



Mechanical and thermal properties of poly(lactic acid) composites with rice straw fiber modified by poly(butyl acrylate)

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ABSTRACT

The rice straw fiber (RSF) was modified by suspension polymerization of butyl acrylate (BA) monomer. The results of Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) indicated that poly(butyl acrylate) (PBA) was adsorbed and coated on RSF. The biodegradable composites were prepared with the modified rice straw fiber (MRSF) and poly(lactic acid) (PLA) by HAAKE rheometer. Mechanical properties showed that the tensile strength of PLA/MRSF composites ($W(\%) = 7.98$) increased by 6 MPa compared with blank sample. The water absorption of the PLA/MRSF composites was lower than PLA/RSF composites. The thermal properties of these composites were investigated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The TGA results confirmed that thermal stability of PLA/RSF composites increased with the PBA increasing. The DSC data showed that RSF played a role as a nucleating agent and PBA made crystallization of PLA more difficult and incomplete.

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1. Introduction

Humanity is facing increasingly difficult challenges in life despite the great advancements in science and technology over the last century. All the needs of modern society, e.g., food, fuel, energy, and materials, are highly dependent on diminishing fossil resources [1]. Rising oil prices and increased environmental awareness have contributed to the advance of research on and development of biocomposites [2]. Biocomposites composed of biodegradable polymer and biofiber (natural fiber) as matrix material and reinforcing element, respectively, have been attracted attention from the viewpoint of protection of the natural environment in recent years [3–5]. Biocomposites show excellent performances such as high strength and stiffness, great versatility, and processing advantages at favourable cost, which make them be extensively used in many applications, ranging from aerospace technology to the automobile industry [6].

Biodegradable polymers as matrix material of biocomposites can reduce the reliance on fossil fuels [7]. The usual biodegradable polymers include poly(butylene succinate) (PBS),

poly(caprolacton) (PCL), poly(hydroxyalkanoates) (PHA), and poly(lactic acid) (PLA), and so on [8,9]. Poly(lactic acid) (PLA) is a degradable thermoplastic polymer with excellent mechanical properties and produced on a large scale from fermentation of corn starch to lactic acid and subsequent chemical polymerization. Pure PLA can be degraded into carbon dioxide, water, and methane with long-term using, compared to other petroleum plastics [10,11]. Moreover, PLA is applied widely [12]. It can be used in either the industrial packaging field or the biocompatible/bioabsorbable medical device market. It can be easily processed into molded parts, film, or fiber, and so on [1,13]. However, the high price and brittleness of PLA currently limit its application and considerable efforts have been made to improve these characteristics of the polymer [14–16].

The nature fiber comes from stalks, leaves, and seeds, such as jute, kenaf, hemp, sisal, flax, wheat straw and rice straw. [17,18]. Compared to synthetic fiber, natural fiber has many advantages such as recoverability, biodegradation, flammability, non-toxicity, and other excellent properties [19,20]. As the plant resources, rice is widely cultivated in China. The gross amount of rice straw reached 0.16 billion ton each year. Rice straw is not only a potential source of energy but also a value-added by-product [21]. At present, rice straw is poorly utilized, about 45–60% incinerated and discarded, which not only waste organic fertilizer source, but also pollute the environment. Therefore, the use of rice straw is significant to either environmental protection or cost of material [22]. Among the many types of natural fillers, rice straw fiber (RSF) has not attracted much

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attention because of low content of cellulose (28–48%) [23–25]. It was reported that the physico-chemical properties of RSF could be modulated by various derivatization techniques. RSF can be used as filler material in the PLA matrix property enhancement and to reduce costs. However, only a few numbers of studies have been so far reported for biodegradable PLA composites based on RSF due to the weak interaction between hydrophilic RSF and hydrophobic PLA [20,23].

In this investigation, composites consisting of PLA and modified rice straw fiber (MRSF) with poly(butyl acrylate) (PBA) were prepared to achieve improved mechanical and thermal properties. PBA should be more compatible than rice straw with PLA because of the interaction of the ester groups of the both, the PLA matrix and PBA [26]. PBA was chosen here to modify compatibly properties of the hydrophilic RSF and hydrophobic PLA because it is a hydrophobic polymer which adsorbed and coated on RSF. To evaluate the modified effect of PBA, the mechanical and thermal properties were characterized by tensile test, water absorption test, DSC, and TGA.

2. Experimental

2.1. Materials

Poly(lactic acid) (PLA A306 injection grade) in pellet form was obtained from Biopla Products Factory (Ningbo, China). It has a specific gravity of 1.2 and a melt flow index around 10–13 g/10 min (190 °C/2.16 kg). Its glass transition temperature (T_g) and melting temperature (T_m) are 55–60 °C and 150–155 °C, respectively. It was dried in a vacuum oven at 80 °C for 6 h before preparation of composites. Rice straw fiber (100–300 μ m) obtained from commercial sources, were dried in a vacuum oven at 100 °C for 6 h to remove moisture before used. Azobisisobutyronitrile (AIBN) (China) was used as the initiator for suspension polymerization reactions. Butyl acrylate (BA) (Tianjing Chem. Reagent Co., China) was purified of inhibitor upon distillation under reduced pressure and kept refrigerated until used. Polyvinyl alcohol (PVA) (1750 \pm 50) was purchased from Shanghai Reagent Co. (China). Distilled water was applied for all the polymerization and treatment processes.

