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Regenerated cellulose/epoxidized natural rubber blend film

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ABSTRACT

Regenerated cellulose/epoxidized natural rubber (ENR-50) blended films were prepared using an environmentally friendly ionic liquid, 1-butyl-3-methylimidazolium chloride (BMIMCl). The films were obtained by casting solution method. The hydrogen bonding interactions between epoxy groups in ENR with hydroxyl groups of the RC in the blends were investigated by Fourier transforms infrared (FTIR) spectroscopy. Field emission scanning electron microscopy (FESEM) revealed that ENR dispersed homogeneously within the RC matrix. The blends show significant enhancement of thermal stability as compared to the regenerated. The elongation at break remarkably improved by about 39% at 20 wt.% loading of ENR in dry state. This work demonstrates an effective approach to processing biodegradable regenerated cellulose and epoxidized natural rubber blend.

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1. Introduction

In recent years, bio-based materials have attracted much attention due to the expansion in environmental pollution caused by non-renewable resources [1]. Cellulose is the most abundant natural polymer with very attractive properties such as high mechanical strength, chemical stability and biodegradability [2,3]. However, cellulose has some drawbacks. It is a very brittle and displays very low elongation even under dry conditions, which limit its applications [4]. Blending solves this problem by offering a simple and relatively cheap way to develop novel materials with a number of valuable properties [5]. Epoxidized natural rubber (ENR) obtained by the modification of natural rubber displays multiple functions such as good wet grip, excellent flexibility and high damping characteristics [6]. In addition, the unique structure of ENR allows good compatibility with polar polymers [7]. There is the possibility of hydrogen bonding between the complementary binding sites of regenerated cellulose and ENR and this will affect the blend properties. In this work, the effect of different ENR contents on mechanical, thermal and morphological properties of RC have been studied. This work provides an effective way to obtain RC/ENR blend films using the green ionic liquid solvent, thereby broadening the application of cellulose in the field of packaging, membrane and biomaterials.

2. Experimental

Materials: Microcrystalline cellulose, with average powder size of 50 μm was supplied by Sigma, Germany. ENR (50% epoxy

degree) was supplied by Malaysian Rubber Board. Other reagents supplied by sigma, Germany were used as received.

Films preparation: A pre-weighed amount of cellulose was added to BMIMCl, heated to 90 °C and mechanically stirred for 3 h before ENR addition. The concentration of cellulose in BMIMCl solution was 6 wt%. ENR was dissolved in THF and mechanically stirred at 40 °C until the ENR was uniformly dissolved in the solvent. ENR/THF solution then mixed with cellulose/ionic liquid solution and stirred for 24 h at 85 °C. After THF extraction, the homogenous viscous solution was casted on a plain glass plate and immersed in distilled water to remove BMIMCl from the blends. The prepared films were washed several times with deionised water and dried at 80 °C for 3 h. Blends samples with compositions of 90/10, 80/20, 70/30 and 60/40 cellulose/ENR in wt% were prepared and designated as RC/ENR-1, RC/ENR-2, RC/ENR-3 and RC/ENR-4 respectively.

Characterization: FTIR spectra were recorded by a Perkin-Elmer 180 with resolution of 4 cm⁻¹ for 32 scans over a wave number range of 4000–400 cm⁻¹. X-ray diffraction measurements were performed on XRD diffractometer (Rigaku Miniflex II) in the 2θ range of 3–70 using Cu Kα radiation. The crystallinity χ_c (%) of cellulose blend films were obtained from the X-ray empirical method proposed by Segal et al. [8].

$$\chi_c (\%) = \frac{I_{Max} - I_{Am}}{I_{Max}} \times 100 \quad (1)$$

where I_{Max} is the intensity at (200) peak, and I_{Am} is the intensity at the minimum between (110) and (200) peak.

FESEM was performed on the cryo-fractured samples were frozen in liquid nitrogen using a JEOL JSM-6701F. The fractured surface of the films was coated with gold prior to analysis. Thermal analysis of the nanocomposite films was measured using Perkin-Elmer TGA 7 (Perkin-Elmer Instruments, USA). The temperature

