



Preparation and characterization of nanocomposite films from oil palm pulp nanocellulose/poly (Vinyl alcohol) by casting method

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ABSTRACT

TEMPO-oxidize nanocellulose (TONC) suspension has been obtained from total chlorine free (TCF) oil palm empty-fruit-bunches (OPEFB) pulp using 4-acetamido-TEMPO (2,2,6,6-tetramethyl piperidin-1-oxyl) mediated oxidation with sodium hypochlorite and sodium bromide in water at 25 °C and pH 10. TONC suspension with varied content from 0.5 to 6% (w/w) reinforced polyvinyl alcohol (PVA) polymer based nanocomposite films were prepared by the casting method. The structural interaction between the TONC and PVA was characterized by the Fourier transform infrared (FT-IR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, X-ray diffraction (XRD) and scanning electron microscopy (SEM). It was found that the 4% (w/w) TONC content reinforced nanocomposite exhibited the highest tensile strength and modulus with an increase of 122% and 291% respectively, compared to PVA while the elongation at break decreased about 42.7%. Thermal stability of PVA based nanocomposite films was improved after incorporation of TONC. Incorporation of TONC in PVA film increases its crystallinity due to strongly linking between the hydroxyl groups of materials however considerable decreases beyond 2 wt% loading are observed. TONC incorporation beyond 2 wt% also reduces the melting temperature peaks and enthalpy of nanocomposite films. FT-IR spectra, NMR and SEM indicate that there is interaction between the TONC and PVA.

1. Introduction

Nanocellulose has been considered a most abundant and inexpensive biopolymer with a wide variety of applications in different areas (Brinchia, Cotana, Fortunati, & Kenny, 2013). The promising applications of nanocellulose have been biodegradability, sustainability, high biocompatibility and most significant for the reinforcement polymeric matrix into nanocomposite materials (Ferraz et al., 2012; Korhonen, Kettunen, Ras, & Ikkala, 2011; Mulligan, 2005; Siqueira, Bras, & Dufresne, 2009). It has vast significance in medical, food, pharmaceutical industries, nanotechnology and also in several materials that are widely used commercially (Khan, Huq, Khan, Riedl, & Lacroix, 2014; Yan et al., 2014). Due to less cost and environmentally safe property, it can be also replace synthetic or petrochemical-based materials in many applications (Brinchi et al., 2013). Earlier, nanocellulose has been isolated from various kinds of cellulosic sources as cotton, wood, plant, animals, bacteria, algae and oil palm biomass

(OPB) (Hassan, Bras, Hassan, Silard, & Mauret, 2014; Wanrosli, Rohaizu, & Ghazali, 2011). At a conservative estimate, for every tons of palm oil produced from a fresh fruit bunch, approximately 1 tons of empty fruit bunch (EFB), 0.7 tons of palm fibers, 0.3 tons of palm kernels and 0.3 tons of palm shells are generated, which amounts to a total palm biomass of 2.3 tons (Chang, 2014). These residues represent an abundant, inexpensive and readily available source of renewable lignocellulosic biomass which can be exploited for use in various applications such as reinforcement in nanocomposites.

Nowadays, nanomaterials are used in packaging industries like food stuff due to degradability and enhance to the quality of new bio-based packaging materials such as edible and biodegradable films from renewable resources (Sorrentino, Gorrasi, & Vittoria, 2007; Tharanathan, 2003). It has potentiality for the preparation of composite films because its biodegradability considered as a promising solution to the environmental pollution of synthetic polymer packaging (Su, Huang, Yuan, Wang, & Li, 2010). Until now biodegradable films has been used

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for packaging extremely limited due to weak barrier and mechanical properties shown by natural polymers. Hence, the natural polymers as cellulose was chemically modified using the TEMPO oxidation and frequently reinforced with a matrix as poly(vinyl alcohol) for their improvement of various properties and applications in further circumstances (Lin, Bruzzese, & Dufresne, 2012; Ng et al., 2015).

Polyvinyl alcohol (PVA) is a semi-crystalline, fully biodegradable, non-toxic, biocompatible and water soluble synthetic polymer which is mainly composed of C–C and has excellent composite film casting property for packaging materials (Chiellini, Cinelli, Imam, & Mao, 2001; Zhang, Burgar, Lourbakos, & Beh, 2004). It has potential ability to interact with the hydrophilic surfaces of the biomaterials due to the strong hydrogen bonding and established in suitable combinations with cellulose or nanocellulose for produce green nanocomposites (Lee et al., 2012). It is incorporated into nanocellulose finds various significant applications in the field of pharmaceuticals, medicine and packaging (Rahman, Afrin, & Haque, 2014).

In the present work, the nanocellulose crystals (TONC) were isolated using the TEMPO-oxidized process from OPEFB cellulose, which was prepared via a chlorine-free bleaching sequence (Leh, Rosli, Zainuddin, & Tanaka, 2008; Rosli, Leh, Zainuddin, & Tanaka, 2003). Usually, the C6 hydroxyls groups oxidized into carboxylate groups by the TEMPO treatment is shown in Fig. 1 (Isogai, Saito, & Fukuzumi, 2011). The TONC/PVA composite films were prepared by mixing TONC and PVA in distill water with various content ratios and subsequently cast in petri dishes, followed by drying of the mixtures. The structural, physical-chemical and mechanical properties were characterized and evaluated by scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM), Fourier transform infrared (FTIR) spectroscopy, nuclear magnetic resonance (^{13}C NMR), x-ray diffraction analysis (XRD), differential scanning calorimetry (DSC) and tensile testing.

