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Effects of spent tea leaf powder on the properties and functions of cellulose green composite films

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ABSTRACT

The aim of the present work was to develop novel bio-based polymer composite films with improved tensile and thermal properties. In this work, cellulose and spent tea leaf powder (STLP) were used as the matrix and filler respectively in the preparation of biocomposite films. To make cellulose solution, cotton linters were dissolved in pre-cooled aqueous solution of 8 wt.% Lithium hydroxide and 15 wt.% urea. Tea is an important beverage of household and hotels and spent tea leaves form a conjugal solid waste. STLP was added to cellulose solution in 5–25 wt.% of weight of cellulose. The cellulose and cellulose/STLP composite films were prepared by regeneration method using ethyl alcohol coagulation bath. The dry films were characterized by optical microscopy, scanning electron microscopy, Fourier transform infrared spectroscopy, thermogravimetric analysis, and tensile tests. The effect of STLP loading on the properties of the cellulose/STLP composite films was studied. The results indicated that the composite films had higher tensile properties and thermal stability than the cellulose matrix. The dye adsorption ability of cellulose and cellulose/STLP composites in wet form was also studied. It was observed that the composite wet films had higher dye adsorption capacity than the matrix. Results of this study indicated STLP to be a promising green filler for polymer matrices.

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1. Introduction

As the industrial revolution progressed, the quest for newer and tailor made materials also increased. Polymers are such materials which find applications in almost every field. But unfortunately, the pollution caused by the synthetic polymers which are petroleum based is also increasing alarmingly [1]. Current production of polymer plastic goods globally is gradually mounting and has crossed 300 million tons [2]. Nearly all polymer plastic materials are non-biodegradable and hence at the time of disposal pose health problems [3]. Biodegradable materials are need of the hour to tackle the 'white' pollution [4]. They are not only environmentally friendly and decomposable, but also acquire outstanding biocompatibility [5,6].

Cellulose is the most abundant naturally occurring biopolymer which can be exploited as a matrix material in making biocomposites [7,8]. It has attracted great interest due to its better properties such as low density, high mechanical strength, low cost, durability, non-toxicity, renewability, biocompatibility, biodegradability, good film-forming performance, chemical stability and ease of making chemical derivatives [7–9]. There are several natural sources of cellulose such as green plants, algae, aerobic bacteria and some animals (tunicates). But cellulose has inherent inter- and intra-molecular hydrogen bonds, which makes it difficult to dissolve in traditional solvents. A variety of more or less exotic solvents were developed to overcome this problem and to make cellulose easier for tailored modification [10]. Over the past few decades, several cellulose solvent systems were developed for dissolving cellulose, such as LiCl/*N*,*N*-diethylacetamide [11], *N*-methylmorpholine-*N*-oxide [12], phosphoric acid [13], LiOH/urea and NaOH/urea [14], and ionic liquids [15]. From among these solvents, LiOH/urea and NaOH/urea are found to be more appropriate and cheaper. These alkali based solvents have ecological safety combined with high activity of interaction with cellulose. Regenerated cellulose from these systems has been successfully prepared in laboratory and at introductory pilot scale [16].

Currently, the ligno-cellulosic materials from agricultural crops and residues and agro industrial by-products are widely used to produce cellulose pulp and paper, feedstock for biofuels,

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reinforcing fillers for polymeric composite films and sorbents for waste water treatments [17–23]. Tea is one of the most popular beverages in the world and is professed as being healthy. Statistics indicate that the annual tea production in the world reached about 4.5 million tons [24]. Mainly tea is obtained from the leaves of *Camellia sinensis* L. Spent tea leaves (STL) remain after the preparation of tea and result as a solid waste product. Waste tea not only pollutes the environment but also represents a loss of valuable resource. To tackle this problem, during the past few decades, some researchers investigated the possibility of exploiting STL as adsorbents for synthetic dyes and toxic metals [25,26], bio-energy [27], and as reinforcing filler for construction [28] and polymer composites [24,29]. The aim of the present work was to use spent tea leaf powder (STLP) as filler in cellulose matrix, make biodegradable composite films and study their properties including the dye adsorption. To the best of our knowledge, this is the first study of making cellulose/STLP green composite films. In this study, the effect of STLP loading on the tensile properties and thermal stability of cellulose/STLP composites was investigated. FTIR spectra were used to probe filler-matrix interactions. The morphology of the composite films was studied by microscopy. The cellulose/STLP composite films in gel form were employed to study their dye adsorption capacity.

2. Materials and methods

2.1. Materials

Cotton linter pulp supplied by Hubei Chemical Fiber Co., Ltd. (Xiangfan, China) was used as received. The degree of polymerization (Dp) of the pulp provided by the manufacturer was ca. 620. LiOH and urea were supplied by Shanghai Chemical Reagent Co., Ltd., China and were used without further purification. Spent Tea leaves (STL) (Brooke Bond Wah Taj Tea, India) were collected from households after the tea was brewed once. This waste was washed thoroughly several times with distilled water and dried in the open air for several days and finally in the hot-air oven for 8 h at 100 °C. The cleaned and dried STL were made into powder using a kitchen grinder-mixer and sieved. The STLP which passed through a 35 μm sieve but retained on 25 μm sieve was used as the filler.

2.2. Dissolution of cellulose

Cellulose solution was prepared as described elsewhere [14]. The solution of aqueous (8 wt.% LiOH + 15 wt.% urea) was prepared and cooled to −12.5 °C. The cotton linter pulp was added (4 wt.%) to the pre cooled solvent and stirred vigorously at room temperature. A clear solution of cellulose was obtained within 2 min of stirring. This solution was centrifuged at a speed of 7200 rpm and temperature of 5 °C for 15 min. This stock solution was stored at 5 °C till it was used.

2.3. Preparation of cellulose/STLP composite films

The STLP was dried in an oven for 24 h to remove moisture if any. The dried powder was added to the cellulose solution in 5, 10, 15, 20 and 25 wt.% by weight of cellulose and mixed thoroughly. Upto 25 wt.% STLP was found to disperse well in the cellulose solution. The cellulose/STLP solutions were degassed to remove any air bubbles. The individual mixture solutions were spread on glass plates and regenerated using ethyl alcohol bath. The obtained gel films were washed thoroughly with distilled water several times to ensure removal of salts. The gel films were dried at ambient and the dry films of 50 μm thicknesses were obtained.

2.4. Morphology

The polarized optical micrographs of the cellulose/STLP composite films were recorded on Leica DMLP polarized optical microscope. The scanning electron micrographs of surface and brittle fractured cross-section of cellulose/STLP composite films were recorded using JEOL JSM 820 scanning electron microscope. The acceleration voltage of the microscope was set at 5 kV.

2.5. FTIR spectroscopic analysis

The FTIR spectra of cellulose, STLP, cellulose/STLP composite films were recorded on a Smart iTR ATR Nicolet is 10 FTIR spectrophotometer. All the spectra were recorded in the 4000–500 cm^{-1} region with 32 scans in each case at a resolution of 4 cm^{-1}. The FTIR spectra of these films before and after dye adsorption were also recorded in a similar manner.

Fig. 1. Flowers wrapped with films of cellulose/STLP composite films with different STLP contents: (a) 0 wt.%, (b) 5 wt.%, (c) 15 wt.% and (d) 25 wt.%.

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