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Nanocellulose coated woven jute/green epoxy composites: Characterization of mechanical and dynamic mechanical behavior

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ABSTRACT

This paper presents the preparation and characterization of nanocellulose coated woven jute/green epoxy composites. Waste jute fibers were used as precursor to purify and extract nanocellulose by chemical treatments. The prepared suspensions with 3, 5 and 10 wt% of nanocellulose were coated over jute fabric followed by preparation of composites by compression molding technique. The surface morphologies of treated jute fibers, jute cellulose nanofibrils (CNF), nanocellulose coated jute fabrics and fractured surfaces of composites were characterized by scanning electron microscopy (SEM). The crystallinity of jute fibers after different chemical treatments was measured by X-ray diffraction (XRD). The effect of nanocellulose coating over jute reinforcement on the tensile, flexural, fracture toughness and dynamic mechanical properties of prepared composites has been investigated. Fracture toughness was measured using single end notched bend (SENB) specimens and dynamic mechanical test was performed in three point bending mode. The results revealed the improvement in tensile modulus, flexural properties and fracture toughness except the decrease in tensile strength of nanocellulose coated woven jute/green epoxy composites as compared to uncoated jute composite. Dynamic mechanical analysis (DMA) results also showed the increase in storage modulus and reduction in tangent delta peak height of nanocellulose coated jute composites.

1. Introduction

Natural fiber reinforced polymer composites (NFPC) have gained considerable attention in the recent years due to their environment and economic benefits and low energy consumption [1,2]. Natural fibers offer many advantages over their synthetic counterparts (e.g. glass and carbon) such as cost effectiveness, light weight, easy to process, renewable, recyclable, available in huge quantities, low fossil-fuel energy requirements and the most importantly their high specific strength to weight ratio [1,3]. Thus natural fibers are considered promising candidates for replacing conventional synthetic reinforcing fibers in composites for semi-structural and structural applications [4].

Cellulose is an abundant, renewable and biodegradable naturally occurring material on earth that can be obtained from numerous resources [5]. It is an infinite source of raw material for environment friendly and biocompatible products. Lignocellulosic fibers such as jute, hemp and flax etc. are rich in cellulose, abundantly available and easy to handle and process. Cellulose micro/-nano fillers in polymers have already attracted considerable interest by improving the strength and stiffness of resulting composites [5–7]. Various types of cellulosic resources are used as precursors to extract and purify cellulose fibrils from lignocellulosic fibers. In the previous literature, cellulose micro/nano fibrils obtained from different precursors such as hemp fibers [8], pineapple [9], isora fibers [5] and jute fibers [10] are used as reinforcing filler in polymer matrices and resulted in the improvement of composite properties. However, in the present study, waste jute fibers are used as precursor to extract and purify nanocellulose which is subsequently coated over the woven jute fabric instead of using it as filler in matrix. Afterwards, the nanocellulose coated jute fabric is used as reinforcement in green epoxy polymer to form a composite material. Therefore, the objective of the present study is to characterize the mechanical and dynamic mechanical properties of composites reinforced with nanocellulose coated jute fabric. To the author's best knowledge, there is no study available in open literature yet that fulfills the above mentioned objective using nanocellulose coated jute fabric as reinforcement and green epoxy resin as matrix system.

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2. Materials and methods

2.1. Material

Woven jute fabric produced from tossa jute (*C. olitorius*) fibers having an areal density of 600 gm^{-2} with 5-end satin weave design was produced on a shuttle loom with 386 tex yarn. Warp and weft densities of the fabric were 6.3 threads per cm and 7.9 threads per cm respectively. Waste jute fibers, sourced from a jute mill, were used as precursor for purification and extraction of nanocellulose. Green epoxy resin CHS-Epoxy G520 and hardener TELALIT 0600 were supplied by Spolchemie, Czech Republic. The main characteristics of the resin system are reported in our previous study [11]. Sulphuric acid (H_2SO_4) and sodium hydroxide (NaOH) were supplied by Lach-Ner, Czech Republic. Sodium sulfate (Na_2SO_4) and sodium hypochlorite (NaOCl) were supplied by Sigma-Aldrich, Czech Republic.

