



Influence of cellulose nanocrystal on strength and properties of low density polyethylene and thermoplastic starch composites



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ABSTRACT

Starch is a renewable and readily available material. Adding starch to low density polyethylene (LDPE) is a sustainable way to reduce the dependency on petroleum based polymers. In this study we investigated the strength and barrier performance of low density polyethylene (LDPE)/thermoplastic starch (TPS) nanocomposites reinforced with cellulose nanocrystals (CNC). In order to assure well dispersion of CNC in nanocomposites, initially CNC was added to TPS and then reinforced TPS were blended in extruder with LDPE at various loading levels. Mechanical properties, glass transition temperature (T_g) and melting point (T_m), moisture absorption and barrier properties of the nanocomposites were studied. All mechanical properties, including tensile strength, modulus of elasticity (MOE) and hardness were considerably improved by CNC. T_g and T_m of the nanocomposites were higher in comparison to CNC free nanocomposites. Water absorption showed a significant decrease as a result of addition of CNC to LDPE/TPS blends. The values of water vapor permeability coefficient (WVP) and water vapor transmission rate (WVTR) were reduced by adding CNC, meaning that CNC considerably improved barrier properties of LDPE/TPS composites. Adding 1% CNC to LDPE/TPS blends was the optimal level of CNC loading leading to the highest improvement in the strength and barrier performance of LDPE/TPS blends and satisfied very well required standard tensile strength for extruded and molded LDPE.

1. Introduction

In order to reduce worldwide environmental pollution caused by non-biodegradable synthetic polymers, development of ecologically safe polymeric materials continues to receive great attention (Shen et al., 2010; Rhim et al., 2013; Heredia-Guerrero et al., 2017). Low density polyethylene (LDPE) as a low-cost polymer with special properties such as good process-ability and high strength, is used extensively in numerous applications (Sabatzadeh et al., 2015). However, LDPE is a non-degradable polymer and increases accumulation of plastic wastes and environmental pollution (Liu et al., 2013; Mortazavi et al., 2014). Blending of conventional synthetic polymers with biodegradable natural polymers is an economic and versatile approach to enhance the biodegradability of petroleum-based polymers, providing partially biodegradable and sustainable plastics with a wide range of desirable properties. Among the biodegradable polymers, starch is a natural, renewable, low-cost and abundant biopolymer that is applied more and more for the preparation of biodegradable blends and composites (Belhassen et al., 2014; Kaushik and Kaur, 2016). In the case of LDPE, the melt blending of LDPE with a biopolymer such as thermoplastic

starch (TPS) could make it partially biodegradable (Psomiadou et al., 1997). It is believed that biodegradation of TPS in the mixture with LDPE creates suitable conditions for the LDPE chains attack by micro-organisms (Dave et al., 1997; Nguyen et al., 2016).

Despite the interesting and promising potential uses of starch to replace conventional polymers, its application faces various drawbacks. The native starch itself cannot be processed with traditional thermo-plastic processing technologies because starch melting temperature (T_m) is generally higher than its decomposition temperature (Liu et al., 2008; Mohammadi Nafchi et al., 2013). This problem can be overcome by addition suitable plasticizers to the native starch (García et al., 2011; Wang et al., 2014; Ivanič et al., 2017). So-called thermostatic starch (TPS) is produced by mixing native starch with plasticizer at a temperature above the gelatinization temperature of starch, typically in the 65–90 °C. The plasticizers reduce the intermolecular forces by increasing the chain mobility and improving the flexibility and extensibility of the biopolymer (Parra et al., 2004; Prachayawarakorn et al., 2010; Rico et al., 2016).

Challenges of TPS based bio-plastics are related to their water sensitivity and poor mechanical properties, which limit their uses in many

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applications (Liu et al., 2009; Akrami et al., 2016). TPS is very hygroscopic and its mechanical properties and dimensional stability are strongly affected by moisture since water is a plasticizer for TPS. In addition, in the presence of moisture, the amorphous TPS tends to reform its hydrogen bonds leading to recrystallization and in turn to material embrittlement (García et al., 2011).

Many attempts have used starch to produce semi-biodegradable materials with superior mechanical properties, mainly by blending with other compounds such as synthetic polymers (Oromiehie et al., 2013; Peres et al., 2016), reinforcing with natural fibers (Ma et al., 2005), cellulose nanofibers (Jonoobi et al., 2010; Babaei et al., 2015; Karimi et al., 2016), nanowhiskers of cellulose (Angles and Dufresne, 2001) and microcrystalline cellulose (Rico et al., 2016). Although, addition of petroleum based polymers to TPS reduces the water sensitivity of TPS and improves mechanical properties of TPS, performance of LDPE polymer and TPS composites is not satisfying. Particularly at high loading of TPS, a drastic reduction in the mechanical properties has been reported, mainly due to poor phase compatibility and adhesion between the hydrophilic starch and the hydrophobic synthetic polymer (St. Pierre et al., 1997; Sabetzadeh et al., 2012). In this regards, addition of different compatibilizers, i.e., maleic anhydridegrafted-polyethylene (MAPE) and vinyltrimethoxy silane (VTMS) could improve interfacial adhesion and consequently the final product properties (Matzinos et al., 2001; Prachayawarakorn and Pomdage, 2014). However, added compatibilizers like VTMS are toxic, and mechanical properties remained still low (Pushpadass et al., 2010).

