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Preparation of a cellulose and water-based resin composite

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ABSTRACT

A cellulose (<50 wt%) and water-based resin composite was successfully prepared. This composite material was designed for ease of use with preferable characteristics similar to those of water. The mixture of the cellulose suspension was filtered and subsequently pressed under 100 psi for 10 min at 100 °C. The water-based resin, which was found to be completely polymerized, acted as a framework for holding the cellulose network structure. The curing condition can begin at room temperature, and the functional group of the water-based resin was the epoxy group. The water absorption of this composite was reduced to 100 times less than that of a neat cellulose sheet.

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1. Introduction

Due to the growing environmental awareness and concerns over the reliable availability of petrochemicals, the polymer industry and academia have been extensively pressed to design and produce polymers and composites based on renewable natural resources. To date, numerous types of bio-based polymers, such as cellulose [1], lignin [2,3], chitin–chitosan [4], and polylactic acid [5,6], have been developed to replace the use of plastics derived from petrochemical products. Cellulose, one of the most abundant naturally occurring bio-based polymers, is commonly found in the cell wall of plants and has received much attention in an effort to improve its engineering performance. This attention is primarily because the remarkable properties of cellulose, such as reinforcing capability, excellent mechanical properties, low density, high stiffness and high surface area [7] can successfully satisfy the various engineering product criteria. To the best of our knowledge, cellulose can be extensively isolated from wood by a pulping process. The main objective of the wood pulping process is to liberate cellulose, which can be accomplished either chemically or mechanically, or via a combination of these treatments. Chemical pulping can produce nearly pure cellulose by dissolution of lignin, hemicelluloses and extractives, while mechanical pulping can liberate nearly intact wood fiber. Cellulose can be successfully derived from wood with an effective cost-benefit comparison. The process and raw material for cellulose processing was easily found and was consequently further developed to industrial commercialization [8]. However, cellulose can also be extracted

* Corresponding author. Tel.: +1 416 826 4866; fax: +1 416 978 3834. *E-mail address:* sarute.ummartyotin@gmail.com (S. Ummartyotin). from certain types of bacteria species, called "Acetobacter Xylinum" [9]. Cellulose derived from this process is commonly called bacterial cellulose. Bacterial cellulose has the advantage of being free from lignin and hemicelluloses, which are commonly present in wood-based cellulose. Bacterial cellulose exhibits a strong degree of crystallinity of approximately 90% [10]. This favorable characteristic resulted in a high Young's modulus as well as a high relative density. Moreover, the particle sizes of bacterial cellulose are uniform. This uniformity is due to the effective control of the growth rate of bacterial cellulose during the bacterial culture process. However, from an industrial point of view, the costbenefit of bacterial cellulose was still high. This high cost has limited the development of bacterial cellulose in some lower price application areas.

Moreover, from the viewpoint of environmental preservation, "Green Technology" [11–13] policies have been widely employed. The use of products and processes that minimize the use and generation of hazardous substances should be promoted. As a means of promoting such products, the definition of a "Green Marker" for engineering application has been proposed, due to the fact that a "Green Marker" can be theoretically used in a predictive way, allowing the initiation of bioremediation strategies prior to the occurrence of irreversible environmental damage of ecological consequences. Due to these reasons, cellulose-based composites have been extensively promoted for use in numerous types of engineering application sectors, such as infrastructure, automotive, medical apparatus, active packaging, and electronics.

Recently, our research group has successfully developed a cellulose-based nanocomposite for use as an organic light-emitting diode substrate [14]. However, in the preparation process of this nanocomposite, the polymer matrix that utilized was required to by completely polymerized via UV-activation. The use of water instead







of organic solvents was preferably monitored for not only ease of fabrication but also environmental considerations. To address this issue, we proposed the use of a novel type of polymer matrix that is compatible with water called "water-based resin" [15]. From the fundamental point of view, this polymer matrix was theoretically designed using water as the media between two or more different types of distinct raw chemical reagents. The removal of this water, which diminished the effect of the water-solvent cage from the chemical reagent, leads to the reagents undergoing effective reaction and polymerization.

Improving the efficiency of this cellulose composite preparation addresses some of the environmental preservation concerns. As a result, the preparation of a cellulose and water-based resin composite is desired. The critical challenge lies in the preparation of cellulose and water-based resin composites that offer the ease of preparation, have reduced porosity, and have excellent mechanical properties. In this research work, we therefore present the use of water-based resin in the cellulose composite preparation process. The formulation between the water-based resin matrix and the cellulose was determined, and the relevant properties are reported.

