



Processing and properties of eco-friendly bio-nanocomposite films filled with cellulose nanocrystals from sugarcane bagasse



Mounir El Achaby^{a,*}, Nassima El Miri^b, Adil Aboulkas^c, Mohamed Zahouily^b, Essaid Bilal^d, Abdellatif Barakat^e, Abderrahim Solhy^a

^a Center for Advanced Materials, Mohammed VI Polytechnic University, Lot 660-Hay Moulay Rachid, 43150 Benguerir, Morocco

^b Moroccan Foundation for Advanced Science Innovation and Research (MAScIR), Rabat Design, Rue Mohamed El Jazouli, Madinat El Irfane 10100-Rabat, Morocco

^c Laboratoire Interdisciplinaire de Recherche en Sciences et Techniques, Faculté polydisciplinaire de Béni-Mellal, Université Sultan Moulay Slimane, BP 592, 23000 Béni-Mellal, Morocco

^d R&D OCP, OCP Group, Complexe industriel Jorf Lasfar, BP 118 El Jadida, Morocco

^e IATE, CIRAD, Montpellier SupAgro, INRA, Université de Montpellier, 34060, Montpellier, France

ARTICLE INFO

Article history:

Received 5 September 2016

Received in revised form

21 November 2016

Accepted 7 December 2016

Available online 14 December 2016

Keywords:

Cellulose nanocrystals

Polymer blend

Bio-nanocomposite films

ABSTRACT

Novel synthesis strategy of eco-friendly bio-nanocomposite films have been exploited using cellulose nanocrystals (CNC) and polyvinyl alcohol/carboxymethyl cellulose (PVA/CMC) blend matrix as a potential in food packaging application. The CNC were extracted from sugarcane bagasse using sulfuric acid hydrolysis, and they were successfully characterized regarding their morphology, size, crystallinity and thermal stability. Thereafter, PVA/CMC-CNC bio-nanocomposite films, at various CNC contents (0.5–10 wt%), were fabricated by the solvent casting method, and their properties were investigated. It was found that the addition of 5 wt% CNC within a PVA/CMC increased the tensile modulus and strength by 141% and 83% respectively, and the water vapor permeability was reduced by 87%. Additionally, the bio-nanocomposites maintained the same transparency level of the PVA/CMC blend film (transmittance of ~90% in the visible region), suggesting that the CNC were dispersed at the nanoscale. In these bio-nanocomposites, the adhesion properties and the large number of functional groups that are present in the CNC's surface and the macromolecular chains of the PVA/CMC blend are exploited to improve the interfacial interactions between the CNC and the blend. Consequently, these eco-friendly structured bio-nanocomposites with superior properties are expected to be useful in food packaging applications.

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1. Introduction

Recently, significant research has been devoted to the use of biopolymers owing to their advantages such as biodegradability, non-toxicity, availability, low cost, and potential wide range of applications, especially in the field of biomaterials and packaging applications [1,2]. Although biopolymers are considered potential replacements for conventional plastic materials, some of their properties must be improved to position them as materials that can be competitive with fossil derivatives, especially their poor mechanical, thermal, and barrier properties [3]. However, the blending of biopolymers and/or adding of nanofillers represents

an effective way to improve the properties of biopolymers and, therefore, broadens their fields of application [1,4].

Blending of highly compatible biopolymers represent an effective way to develop new innovative biomaterials [5]. Due to the presence of strong intermolecular interactions, films produced from the blending of biopolymers usually exhibit improved properties as compared to films made from an individual biopolymer [6,7]. On the other hand, the processing of bio-nanocomposite materials with low loading of nanofillers has already been proven as an effective strategy to produce new nanostructured materials with innovative properties and high performances for packaging applications [1,4,8,9]. The incorporation of well-dispersed nanofillers into a polymeric matrix may result in improved physico-chemical properties of the resulting bio-nanocomposites, especially the mechanical, optical, thermal, and barrier properties [3,10–12].

Polyvinyl alcohol (PVA) and carboxymethyl cellulose (CMC) biopolymers have been widely used for development of biodegrad-

* Corresponding author.

E-mail address: mounir.elachaby@um6p.ma (M. El Achaby).

able films for packaging applications [6,9]. The PVA and CMC are miscible and compatible biopolymers, due to the presence of multifunctional groups on their macromolecular chains [13–16]. Consequently, the blending of these polymers can enable the preparation of biodegradable materials with properties that allow their application in the emerging field of bio-packaging [17,18]. More importantly, the PVA/CMC blend can be used as a new biocompatible and homogeneous blend matrix for bio-nanocomposite development to achieve high-performance packaging materials. Recently, elongated cellulose nanocrystals (CNC) for bio-nanocomposite development have attracted attention in the field of nanotechnology. These nanoparticles, which can be sphere-like, rod-like, ribbon-like, or needle-like, have a length ranging from 100 nm up to 1–2 μm , and a diameter of about 5–20 nm [3]. The main features that stimulate the use of CNC as polymer reinforcement agents are its large specific surface area (estimated to be several hundreds of square meters per gram) and its very high modulus of elasticity (approximately 150 GPa) [19,20]. Other attractive advantages of CNC are their low density (about 1.566 g cm⁻³), biocompatibility, and biodegradability [21]. Additionally, CNC can be extracted from abundant and renewable natural sources [22]. It has been widely demonstrated that the incorporation of CNC into biopolymers can result in bio-nanocomposite materials with a good appearance and high mechanical, thermal, and barrier properties [3,19,23–25], which are the main properties required for packaging applications [1,26].

