



Reactive blending of thermoplastic starch, epoxidized natural rubber and chitosan

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ARTICLE INFO

Article history:

Received 22 June 2016

Received in revised form 12 September 2016

Accepted 21 September 2016

Available online 22 September 2016

Keywords:

Thermoplastic starch (TPS)

Epoxidized natural rubber (ENR)

Chitosan (CTS)

Reaction

Epoxy

ABSTRACT

Thermoplastic starch (TPS) was prepared by melt blending cassava starch and glycerol (70/30) at 140 °C. Chitosan (CTS) was incorporated during TPS preparation. The TPS/CTS sample was melt blended with epoxidized natural rubber (ENR) at 140 °C. In the TPS/ENR/CTS blend, adding CTS and ENR improved the tensile strength and elongation at break, respectively. Morphology of the TPS/ENR blend showed the dispersion of large-sized ENR particles in the TPS matrix. Adding CTS reduced the size of the ENR particles. Incorporating CTS also enhanced the melt viscosity of the blend, which suggested a reaction between CTS and ENR. FTIR confirmed that the amino groups of CTS reacted with the epoxy groups of ENR. This reaction between the CTS amino groups with the ENR epoxy groups improved the mechanical properties of the TPS/ENR/CTS blend.

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1. Introduction

Thermoplastic starch (TPS) can be prepared by blending starch with a plasticizer, such as water, glycerol or sorbitol [1]. The plasticizer penetrates into the granules of the starch, interrupting its crystalline structure and inducing the formation of an amorphous structure when subjected to high temperatures and shear forces during the melting process [2]. Therefore, the amorphous starch, following the addition of a plasticizer, behaves like a thermoplastic polymer during the melt stage. Some polymers that have been blended with TPS to improve its mechanical properties include: polypropylene [3], polyethylene [4,5], poly(lactic acid) [6–8] and poly(butylensadipate-co-terephthalate) [9].

Natural rubber (NR) that contains epoxy groups can be modified into epoxidized natural rubber (ENR). Natural rubber can be epoxidized to various degrees: 25%, 50%, and 75% epoxidation is referred to as ENR-25, ENR-50, and ENR-75, respectively. A few studies have reported using ENR to improve the toughness of PLA [10–13]. The improved toughness of PLA by 20% epoxidized natural rubber has been reported [10]. Akbari et al. reported that adding ENR improved the impact toughness of a PLA/talc blend [11]. Wang et al. reported that dicumyl peroxide (DCP) improved the compatibility between PLA and ENR, as well as the impact toughness [13]. Little research exists on the blending of TPS with rubber. Carvalho et al. investigated the morphology of a TPS and natural rubber latex (NRL) blend and found that the phase morphology of the blend depended on the glycerol content [14]. ENR is used in various fields, such as polymer blends [15], polymer modification [16] and polymer composites [17].

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Chitosan (CTS) is a linear polymer of α (1 \rightarrow 4)-linked 2-amino-2-deoxy- β -D-glucopyranose, which is derived from N-deacetylation [18]. Chitosan, a natural polymer, has remarkable properties, including its high mechanical properties, biodegradability, bio-compatibility, antimicrobial activity, and non-toxicity. Chitosan easily reacts with other reactive groups, due to the presence of NH_2 groups. Chitosan's structure has been modified by chemical modification, graft reactions, and ionic interactions. Chitosan is highly hydrophobic, or insoluble in water, but becomes soluble in water with the addition of acids. Because of its physical and chemical properties, chitosan has been used in a range of applications, including pharmaceutical, food, textile, agricultural, biomedical, and cosmetic products [18].

The objective of this research was to develop TPS blending with ENR rubber and CTS. We investigated the effect of adding chitosan to a TPS and ENR blend on its mechanical properties, morphology, rheological properties, and reaction mechanism.

2. Material and methods

Cassava starch (Dragon Fish brand, amylose/amylopectin content 17%/83%, moisture content of 11% total weight and molecular weight of 1.34×10^8 g/mol) was purchased from Tong Chan registered ordinary partnership, Thailand. Glycerol and chitosan (CTS) (deacetylation degree of 85% and molecular weight of 500 kDa) were purchased from Union Science Co., Ltd., Thailand. Epoxidized natural rubber (ENR) with 25% epoxidation was purchased from Muang Mai Guthrie Public Co., Ltd., Thailand. Lactic acid (99%) was produced by Merck (Darmstadt, Germany).

2.1. Sample preparation

Cassava starch was premixed with glycerol (70/30) and distilled water (100 mL/50 g starch) by overhead stirrer at 500 rpm in a water bath at 90 °C. Chitosan is highly hydrophobic, or insoluble in water, but becomes soluble in water with the addition of acids. Chitosan was added during the premixing process along with lactic acid (2% v/v of aqueous lactic acid solution, 100 mL), then melt-blended by a two-roll mill (Pirom-Olarn Co. Ltd., Thailand, PI-140) at 140 °C for 10 min to prepare the TPS and TPS/CTS blend. TPS or TPS/CTS were melt-blended with ENR at 140 °C for 5 min by a brabender internal mixer (Labo Plastomill, Toyoseiki Co. Ltd., Japan). Table 1 shows the composition of TPS, ENR and CTS of the blends. The samples were put in a mold and compressed into sheets for tensile tests and into a film for FTIR by a hot-compress machine with pressure of 500 psi at 140 °C for 3 min.

2.2. Scanning electron microscopy (SEM)

Morphology of the samples was observed by scanning electron microscopy (SEM) (SM-200, Topcon Corp., Japan). The samples were prepared as sheets by compression molding at 140 °C for 3 min. The length, width and thickness were 30 mm, 5 mm and 0.5 mm, respectively. Liquid nitrogen was used to break the samples, and then the fractured surface of the samples was extracted by immersing samples into toluene at 60 °C for 24 h. The extracted surface of the samples was coated with a thin layer of gold, and observed with an acceleration voltage of 10 kV.

2.3. Tensile properties measurements

Tensile properties of the samples were measured using a tensile tester (Tension UTM-II-20; Orientec Co. Ltd., Japan) at 2 mm/min crosshead speed. The bone-shaped samples were prepared as sheets by compression molding at 140 °C for 3 min. The gage length, width and thickness of the sample were 10 mm, 3 mm and 0.5 mm, respectively.

2.4. Contact angle

Water droplet contact angle was observed by drop shape analysis (DSA30E, Krüss Co. Ltd., Germany) to estimate the wettability of samples. The samples were prepared as sheets by hot-compression at 140 °C for 3 min. Water was dropped onto the surface of samples and images were recorded at 1 min, 2 min and 3 min.

Table 1
The composition and codes of blends prepared from thermoplastic starch (TPS), epoxidized natural rubber (ENR) and chitosan CTS.

Sample	Composition (wt%)		
	TPS	ENR	CTS
TPS/ENR	90	10	–
TPS/ENR/CTS1	89	10	1
TPS/ENR/CTS2.5	87.5	10	2.5
TPS/ENR/CTS5	85	10	5

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