Biodegradability of Flax Noil Fibers Reinforced with Poly(Lactic Acid) Composites

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Abstract: The composites of poly(lactic acid) (PLA) with untreated or alkali treated (A-) or silane-coupling treated (SC-) flax noil fibers (flax) were prepared by non-woven method and hot pressing technology. The biodegradability of the composites was evaluated by activated soil-burial test. The presence of untreated flax or A-flax or SC-flax led to the acceleration of weight loss due to preferential degradation of flax, which was demonstrated in the SEM micrographs and FTIR spectra. Rates of weight loss decreased in the order flax/PLA (24.0%/35days) >A-flax/PLA (20.6%/35days) >SC-flax/PLA (17.8%/35days) and also decreased with interface shear strengths of the composites. The weight loss of PLA and flax after 35 days was 4% and 52.5% respectively.

Keywords: Biodegradability, poly (lactic acid) (PLA), flax, non-woven method, interface property.

1. Introduction

Natural flax noil fibers offer good opportunities as reinforcement materials for composites due to its advantages, such as renewability, high specific properties, low cost, biodegradability, non-hazardous nature and so on [1-5]. For the purpose of making completely biodegradable green composite, biodegradable polymers has been chosen as matrix. Among all kinds of biodegradable polymers, PLA seem to score well on all the necessary properties: the density is low; degradation behavior, mechanical properties and glass transition temperatures are acceptable and their melt points are almost ideal for producing flax fiber reinforced composites; above all, PLA is not quite expensive and already commercially available [6].

Some researchers have already identified the possibilities for biodegradable composite products by combining PLA with flax fibers [7-10]. In order to improve the interfacial bonding of the composite, S. Goutianos[9] and R. A. Shanks [10] adopted alkali treatment to treat the fibers. Although biodegradability is one of the most important properties of the bio-composites, little was reported on this aspect of the composite.

Cao Y et al [12] performed the biodegradability test in a series of plastic boxes containing characterized soil, which was a 1:1(mass ratio) mixture of red gravel soil and leaf mold for gardening. The pH of the soil was about 7 and each specimen was buried at a depth of 8cm from the surface in the soil. Cao Y et al [12] found that the addition of bagasse fiber to the polycaprolactone composite caused the acceleration of weight loss due the preferential biodegradation of fiber. The weight loss increased with the increase of the fiber content. Wu CS et al. [13] did the biodegradability test in soil environment. Five samples were weighed and then buried in boxes of alluvial-type soil, obtained from farmland topsoil before planting in May 2005. The soil was sifted to remove large clumps and plant debris. The soil was maintained at ~20% moisture in weight and samples were buried at a depth of 15 cm. Wu CS et al. [13] found the biodegradation of the poly(3-hydroxybutyric acid) composite increased with the increase of the wood fiber.

In our previous reports, we have revealed that the mechanical properties of the flax noil fibers (flax) reinforced PLA (flax/PLA) composites are quite promising compared to flax/PP composites, which has been used as automotive interiors recently [7,11]. In this study the alkali and silane-coupling reagents were used so as to improve the interface bonding property of the composites. The objective of this work is to study the effect of fiber content and interface bonding property on the biodegradability of the composites, which will be used to build the biodegradability prediction model in future.

2. Experimental

2.1 Materials

A commercially available PLA fiber is used as the polymeric matrix. The PLA fibers are in length of 38mm, melting point of 168℃. Flax noil fibers are

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used for reinforcement, whose diameter ranges from 15 to 155μm. The average length, diameter and tensile strength of flax are 71.3mm, 63.89μm and 417.57MPa respectively.

2.2 Surface modification

Flax noil fibers are initially treated before mixing with PLA fibers. The fibers are firstly opened by the opener so as to wipe off the impurities, over-length & under-length fibers and improve the orderliness level of the length and fineness of the fibers. Then the fibers are immersed into the 1% alkali solution at room temperature for 1h, followed by washing with distilled water until no sodium hydroxide is left. Subsequently, the fibers are dried at 80℃ for 2hrs followed by drying to constant weight in air.

As far as silane-coupling treatment, the fibers are soaked in 1% $H_2N(CH_2)_3Si(OC_2H_5)_3$ solution for 2hrs, then dried at 80℃ for 1.5hrs followed by drying in air to constant weight.

The flax fibers with no treatment, alkali treatment (A-) and silane-coupling treatment (SC-) are prepared separately before performing the composite.

With the addition of 45 wt% flax the PLA composite shows the best mechanical properties according to our earlier results [11], by which this fraction of flax is selected to perform the composites with surface treatment.

2.3 Composite preparation

The flax and PLA fibers are mixed together by non-woven technology. Firstly, the pulling fibers are opened and scotched by the opener and then the mixture is carded by the carding machine in order to make an even fiber web. Secondly, the fiber web was piled up longitudinally so as to make the preform with the density of 1400g/m^3 and fibers weight fraction of 40, 45, 50, 60% respectively. Thirdly, the preform is molded into composite at a temperature of 190℃, pressure of 12.5MPa and time period of 20min.

2.4 Characterization

Biodegradability of the composites is determined by measuring the weight loss of the thin-plate specimens [12-13]. The specimens are buried in the activated soil (soil) provided by Tianjin Wastewater Treatment Plant. The microorganism content of the soil is 2g/L. According to ISO 14852:1999(E) [14], the solid concentration of the soil is 20g/L. In order to maintain the concentration, the container of the soil is sealed with black plastic bag.

The samples are vertically buried in the soil at a depth of 10cm. The plastic web bags are used to hold the samples in the liquid soil, followed by being picked out from the soil after 5, 10, 15, 20, 25, 30, 35 days, respectively. The specimens are picked out from the bag and washed with distilled water, followed by being dried to a constant weight at 80℃ in an oven.

Digital pictures of the specimens are taken by a digital camera (Canon PC 1192, Canon, Japan).

The morphology of the specimen surface is observed by a scanning electron microscope (SEM) (KYKY 2800, the Research and Development Center of Scientific Instruments of Chinese Academy of Science, China). Prior to observation, the specimens are coated with silver so as to achieve optimal imaging.

The FTIR of the specimens are taken by the Fourier transform infrared spectrometer (Vertor 2.2, Bruker, Germany).

3. Results and discussion

3.1 Composite surface

Figure 1 shows the surfaces of 45flax/PLA (the weight fraction of the flax is 45%) before and after biodegradation of 35 days. It can be seen that there is a significant difference between Figure 1(a) and Figure 1 (b). The 45flax/PLA buried for 35 days could not be fully recovered because of considerable fragmentation. There are lots of cavities on the surface of the composite, from which it can be concluded that biodegradation has occurred on the surface of the composite.

3.2 Effect of fiber content on the biodegradability of composites

Figure 2 shows that the weight loss increases with burial-period for all the samples. It can be concluded that flax has a much higher biodegradability than PLA. The biodegradability of the composites increase with the fiber content, which agrees well with the results reported by Yong Cao [12] and Chin-San Wu [13]. It is marked that the weight loss of 60flax/PLA (42.1%) is about twice as much as 40flax/PLA (20.6%). As shown in Figure 3, the SEM micrographs of 60flax/PLA after 35days burial shows more hollow space and gaps than 40flax/PLA. Based on the phenomena mentioned above we could conclude that the biodegradation of flax is the main reason for the biodegradation of