2.2. Preparation of MRSF

Dried RSF and BA monomer were well-mixed in a three necked round bottom flask under ultrasonic at temperature of 20 °C, for 30 min. And then, a certain quantity of initiator, dispersant, and distilled water was added. The system was under nitrogen atmosphere and equipped with a water bath, mechanical stirrer, reflux condenser and thermometer. The weight ratio to RSF and BA monomer was 100:15, 100:20, 100:25, 100:30, 100:40, 100:50, 100:80, 100:100, 100:120 and 100:150, respectively, RSF: distilled water was 1:20 and BA monomer:AIBN:PVA was 100:3:2. The nitrogen atmosphere and agitation were maintained throughout the experiment. After reaction for 6 h at 75 °C, MRSF was obtained by leaching the reaction solution with the buchner funnel and was washed with distilled water several times. And then, the washed MFSF was dried in an oven at 80 °C for 8 h to remove moisture and then was further dried in a vacuum oven at 100 °C for 4 h to remove residual moisture.

The percentage of weight of PBA reacted on RSF, $W(\%)$, and yield of homopolymerization of PBA on RSF, $Y(\%)$, are calculated from Eqs. (1) and (2), respectively:

$$W(\%) = \frac{M_{\text{MRSF}} - M_{\text{RSF}}}{M_{\text{RSF}}} \times 100 \quad (1)$$

$$Y(\%) = \frac{M_{\text{MRSF}} - M_{\text{RSF}}}{M_{\text{BA}}} \times 100 \quad (2)$$

Table 1
Individual batch composition.

| Samples | PLA (g) | MRSF | | | |
|---------|---------|---------|--------------|-------|-------|
| | | RSF (g) | RSF:BA (g/g) | W(%) | Y(%) |
| S-1 | 45 | 5 | 100:0 | 0 | 0 |
| S-2 | 45 | 5 | 100:15 | 4.280 | 26.00 |
| S-3 | 45 | 5 | 100:20 | 7.980 | 33.28 |
| S-4 | 45 | 5 | 100:25 | 16.48 | 54.98 |
| S-5 | 45 | 5 | 100:30 | 23.10 | 64.27 |
| S-6 | 45 | 5 | 100:40 | 35.65 | 74.35 |
| S-7 | 45 | 5 | 100:50 | 47.45 | 79.17 |
| S-8 | 45 | 5 | 100:80 | 87.99 | 91.76 |
| S-9 | 45 | 5 | 100:100 | 112.5 | 93.87 |
| S-10 | 45 | 5 | 100:120 | 132.9 | 92.43 |
| S-11 | 45 | 5 | 100:150 | 172.7 | 96.08 |

where M_{RSF} is the original weight of RSF, M_{MRSF} is the weight of the RSF after modification, and M_{BA} is the weight of BA monomer.

2.3. Preparation of composites

The PLA and MRSF were weighted at the desired addition level (Table 1) and premixed in a glass beaker. The mixture was then melt-compounded in an HAAKE rheometer (HAAKE Rheomix 600 P German) at 175 °C for 5 min at a mixing speed of 80 rpm/min. The melted compounds were hot-press molded at 175 °C.

2.4. Characterization

2.4.1. Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) spectroscopy was performed on a Nicolet NEXUS 670 FTIR spectroscopy. The samples ((M)RSF) were dried completely and pressed with potassium bromide (KBr) using disks. The spectra were used to analyzing the change in the chemical structure of surface-treated fiber.

2.4.2. Tensile testing

The tensile test was done by using a tensile test machine (XL-100, Jilin Province Jinli Test Technology Co., Ltd.) adapted to the standard GB/T 1440-92. Specimens about 1 mm in thickness (as shown in Fig. 1) were cut from the plaques for the different measurements realized in the present study. The samples were placed for 48 h at room temperature (\sim 25 °C) and atmospheric conditions (relative humidity of \sim 50 \pm 5%) before test. The tensile rate was 1 mm/min.

2.4.3. Scanning electron microscopy

A field emission scanning electron microscope (FE-SEM, Hitachi High-Technologies CO.S-4300 model, Japan) was used to get SEM images of (M)RSF and PLA/(M)RSF composites. All specimens were fractured perpendicularly to the flow direction after immersion in liquid nitrogen for about 5 min. The fracture surface was sputter coated with gold to provide enhanced conductivity.

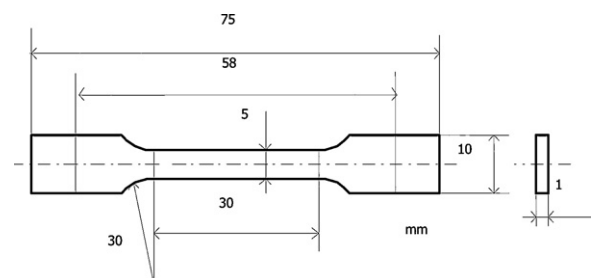


Fig. 1. Shape and measurement of specimens (mm).

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