:

$$\text{Weight loss} = \frac{W_0 - W_t}{W_0} \times 100 \quad (3)$$

where W_0 is the initial mass and W_t is the remaining mass at any given time, t. All results are the average of three replicates.

2.6. SEM observations

Tensile facture surfaces were examined by scanning electron microscopy (SEM). Prior to SEM observation, all samples were sputter coated with a thin layer of gold to avoid electrical charging.

3. Results and discussion

3.1. Mechanical properties

Tensile properties of the BC/starch composites with different fibre loadings are presented in Table 1. For comparison, the values for the pure starch are also presented in this table. The tensile strength and Young's modulus are higher for the composites compared to those of the unreinforced starch. An increasing trend in tensile strength and modulus with fibre content is found from Table 1. The tensile strength of the BC/starch composites is 2.03, 2.18, and 2.37 times of the pure starch when the fibre loading is 7.8, 15.1, and 22.0 wt.%, respectively. The tensile modulus increases by 111.7%, 116.7%, and 132.4%, respectively, at 7.8, 15.1, and 22.0 wt.% fibre loadings. This enhancement indicates

Table 1
Typical tensile properties of BC/starch composites.

BC content (wt.%)	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)
0.0	13.1 ± 0.25	155.1 ± 2.2	39.4 ± 0.6
7.8	26.72 ± 0.68	328.3 ± 1.5	6.7 ± 0.1
15.1	28.55 ± 1.12	336.1 ± 1.8	5.4 ± 0.1
22.0	31.06 ± 0.89	361.4 ± 1.9	5.3 ± 0.1

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