2.2. Purification and extraction of nanocellulose from waste jute fibers

Waste jute fibers were chopped to approximate length of 5–10 mm and immersed in 2% sodium hydroxide (NaOH) solution for 2 h at 80 °C temperature maintaining a liquor ratio of 50:1. The process was repeated three times. The fibers were then washed with tap water several times to remove any traces of NaOH sticking to the fibers surface. The jute fibers were then bleached with 7.0 g/l sodium hypochlorite (NaOCl) solution at room temperature for 2 h under pH 10–11 and subsequently antichlored with 0.1% sodium sulphate (Na_2SO_4) at 50 °C for 20 min. Finally, the fibers were washed with tap water several times until the final pH was maintained at 7.0 and then allowed to dry at room temperature for 48 h and at 100 °C in an oven for 2 h. Alkali and bleaching treatments were used to purify cellulose and to remove maximum amount of hemicellulose and lignin from the fibers. The bleached jute fibers were milled using a high-energy planetary ball mill of Fritsch pulverisette 7. Milling process relies on the principle of energy release at the point of impact between balls as well as on the high grinding action created by friction of balls on the wall [12]. The sintered corundum container of 80 ml capacity and zirconium balls of 10 mm diameter were chosen for 20 min of milling. The ball to material ratio (BMR) was kept at 10:1 and the speed was kept at 850 rpm. Acid hydrolysis of milled jute fibers was conducted for 1 h at 45 °C under mechanical stirring using 65% (w/w) H_2SO_4. The fiber content during acid hydrolysis was 5% (w/w). The amorphous regions are preferably attacked by acids while crystalline regions are mostly insoluble in acids. The basic principle of acid hydrolysis is to release hydronium ions (H^+) for hydrolytic cleavage of glycosidic bonds in cellulose molecular chains within amorphous regions along the cellulose nanofibrils (CNF), thereby breaking the hierarchical structure of the fibril bundles into crystalline CNF [13]. After the hydrolysis, the suspension was diluted with cold water (4 °C) to stop the reaction, neutralized with NaOH solution and discolored by NaOCl solution. The supernatant was removed from the sediment and replaced by new distilled water several times. Finally, 3%, 5% and 10% (w/w) nanocellulose suspensions were prepared by increasing cellulose concentration and decreasing water concentration through vacuum filtration using Buchner funnel.

2.3. Coating of jute fabric with nanocellulose

The prepared nanocellulose suspensions (3, 5, and 10 wt%) were ultasonicated for 5 min with Bandelin ultrasonic probe and then applied on the surface of jute fabric by roller padding at room temperature. Finally, the coated fabrics were dried at 70 °C for 60 min.

2.4. Preparation of composites

The composites were prepared by hand layup method. The resin and hardener were mixed in a ratio of 100:32 (by weight) according to manufacturer recommendations, before hand-layup. The prepared resin mixture was poured on fabric layers and spread out by a hand roller. The gentle rolling action of hand roller confirmed the wetting of jute fabrics and the excess resin was squeezed out of the panel layup by the roller. The composite layup along with Teflon sheets were sandwiched between a pair of steel plates and cured at 120 °C for 1.0 h in mechanical convection oven with predetermined weight on it to maintain uniform pressure of about 50 kPa [11]. The fiber volume fraction (V_f) of all composites was in the range of 0.25–0.27. The prepared composite samples were designated as CF0 (uncoated), CF3 (3 wt% nanocellulose coated), CF5 (5 wt% nanocellulose coated) and CF10 (10 wt% nanocellulose coated).

2.5. Characterization and testing

2.5.1. Scanning electron microscopy (SEM)

The morphologies of alkali treated and bleached jute fibers, cellulose nanofibrils and nanocellulose coated jute fabrics were observed with Vega-Tescan TS5130 Scanning Electron Microscope at 30 kV accelerating voltage. The surface of fibers was gold coated prior to SEM inspection. Additionally, in order to measure the size distribution using SEM image, the morphology of cellulose nanofibrils was also observed on Zeiss Ultra Plus field emission scanning electron microscope (FE-SEM) at low accelerating voltage (1.0 kV) and low probe current (≈10 pA) to eliminate charging effect and sample damage due to interaction with primary electrons. The software used for image analysis was NIS Elements BR 3.22.

2.5.2. X-ray diffraction (XRD)

X-ray diffraction patterns were recorded on a PANalytical X′ Pert PRO MPD diffraction system for untreated jute, bleached jute and jute cellulose nanofibrils in order to examine the change in crystallinity of the material after bleaching and acid hydrolysis.

2.5.3. Mechanical characterization of composites

Tensile properties of the composites were characterized on an MTS series 370 servo-hydraulic load frame equipped with 647 hydraulic wedge grip of 100 kN load capacity at a cross head speed of 2 mm/min and gauge length of 100 mm in accordance with ASTM D3039-00 using rectangular specimens of dimension 200 × 20 × h mm^3, where "h" is the actual thickness of specimen in the range of 4.0–4.5 mm. Flexural test was performed in three

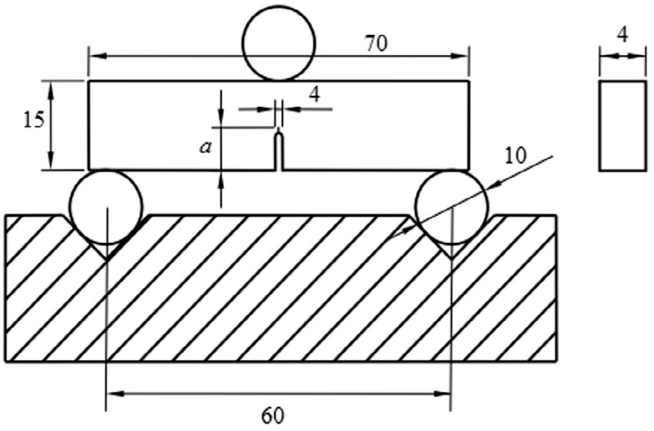

Fig. 1. SENB (single edge notch bending) specimen and fixture dimensions for flexural test (dimensions in mm).