The solvent casting method is frequently used for the processing of biopolymer blend and bio-nanocomposite films for food, pharmaceutical, and cosmetic packaging applications [27]. The interfacial adhesion and the condition of mixing during fabrication of biopolymer-based films, *via* the solvent casting method, strongly affect the network structure, physical properties, and film performance. Also, in the film casting process, the final film quality is strongly related to the rheological behavior of the film-forming solutions (FFS). Therefore, knowledge of the FFS rheological properties is important for the design and processing of films by casting [28–30]. The rheological properties of FFS based on biopolymer blends and bio-nanocomposites can affect their spreadability during casting process and the thickness, uniformity and performances of resulting cast films [30]. Additionally, the formation of 3D-interconnected network *via* strong interfacial interactions between nanofillers and macromolecular chains of polymers can be also deduced from the rheological properties [24]. The mechanical properties of CNC-reinforced biopolymer bio-nanocomposite cast films are often reported in the literature, but the FFS state and their rheological properties prior to the casting have received only limited attention.

Owing to their wide availability and good biodegradability as well as their good film-forming ability, films prepared from neat PVA or CMC have relatively poor mechanical and high water vapor permeability properties, which can limit their use as food packaging materials. Nevertheless, these drawbacks can be overcome by blending these biopolymers in a new PVA/CMC material, that can play the role of a new polymeric matrix for manufacturing bio-nanocomposites by using nanostructured fillers (such as CNC), without compromising the transparency and biodegradability of the original biopolymers. The aim of this work is to use the PVA/CMC blend as new biopolymeric matrix for developing bio-nanocomposite films, with improved water vapor permeability (WVP) and mechanical properties, using CNC isolated from sugarcane bagasse as nanoreinforcing agents. Previous experimental results showed that the composition 50/50 of CMC/PVA exhibits an enhanced properties [14–16]. This indicates the compatibility between PVA and CMC at this ratio. Such compatibility arises due to the presence of the carboxymethyl groups bonded to some of the hydroxyl groups on the cellulose in CMC and the hydroxyl groups

in PVA capable of hydrogen bonding. In addition, the presence of hydroxyl groups on the surface of CNC ensures their compatibility with the PVA/CMC biopolymer blend. This compatibility between the CNC and the PVA/CMC blend can cause noncovalent interactions, therefore driving the high reinforcement effect of CNC in resulting PVA/CMC-CNC bio-nanocomposites. The rheological properties of FFS and the structural, transparency, thermal stability, tensile, and WVP properties of the resulting bio-nanocomposite films were evaluated and further discussed in this report. The results are very important for evaluating such films' possible applications as packaging material.

2. Materials and experimental details

2.1. Materials

Sugarcane bagasse fibers (SCB) was obtained from the SUNABEL-COSUMAR Group in Morocco. The moisture content of the raw SCB fibers was about 7 wt%. PVA (86–89% hydrolyzed) and CMC were purchased from Alfa-Easer. Analytical grade chemicals used in this work were purchased from Sigma-Aldrich and used without further purification.

2.2. Isolation and characterization of cellulose nanocrystals

The CNC aqueous suspension was prepared from SCB by using the sulfuric acid hydrolysis process, according to our previous work [31].

2.3. Bio-nanocomposite films processing

Bio-nanocomposite films were produced by the casting/evaporation technique. To begin, PVA solution was obtained by dissolving 1.5 g of the PVA powder in 40 mL of water under stirring for 1 h at 90 °C. Simultaneously, 1.5 g of CMC powder was dissolved in 150 mL of water under stirring for 2 h at ambient temperature. After cooling down to room temperature, the both PVA and CMC solutions were mixed together under stirring for 1 h at ambient temperature, resulting in a homogeneous solution of PVA/CMC (50/50 w/w). Thereafter, CNC aqueous suspension containing the desired amount of CNC (0.5, 2.5, 5, 10 wt%) was added to the PVA/CMC mixture under stirring for 30 min. Next, the obtained PVA/CMC-CNC film-forming solutions were sonicated for another 30 min. Subsequently, the PVA/CMC-CNC film-forming solutions were casted onto Petri dishes, and the water was evaporated at ambient temperature for 2 days. Finally, the obtained films were dried for 4 h at 80 °C for complete removal of the water. The PVA/CMC (50/50 w/w) blend film was also prepared according to the procedure mentioned above without the addition of the CNC. The films were coded as PVA/CMC, PVA/CMC-CNC-0.5, PVA/CMC-CNC-2.5, PVA/CMC-CNC-5, and PVA/CMC-CNC-10. The number indicates the weight fraction of CNC.

2.4. Characterization techniques

Atomic force microscopy (AFM) was performed with a Veeco Dimension ICON. The tapping mode was used to capture height images at a scan rate of 1.5 Hz. The sample used for AFM characterizations was deposited from a CNC dispersion on a mica sheet. Transmission electron microscopy (TEM) for CNC characterization was performed using a Philips CM200 microscope operating at 80 kV. Droplets of CNC suspensions were deposited on glow-discharged carbon-coated grids. The liquid in excess was blotted away with filter paper and a drop of 2% (w/v) uranyl acetate negative stain was added prior to drying. Zeta potential and diameter distribution, *via* dynamic light scattering (DLS), of CNC aqueous

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