Reinforcing TPS with nano biomaterials is promising alternative to increase mechanical performance, while preserving the sustainable character of final composites (Arun et al., 2012; Alidadi-Shamsabadi et al., 2015). Cellulose nanocrystal (CNC) which is produced by acidic hydrolysis of amorphous parts of cellulose shows an excellent reinforcing potential for wide range of polymers (Kallel et al., 2016; Zhuo et al., 2017). The presence of the well-dispersed cellulose crystallites in various polymer matrices even at low concentrations enhances mechanical strength performance of final product, especially stiffness (Jonoobi et al., 2010; Kaboorani et al., 2016; Kaboorani et al., 2017). Today, cellulose nanocrystal (CNC) as sustainable, odorless, white, fine, and crystalline dry powder that is widely available in quantities suitable for laboratory research and pilot-scale development (Zhang et al., 2013; Li et al., 2013).

In this study we examined the preparation and characterization of LDPE/TPS based nanocomposites with various TPS content from 30 to 50% (wt.%) and its effects on the nanocomposites properties. In order to overcome the weakness of LDPE/TPS composites, addition of CNC in 1% and 2% to the composites was studied and microstructural, mechanical, barrier properties, moisture absorption and thermal characterizations of LDPE/TPS composites were also investigated.

2. Experimental

2.1. Materials

Glycerol as a plasticizer was purchased from Hansa Company (Germany). Low-density polyethylene (LDPE) with MFI of 4.7 g/10 min (tested at 21.6 N and 190 °C) was obtained from Karajin Co. Ltd. (Karaj, Iran). Cellulose nanocrystal (CNC), kindly provided by Forest Products Laboratory in Madison, WI., USA and was used as the reinforcing material, (in 5.5% suspension). The properties of used CNC has been reported previously by Kaboorani and Riedl (2015). Native corn starch containing 12% moisture, trace amount of native lipid, 0.7% protein, 0.3% ash, and $26 \pm 1\%$ amylose was kindly supplied by Glucosan Co. Karaj, Iran.

2.2. Thermoplastic starch (TPS) preparation

In the first phase and to provide the fine dispersion of CNC in starch matrix, a homogenous suspension of CNC (2% wt.) was prepared by mixing CNC (5.5% suspension) with distilled water and the CNC was dispersed for 20 min at ambient temperature using sonication (SONICA 2200EP S3, Italy). Then, in order to transfer the granular starch into TPS containing CNC, for each formulation a given amount of native corn starch and glycerol (33% wt. of starch) were added to given amount of prepared suspension of CNC. The blends were heated and kept at 80 °C for 30 min while stirring (450 RPM), until starch plastification occurred. The prepared TPS were transformed to LDPE bags and stored overnight. Then, TPS were poured into non-stick plates and dried in an oven under air circulation at 40 °C for 24 h. The resulted TPS containing CNC was palletized to obtain 40–60 mesh pellets.

In the second phase of sample preparation, the palletized TPS containing various amounts of CNC (from 0 to 2 wt.% of final composites) was mixed with a LDPE in a granulated form and melt blended using a twin screw extruder (model: TSE 20; Brabender, Germany). The temperature profile along the six heating zones of the extruder from feeder to die was set at 130–140–145–150–155–160 °C at screw speed of 60 RPM. The extruded materials were emerged in the form of continuous strands through the die. The strands air-cooled and then palletized by a pelletizer. Finally, the pellets were dried at 40 °C for 24 h in an oven to remove the moisture and then cooled to room temperature and transferred to LDPE bags and sealed to avoid moisture adsorption before composites fabrication. TPS and CNC contents in the composites varied from 30 to 50 wt.%, and 0–2 wt.%, respectively (Table 1).

Combination of hand layup and compression molding technique was used for fabrication of composites with thickness of 2 mm from different formulations (Table 1). The pellets were carefully distributed in all locations of the mold, and allowed to pre-melt at 140 °C in a flat mold for 10 min without pressing. This was followed by hot pressing at 140 °C under pressure of 5 MPa for 10 min. Finally, the samples were cooled to room temperature in a cold press under pressure of 2 MPa.

2.3. Characterization

2.3.1. Tensile strength

Specimens of 80 mm × 8 mm × 2 mm dimensions were cut from the compression molded composites for each formulation. At least 10 specimens from each formulation have been made. Measurements of tensile strength were performed according to ASTM 638-03 test method. The tests were performed using an Instron 4486 testing machine equipped with a computerized data acquisition system, in standard conditions of 23 ± 2 °C and $50 \pm 5\%$ relative humidity. Measurements were done at 20 mm/min crosshead speed.

Table 1
Sample codes of the LDPE/TPS and the LDPE/TPS/CNC composites.

Treatment code	LDPE (wt.%)	TPS (wt.%)	CNC (wt.%)
a ₁ -TPS30CNC0	70	30	0
a ₂ -TPS29CNC1	70	29	1
a ₃ -TPS28CNC2	70	28	2
b ₁ -TPS40CNC0	60	40	0
b ₂ -TPS39CNC1	60	39	1
b ₃ -TPS39CNC2	60	38	2
c ₁ -TPS50CNC0	50	50	0
c ₂ -TPS49CNC1	50	49	1
c ₃ -TPS48CNC2	50	48	2

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