



Bionanocomposites of regenerated cellulose/zeolite prepared using environmentally benign ionic liquid solvent



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ABSTRACT

Bionanocomposite films based on regenerated cellulose (RC) and incorporated with zeolite at different concentrations were fabricated by dissolving cellulose in 1-ethyl-3-methylimidazolium chloride (EMIMCl) ionic liquid using a simple green method. The interactions between the zeolite and the cellulose matrix were confirmed by Fourier transform infrared spectra. Mechanical properties of the nanocomposite films significantly improved as compared with the pure regenerated cellulose film, without the loss of extensibility. Zeolite incorporation enhanced the thermal stability and char yield of the nanocomposites. The scanning electron microscopy and transmission electron microscopy showed that zeolite was uniformly dispersed in the regenerated cellulose matrix. In vitro cytotoxicity test demonstrated that both RC and RC/zeolite nanocomposite films are cytocompatible. These results indicate that the prepared nanocomposites have potential applications in biodegradable packaging, membranes and biomedical areas.

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1. Introduction

Cellulose, the most abundant and renewable resource in nature, is obtained from wood pulp and cotton and is considered a renewable chemical resource that can replace petroleum-based materials (Ma, Zhou, Li, Li, & Ou, 2011; Soheilmoghaddam, Wahit, & Ibrahim Akos, 2013; Soheilmoghaddam, Wahit, Mahmoudian, & Hanid, 2013). Cellulose a linear polysaccharide composed of β -1-4-linked D-glucopyranose repeating units, has drawn much attention due to its unique properties such as high mechanical strength, chemical stability, biodegradability and multitude of chemical derivations (Heinze & Liebert, 2001; Klemm, Heublein, Fink, & Bohn, 2005). However, on account of the extensive large network connecting hydrogen bonds and partially crystal structure, cellulose cannot be dissolved in water or most conventional organic solvents (Cai & Zhang, 2005; Song, Luo, Wang, Hao, & Gao, 2013). Therefore, several solvent systems such as N-methyl morpholine N-oxide (NMMO) (Fink, Weigel, Purz, & Ganster, 2001), lithium chloride/1,3-dimethyl-2-imidazolidinone (LiCl/DMI) (Takaragi, Minoda, Miyamoto, Liu, & Zhang, 1999),

lithium chloride/N,N-dimethylacetamide (LiCl/DMAc) (McCormick & Callais, 1987) and phosphoric acid (Northolt et al., 2001) have been developed for the preparation of regenerated cellulose materials. Most of the systems developed thus far are still seen as unsuccessful in the eyes of industry and environmentalist because of their toxicity, difficult of solvent recovery and several adverse side reactions (Soheilmoghaddam, Wahit, & Ibrahim Akos, 2013; Soheilmoghaddam, Wahit, Mahmoudian, et al., 2013; Song et al., 2013).

Recently, a green solvent system known as ionic liquid (IL) has been gaining interest because of its potential to regenerate and chemically modify the cellulose (Swatloski, Spear, Holbrey, & Rogers, 2002). Ionic liquids are characterized by excellent solubility, low toxicity, thermal stability, near zero volatility and recyclability (Hameed, Guo, Tay, & Kazarian, 2011). The desirable properties of ILs can also be tailored since they offer great flexibility in the design of cationic and anionic structures and as such they are termed the “designers solvent” (Azubuike, Rodríguez, Okhamafe, & Rogers, 2012). Literature has shown a variety of ionic liquids (ILs) to be good cellulose and biomass non-derivating solvents (Casas et al., 2012; Fort et al., 2007; Jiang et al., 2011; Pu, Jiang, & Ragauskas, 2007). The most promising anions for cellulose dissolution are acetate and chloride, whereas different cations can be employed (Casas et al., 2012). The most common IL for cellulose dissolution

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is 1-ethyl-3-methylimidazolium chloride ([EMIM]⁺[Cl]⁻), producing a cellulose solution without derivation (Lan, Liu, Yue, Sun, & Kennedy, 2011; Swatloski et al., 2002).

Recently, organic–inorganic hybrid cellulose nanocomposites have attracted much attention due to their outstanding mechanical, optical, electrical and flame retardancy properties which are widely used in the fields of biomedicine, food packaging and water treatment (Han, Yan, Chen, Li, & Bangal, 2011). In the few past decades, polymer/zeolite nanocomposites have received a lot of attention as they perform better as compared to pure polymers. Zeolites are naturally occurring microporous framework silicates with a three-dimensional cage structure. They possess permanent negative charges in their structural framework which is balanced by exchangeable cations (Wang & Peng, 2010), thus they are widely used in adsorption, ion-exchange, catalysis and separation (Tosheva & Valtchev, 2005; Yu, Gong, Zeng, & Zhang, 2013). Zeolites have high mechanical strength, good thermal and chemical stability and nanocomposites reinforced with zeolites can be used over a wide range of operating conditions (Kittur, Kulkarni, Aralaguppi, & Kariduraganavar, 2005). Zeolites are also found to exhibit bioactivity and biocompatibility, thus they can be used as antibacterial materials, antitumor, anti-thrombotic agents, hemostatic, drug carriers and bone regenerants (Fatouros et al., 2011; McDonnell, Beving, Wang, Chen, & Yan, 2005; Wheatley et al., 2005). It is expected that the incorporation of zeolites into regenerated cellulose matrix may enhance the mechanical and thermal properties of the regenerated cellulose and also improve its biocompatibility.