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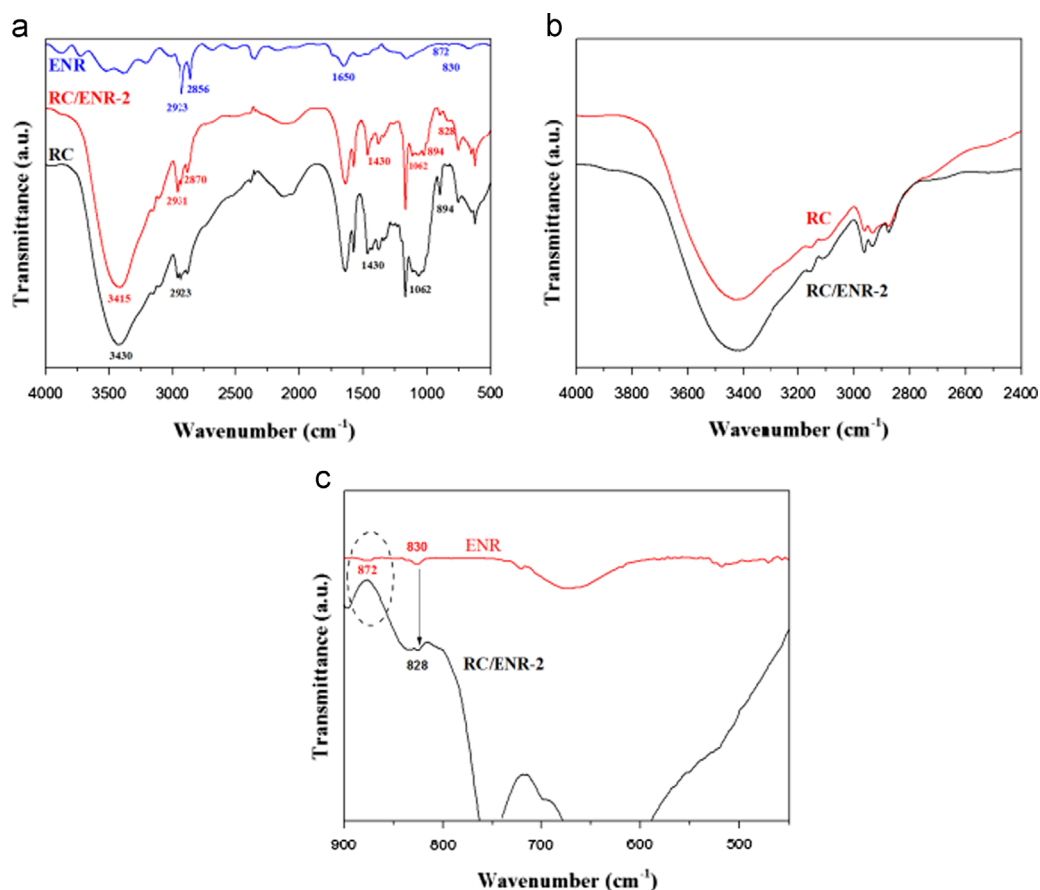


Fig.1. FTIR spectra of (a) ENR, RC and RC/ENR with 20 wt.% ENR content, (b) RC and RC/ENR-2 ENR and (c) ENR and RC/ENR-2.

ranged from 30 to 700 °C with a heating rate of 10 °C min⁻¹ under nitrogen. The tensile properties of the films with dimensions of 60 × 13 × 0.03 mm³ in dry and wet (soaked in water for 1 h) states were measured on LRX Tensile Testing Machine (Lloyd, USA) at a crosshead speed of 10 mm/min at room temperature.

3. Results and discussion

Fourier transforms infrared (FTIR): Fig. 1 depicts the FTIR spectra of RC, ENR and RC/ENR-2 blends. In the case of ENR, the absorptions at 2923 cm⁻¹ and 2856 cm⁻¹ are assigned to asymmetric and symmetric stretching vibration of methyl (-CH₃) and methylene (-CH₂) group respectively. The band at 1650 cm⁻¹ is due to C=C stretching. The characteristic peaks at 872 cm⁻¹ and 830 cm⁻¹ are assigned to the asymmetric and symmetric vibration of the epoxy ring [9]. The FTIR spectrum of RC and RC/ENR-2 shows broad band characteristic at 3000–3600 cm⁻¹. This is attributed to the O–H stretch in cellulose structure. The peaks at 1430 cm⁻¹ and 2923 cm⁻¹ are assigned to CH₂ symmetric bending and C–H stretching vibration respectively. The C–O stretching vibration and alcoholic group of C–OH peaks are situated at 894 cm⁻¹ and 1062 cm⁻¹ respectively [10]. Observations have shown that the characteristic peaks of the blends consist of ENR and RC characteristic peaks. However, the peak positions between 3600 and 3000 cm⁻¹ and 830 cm⁻¹ band intensities for RC/ENR-2 nanocomposite films shifted to lower wave numbers as compared to the RC film peak. The ENR peak at 872 cm⁻¹ disappeared as shown in (Fig. 1(a)–(b)). This confirms the reaction between epoxy groups in ENR with hydroxyl groups of the RC.

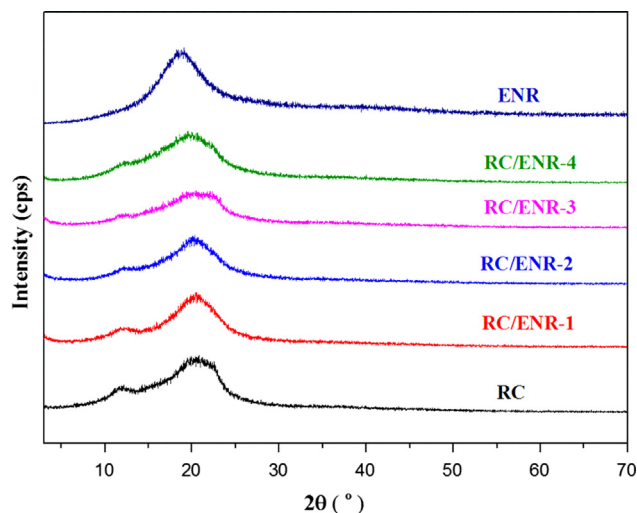


Fig.2. XRD patterns of RC/ENR blends.

X-ray diffraction (XRD): Fig. 2 shows the XRD spectra of RC, ENR and RC/ENR blends. Pure RC diffractogram shows two characteristic peaks at $2\theta = 11.89^\circ$ assigned to $1\bar{1}0$ plane and $2\theta = 20.99^\circ$ assigned to (110)/(020) lattice planes of cellulose II crystalline structure. This demonstrated that the cellulose solution was regenerated completely in the water coagulant to form regenerated cellulose films [11]. Although RC is blended with ENR, the diffraction peaks of the blend films are very similar to that of pure RC. However, the XRD diffraction intensity of blends at $2\theta = 12.2^\circ$ reduced as compared to that of the pure RC film thereby signifying

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