2. Materials and methods

2.1. Materials

Water-based resin was obtained from BASF Corporation Company (Florham Park, NJ, USA). The main component was the mixture of carboxylated solution polymer and modified polyacrylate acid, and 50 wt% of water was used as solvent media. The trade name of this chemical reagent is Acrodur 950 L. Prior to use this chemical reagent, storage in a closed system was performed due to the air-sensitive nature of the reagent. Cellulose was extracted from wheat straw and wood, of which, the main component was reported as cellulose. Characterization of the cellulose extracted from wheat straw was reported in our previous article, and its characteristic properties matched with the cellulose derived from biomass [8]. All of the chemical reagents were used as-received and without any further purification.

2.2. Materials characterizations

2.2.1. Determination of the structural properties

A Fourier transform infrared spectrometer equipped with attenuated total reflectance (FITR-ATR) was used to investigate structural properties of the water-based resin. FTIR was performed on a Bruker Vector 22 mid-IR spectroscope (Bruker, Germany) equipped with an ATR crystal (50 mm \times 10 mm \times 2 mm) in the 45° incident angle configuration. All FTIR absorption spectra were recorded over the 4500–500 cm⁻¹ region at a resolution of 8 cm⁻¹ in 1024 scans using a deuterated triglycine sulfate (DTGS) detector. A straight line between the two lowest points in the respective spectral region was chosen as a baseline. The functional group region was taken and compared to the literature [16,17].

2.2.2. Determination of the morphological properties

The morphological properties were determined using a scanning electron microscope (SEM) (JOEL JSM-6301F). The SEM operated at an acceleration voltage of 15 kV and a magnification of $2000 \times$, $5000 \times$ and $30,000 \times$. The samples were stored in desiccators to prevent the effects of humidity. Then, each sample was place onto carbon tape and sputtered with gold particles prior to analysis.

2.2.3. Determination of the water vapor transmission rate (WVTR) properties

WVTR was measured in accordance with the ASTME96-66 standard. The controlled environment inside a closed desiccator was maintained at $50 \pm 2\%$ relative humidity throughout the experiment using a saturated aqueous solution of Ca(NO₃)₂, which will control the humidity to be 51% at 24.5 °C. The experiment was performed at room temperature (25 °C). The thicknesses of the samples were approximately 2.0–2.5 mm. The dried samples from the oven were stored in the desiccator for at least 2 h prior to the experiment to equilibrate the sample with the atmospheric moisture inside. Demineralized water was placed into a permeability Payne cup (2.5 cm diameter). The water level was kept to at least 2 cm from the sample to avoid contact between the sample and the water. The sample was then attached to the Payne cup. The sample weight change as a function of time was monitored every day for 2 weeks. The WVTR was calculated as follows:

WVTR =
$$G/tA$$

where G is the gain weight, t is the time and A is the test area.

2.2.4. Determination of the tensile properties

The tensile tests were conducted in accordance to the ISO Standard number ISO 527:1996. The sample was cut using a Zwick cutter into dog-bone specimens of type 5 A. The samples had the overall length and the gauge length of 75 mm and 20 mm, respectively. The initial distance between grips was 50 mm and its width at the grip end was 12.5 mm. The width at the narrowest part was 4 mm and the thickness was 0.3–0.5 mm. The testing speed was 1 mm/min. The specimens were preconditioned at 20 °C in 54% relative humidity in a desiccator containing a saturated solution of Mg(NO₃)₂ for at least 48 h prior to testing. The tensile test were conducted using an Instron universal material testing machine (Instron 4502, Instron Corporation, MA, USA) equipped with a 1 kN load cell. At least 5 specimens were tested per sample to obtain a statistical average.

2.3. Methods

2.3.1. Determination of the structural properties of cellulose and water-based resin

Cellulose suspension was extracted from biomass in Sain's work. Wood and wheat straw were used as raw materials. The raw materials were chemically and mechanically extracted in remove lignin and hemicelluloses, while de-ionized water (DI) was used as the solvent. The consistency of cellulose of the suspension was adjusted to 2.7 wt%. After the extraction process, cellulose suspension was stored at low temperature (~ 4 °C) to prevent any biological growth on the fiber. The suspension was characterized, and its properties were matched with those of cellulose.

The physical properties of as-received water-based resin were characterized during water removal in a fume hood, and the significant change of the structural properties was investigated using the FTIR-ATR technique.

2.3.2. Cellulose and water-based resin composite preparation

Less than 50 wt% of cellulose suspension was mixed with water-based resin. The water was removed from the suspension through filtration using a Buchner funnel fitted with a membrane filter (0.1 μ m mesh, 90 mm diameter), which was connected to a Buchner flask and a vacuum pump. The filtration process was continued until the mixture dried. The dried mixture was pressed under a hot press of compression force of 100 mpi and of temperature of 100 °C for water removal and the resin cure process.

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