In this work, regenerated cellulose/zeolite nanocomposite films were prepared via solution casting method. Ionic liquid was used as the key component medium for the dispersion of zeolite nanofiller and cellulose dissolution. The morphological, physical and mechanical properties of the bionanocomposite film were investigated in detail. Finally, cell viability assay was applied to understand the effect of zeolite on cellulose biocompatibility.

2. Experimental

2.1. Materials

Microcrystalline cellulose, a commercial reagent from Sigma with an average powder size of 50 μm, Avicel type with the degree of polymerization of 350 was used. The ionic liquid, 1-ethyl-3-methylimidazolium chloride (EMIMCl) with ≥95% purity. Zeolite (ZSM-5) with an average particle size of 300 nm and BET surface area of >362 m²/g was purchased from advanced chemical supplier (ACS, USA).

2.2. Preparation of bionanocomposite films

The zeolite was dispersed in EMIMCl for 90 min using a sonicator (Model FB15053, 600 W maximum power, Fisher Scientific Co., Germany) before the addition of cellulose powder. In order to dissolve the cellulose in EMIMCl ionic liquid, the mixture was heated at 90 °C for 24 h with constant stirring. The ratio of EMIMCl/cellulose was 94/6 (wt.%). After degassing the solution in a vacuum oven, it was casted on a glass plate and immersed immediately in a distilled water bath at room temperature for 24 h to remove EMIMCl from the films. The obtained nanocomposite films were washed with water and dried in a vacuum oven at 40 °C for 2 h. All the dried films were stored in a moisture controlled desiccator for further testing. The RC/zeolite nanocomposites films prepared with different zeolite content (2, 4, 6 and 8 wt.% with respect to cellulose content) were coded as RC/Z2, RC/Z4, RC/Z6 and RC/Z8.

The pure RC film was prepared with the same procedures as above without zeolite loading.

2.3. Characterization

2.3.1. UV–vis spectroscopy (UV–vis)

The optical transmittance (T_r) of the pure RC and RC/zeolite bionanocomposite films were measured with a UV–vis spectroscope (Shimadzu UV-3101PC, Japan) at wave lengths ranging from 200 to 800 nm.

2.3.2. Fourier transformed infrared spectroscopy (FTIR) measurement

The FTIR spectra were recorded on an attenuated total reflection Fourier transform infrared (ATR-FTIR) instrument (Nicolet Nexus 670, Thermo Fisher, USA) in the 157 range of 600–4000 cm⁻¹ with a resolution of 4 cm⁻¹.

2.3.3. X-ray diffractometry (XRD)

X-ray diffraction (XRD) patterns were obtained using XRD diffractometer (Rigaku Miniflex II). Patterns with Cu K α radiation ($\lambda = 0.15406$ nm) at 40 kV and 30 mA were recorded in the region of 2θ from 3 to 60.

2.3.4. Field emission scanning electron microscopy (FESEM)

The morphology of RC and RC/zeolite nanocomposite films was investigated by FESEM using a JEOL JSM-6701F SEM machine operating at an acceleration voltage of 5 kV. The micrographs were taken from the surface of cryofractured samples previously coated with gold. The elemental composition of RC and RC/zeolite nanocomposites was determined by energy dispersive X-ray (EDX) analysis (JEOL JSM-6701F SEM) coupled with FESEM.

2.3.5. Transmission electronic microscopy (TEM)

Ultrathin slices with the thickness of approximately 70–90 nm were obtained from nanocomposite film imbedded into an epoxy resin by ultra microtome (Power Tome XL) under cryogenic condition, and then placed into a carbon-coated copper grid. The dispersion state of zeolite within RC was investigated using Philips CM12 at an accelerating voltage of 200 kV.

2.3.6. Mechanical properties

Tensile properties were measured on an LRX Tensile Testing Machine (Lloyd, USA) according to ASTM D882-10 at a crosshead speed of 10 mm/min at room temperature. A gauge length of 30 mm was used. The specimens were cut into strips of 60 mm × 13 mm × 0.03 mm. The experiment was repeated five times and average values were taken.

2.3.7. Contact angle

Contact angles of water droplets with a volume of 2 μL on the films were measured at 23 °C and 50% RH using Face Water contact angle metre, Model CA-DT.A, Brand Kyowa.

2.3.8. Moisture uptake

Samples with dimensions (60 mm × 13 mm × 0.03 mm) were dried until constant weight in an oven at 85 °C for 24 h to remove moisture before the moisture absorption test. These samples were weighed immediately after removal from the oven. The samples were kept in a 75% constant relative humidity environment generated in a hermetic glass container with aqueous saturated NaCl solutions (ASTM E 104-02) at 25 °C. The moisture uptake (%MU) was calculated using Eq. (1):

$$\%MU = \frac{w_t - w_0}{w_0} \times 100 \quad (1)$$

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