2. Experimental

2.1. Materials

Oil palm empty fruit bunches (OPEFB) were obtained for use in this study from a local palm oil mill in Perak, Malaysia. Before pulping, the OPEFB was cut to approximately 2 inches long, which was then soaked and washed with distilled water to remove dirt and unwanted materials and air dried to average moisture content of 12.5% and stored in polyethylene bags. The chemicals used for pulping, bleaching, oxidation and composites such as sodium hydroxide (NaOH), magnesium sulfate (MgSO_4), hydrogen peroxide (H_2O_2), hydrochloric acid (HCl), 4-acetamido-TEMPO (2,2,6,6-tetramethylpiperidin-1-oxyl) 99.8%,

sodium hypochlorite (NaClO), sodium chlorite (NaClO_2) 99.8%, sodium bromide (NaBr , 99.0%), acetic acid (CH_3COOH) 99.8%, ethanol, and polyvinyl alcohol (98%) were obtained from E. Merck (Germany).

2.2. Methods

2.2.1. Preparation of OPEFB-TCF cellulose

OPEFB-TCF cellulose was prepared using the environmentally benign process as described by Wanrosli et al. (Wanrosli, Zainuddin, & Lee, 2004). Basically, it involves water pre-hydrolysis of the OPEFB fibers, followed by soda-anthraquinone pulping. The unbleached pulp was then bleached using a TCF sequence of oxygen (O), ozone (Z) and peroxide (P).

2.2.2. Preparation of TEMPO-oxidized nanocellulose (TONC)

OPEFB-TCF cellulose (10 g) was suspended in 1 L of distill water in a beaker, containing TEMPO (0.230 g), sodium bromide (0.5 g), and stirred for 5 min. The reaction was initiated by setting the ultrasonic water bath (model Branson 8510) at a frequency of 40 kHz and 320 W power output with circulating cooling water used to maintain the temperature at 25 °C. Additions of 60 mL sodium hypochlorite solution in the reaction mixture drop by drop, during the 1 h and continue stirring at 500 rpm for 4 h with controlled pH at 10 ± 0.2 by the addition of 0.5 M NaOH/HCl. The stirring was stopped and 100 mL ethanol was added to it. The oxidized slurry was neutralized by adding 0.5 M HCl, vacuum-filtered and thoroughly washed with distill water. The pulp was suspended in 500 mL of distill water in a glass bottle, 40 mL of sodium chlorite (34 g/L) and acetic acid (5 M) solutions added to it. The solution was kept in a water bath at 70 °C for 2 h, cooled at room temperature and washed with distill water. TEMPO-oxidized pulp was transferred in a beaker and sonicated (7 microtip limit) with 15 s interpose/min on ice bath and centrifuged at 3500 rpm for 1 h to separate the supernatant containing TONC suspension from the unfibrillated and partially fibrillated fractions.

2.2.3. Preparation of TONC/PVA nanocomposite films

The solution of PVA (10 wt%) was prepared in distilled water and poured into a round bottom flask equipped with a condenser for 1 h stirring at 90 °C. TONC suspension was added to it in various concentrations 0.5, 1, 2, 4 and 6% (w/w) respectively. The mixtures were further stirred for 2 h and keep at room temperature for 0.5 h. The final suspensions were cast in polypropylene petri dishes and placed in a vacuum oven at the 50 °C for 48 h. The resulting films thus obtained were kept in the desiccators until the characterization.

2.3. Characterization techniques

2.3.1. Tensile properties

Tensile properties (tensile strength, Young's modulus and elongation at break) were performed on a TA-XT Flux Texture Analyzer. The tensile deformation was determined at a crosshead speed of 10 mm/min. The dimensions of the films according to the standard test method EN 150 527-2 , 1996 was as follows: length 60 mm, width 30 mm, thickness 0.04 mm and load cell 30 kg. The measurements were performed at room temperature and calculated over five test samples, expressing the results as the mean value.

2.3.2. Differential scanning calorimetry (DSC)

DSC analysis was performed with a DSC-60 Instrument (Shimadzu D 60). Approximately 10 mg sample of the films was heated from 30 °C to 400 °C at a heating rate of

10 °C/min under nitrogen atmosphere. The DSC curves were used to determine the glass transition temperature, melting temperature and melting enthalpy. The degree of crystallinity (X_c) was calculated by using the following Eq. (1) (Frone, Berlioz, Chailan, & Panaitescu, 2013).

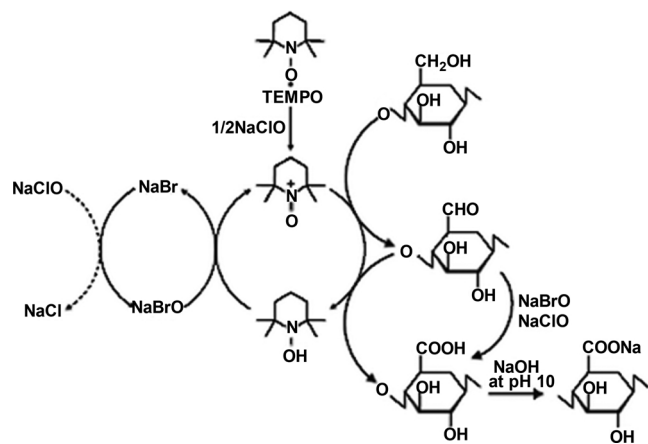


Fig. 1. Regioselective oxidation of cellulose by TEMPO-mediated oxidation (Isogai et al., 2011).

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