



Research Paper

Preparation and characterization of poly(lactic acid) plasticized with vegetable oils and reinforced with sisal fibers



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ABSTRACT

The aim of this work is to prepare a poly(lactic acid) (PLA) based formulation which shows better toughness than neat PLA and maintains similar tensile strength and modulus values. For this purpose, PLA was plasticized with epoxidized and non-epoxidized linseed and soybean oils and the effect of different plasticizers contents as well as the presence of epoxy groups on the mechanical and thermal properties of plasticized PLA systems were evaluated. On the other hand, PLA systems plasticized with 10 wt% of vegetable oil were reinforced with 30 wt% NaOH treated sisal fibers, and the effects of combination of plasticizers and fibers on the degree of crystallinity, thermal and mechanical properties were studied. It must be highlighted that by the combination of epoxidized vegetable oils and fibers, the elongation at break and tensile modulus values can be improved around 70% and 30%, respectively, respect to neat PLA maintaining similar tensile and impact strength values.

1. Introduction

Environmental awareness and scarcity of fossil resources have caused an interest in the development of new “eco” materials being the main features of these materials their natural origin and/or potential biodegradability. So that, the substitution of commodity plastics from petrochemical origin by bio-based polymers, which do not generate harmful wastes at the end of the life cycle, is a challenge for the plastic and polymer composite industries (Meriçer et al., 2016). For example, poly(lactic acid) (PLA) which has been obtained from renewable agricultural raw materials is a commercially available, fully biodegradable polymer and it is one of the most promising biodegradable polymers for industrial plastic applications due to its high mechanical properties and good processability (Lu et al., 2014; Qu et al., 2010). Nevertheless, its elevated price, brittleness, slow crystallization rate and low thermal stability have restricted in some extent the industrial applicability of PLA (Carbonell-Verdu et al., 2017; Dai et al., 2014; Martin and Averous, 2001; Wang et al., 2013; Al-Itry et al., 2012). Therefore, in order to overcome these drawbacks, PLA has been blended with bio-based toughening agents and low cost renewable fillers to produce environment friendly composite materials with superior properties (Dai et al., 2014).

The use of compatible plasticizers for PLA could be a suitable approach for reducing the fragility of PLA since plasticizers lead to decrease the glass transition temperature of PLA resulting in more flexible

materials (Carbonell-Verdu et al., 2017; Balart et al., 2016; Martin and Averous, 2001; Silverajah et al., 2012b; Tee et al., 2016). The growing interest for plasticizers with low migration levels and low toxicity has focused the researchers attention on bio-based plasticizers made from vegetable oils, citrates and sugar derivatives as an alternative to common plasticizers (Choi and Park, 2004; Pedersen et al., 2008). Epoxidized oils are recognized as reactive plasticizers due to the reactivity between epoxy functional groups and the –OH and –COOH groups of PLA polymer (Anakabe et al., 2016; Broström et al. 2004; Dai et al., 2014; Mihai et al., 2010). Epoxidized vegetable oils can be manufactured through the epoxidation of C=C bonds of oils and plasticizers with null volatility and extremely low migration tendency can be obtained (Balart et al., 2016; Dai et al., 2014; Xiong et al., 2013).

Lignocellulosic fibers are favorable materials for many industrial uses due to exhibit low cost, low density, easy processability, excellent strength and high specific modulus (Yusoff et al., 2016; Pickering and Aruan Efendy, 2016). The mechanical properties and reinforcement ability of raw sisal fibers are limited compared to other lignocellulosic fibers, such as flax and hemp. However, sisal fibers used in this work were treated with NaOH and it was observed in previous works (Orue et al., 2016, 2015) that NaOH treatment not only removed non-cellulosic components increasing the cellulose content of fiber, but also created voids inside the fiber structure improving the mechanical interlocking between the NaOH treated sisal fibers and PLA matrix. In addition, all lignocellulosic fibers can accelerate the crystallization

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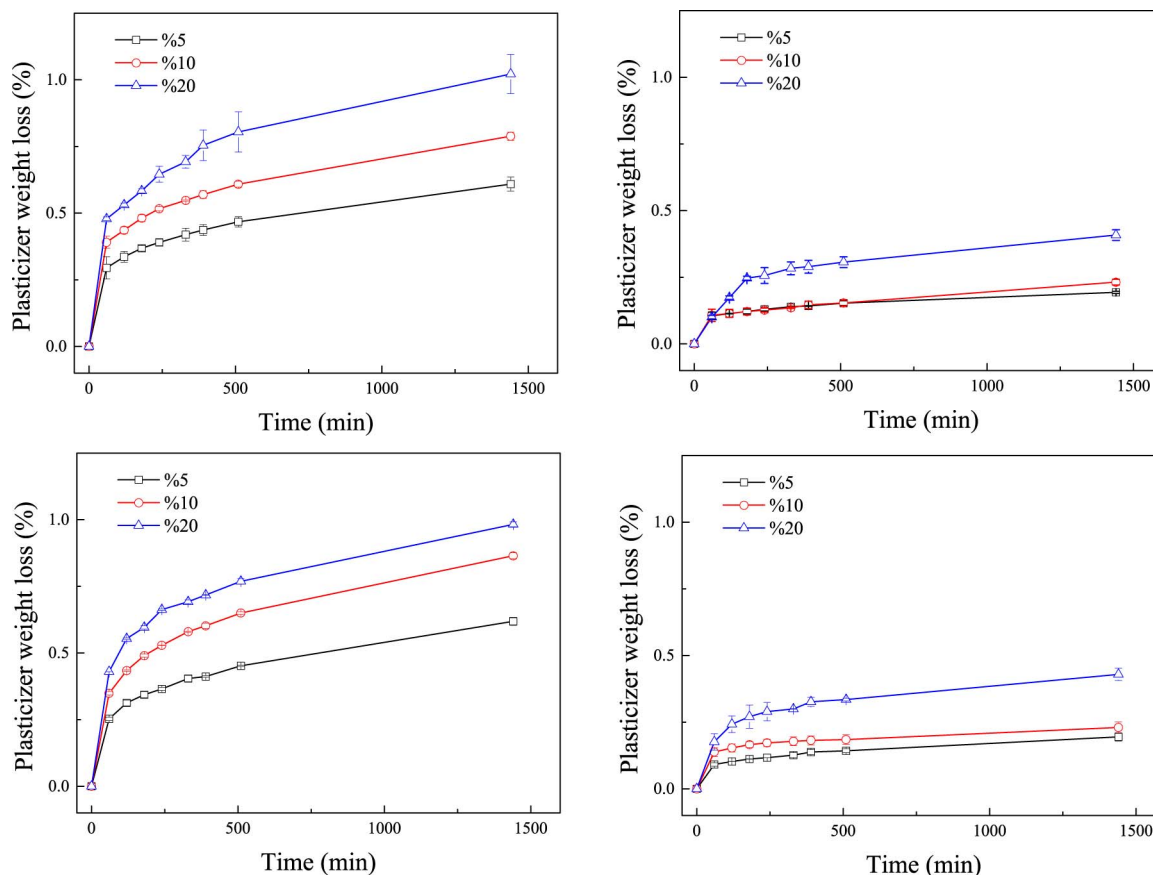


Fig. 1. Plasticizer weight loss of PLA blends plasticized with different amounts of non-epoxidized and epoxidized vegetable oils: (a) PLA/LO, (b) PLA/ELO, (c) PLA/SBO and (d) PLA/ESBO blends.

process of PLA due to the nucleating effect (Sarasini et al., 2013; Tawakkal et al., 2014; Wang et al., 2011).

It was observed in a previous work that tensile strength and modulus values of composites based on 30 wt% NaOH treated fibers were increased respect to neat PLA, but the elongation at break values decreased considerably (Orue et al., 2016). Therefore, the use of compatible plasticizers for PLA could be a suitable approach for reducing the fragility of PLA based composites (Balart et al., 2016).

The main aim of this study is to obtain a PLA based formulation which shows better toughness than neat PLA and maintains similar tensile strength and modulus values. For this purpose, PLA was plasticized with epoxidized and non-epoxidized linseed and soybean oils and the effect of different plasticizer contents as well as the presence of epoxy groups on the mechanical and thermal properties of plasticized PLA systems were evaluated. On the other hand, PLA systems plasticized with 10 wt% of vegetable oil were reinforced with 30 wt% NaOH treated sisal fibers, and the effects of combination of plasticizer and fibers on the degree of crystallinity, thermal and mechanical properties were studied. Ultimately, it must be highlighted that by the combination of epoxidized vegetable oils and NaOH treated sisal fibers, the elongation at break and tensile modulus values can be improved around 70% and 30%, respectively, respect to neat PLA maintaining similar tensile and impact strength values.

2. Experimental part

2.1. Materials and chemicals

PLA (Ingeo™, 2003D) was provided in pellet form by NatureWorks LLC (Minnetonka, USA). According to the supplier, the D-isomer content of PLA was 4% with a melt flow index of 6 g/10 min at 210 °C and

a density of 1.24 g/cm³. Linseed oil (LO) and soybean oil (SBO) as well as epoxidized linseed oil (ELO) and epoxidized soybean oil (ESBO) were kindly supplied by Traquisa S.L. (Barcelona, Spain). According to the supplier, ELO has an epoxidized oxygen content around 8% and iodine value of 5% whereas ESBO presents an epoxidized oxygen content around 7% and iodine value of 3%. ¹³C nuclear magnetic resonance (13C NMR) results of vegetable oils showed that the epoxidation reaction of linseed and soybean oils was totally carried out. Sisal fibers used in this work were kindly supplied by Celulosa de Levante S.A. (Tortosa, Spain) and sodium hydroxide pellets, supplied by Panreac, were used as fiber surface modifier. NaOH treatment of fiber was carried out following the conditions described in a previous work (Orue et al., 2015).

2.2. Compounding and processing of materials

Previous to PLA plasticization with different plasticizer amounts (5, 10 and 20 wt%), it was dried for 2 h at 90 °C to prevent hydrolytic degradation during processing. Firstly, PLA pellets were manually mixed with plasticizers in a beaker and then, mixtures were melt-blended in a HAAKE Rheomix 600 (Thermo Scientific, Karlsruhe, Germany) with two Banbury rotors at 185 °C with a rotor speed of 50 rpm for 5 min. After obtaining all blends, they were cooled-down until room temperature and pelletized through a sieve of 4 mm using the cutting mill SM200 (RETSCH, Hann, Germany). All pelletized samples were kept in an oven at 90 °C for 12 h prior to obtain tensile test specimens (ISO 527-2-5A) in a HAAKE Minijet II (Thermo Scientific, Karlsruhe, Germany) injection machine. The selected injection and mold temperature were 185 °C and 80 °C, respectively, whereas the molding pressure was set up at 650 bar at 8 s.

In order to obtain plasticized composites with 30 wt% of fiber,

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