



# Modeling and analysis of the creep behavior of jute/green epoxy composites incorporated with chemically treated pulverized nano/micro jute fibers



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## ABSTRACT

This paper reports the creep behavior of alkali treated jute/green epoxy composites incorporated with various loadings (1, 5 and 10 wt%) of chemically treated pulverized jute fibers (PJF) at different environment temperatures. Composites were prepared by hand layup method and compression molding technique. The creep and dynamic mechanical tests were performed in three-point bending mode by dynamic mechanical analyzer (DMA). The incorporation of PJF is found to significantly improve the creep resistance and strain rate of composites. Three creep models i.e. Burger's model, Findley's power law model and a simpler two-parameter power law model were used to model the creep behavior in this study. The time temperature superposition principle (TTSP) was applied to predict the long-term creep performance. The Findley's power law model was found to be satisfactory in predicting the long-term creep behavior. Dynamic mechanical thermal analysis (DMTA) results revealed the increase in storage modulus, glass transition temperature and reduction in the tangent delta peak height of composites with higher loading of PJF.

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## 1. Introduction

Natural fiber polymer composites (NFPC) are increasingly used nowadays in industrial applications as a substitute of polymer composites made with mostly used synthetic fibers such as glass and carbon etc. due to their environmental and economic benefits. Natural fibers are renewable, biodegradable, cost effective, safe to use, available in huge quantities, low fossil-fuel energy requirements and the most importantly their high specific strength to weight ratio (John and Anandjiwala, 2009; Mishra et al., 2003). This is of distinctive importance especially in interior transportation applications as it leads to reduction of vehicle weight for higher fuel efficiency and energy saving. Polymer composites used in engineering applications are often subjected to stress for a long time and at high temperatures. Creep (a progressive deformation of a material at constant stress) is very important end-use property for material applications requiring long term durability and reliability (Kreml and Khan, 2003). Considerable studies can be found in

literature on the creep behavior of natural fiber polymer composites. Different mathematical modeling techniques have also been applied to analyze the creep behavior of composite materials (Acha et al., 2007; Bledzki and Faruk, 2004; Hao et al., 2014; Jia et al., 2011; Marcovich and Villar, 2003; Xu et al., 2010). Researchers have also tried to study the creep behavior of polymer composites by addition of different kinds of fillers in matrices (Jia et al., 2011; Shen et al., 2004; Siengchin, 2013; Siengchin and Karger-Kocsis, 2009; Yang et al., 2006a,b). Addition of nano/micro fillers to polymers has shown improvements in the strength and stiffness of the resulting composites, however, research shows that these fillers tend to plasticize the composites. To the author's best knowledge, there is no study available in open literature on the creep behavior of alkali treated jute reinforced green epoxy composites incorporated with chemically treated pulverized jute fibers as reinforcing fillers. Among all the natural reinforcing materials, jute appears to be a promising fiber due to its high toughness and aspect ratio in comparison with other natural reinforcements (Acha et al., 2007) and occupies the second place in terms of world production levels of cellulosic fibers (Cai et al., 2000). The objective of the present study is to investigate the incorporation of pulverized nano/micro jute fibers prepared from waste jute on the creep behavior under

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the conditions of different temperatures and dynamic mechanical behavior of alkali treated woven jute/green epoxy composites. The modeling of experimental creep data is satisfactorily conducted using different creep models. Furthermore, the time-temperature superposition principle (TTSP) is employed in order to predict the long-term creep behavior of composites based on experimental data.

## 2. Materials and methods

### 2.1. Materials

Jute woven fabric produced from tossa jute (*Corchorus olitorius*) fibers having an areal density of  $600\text{ gm}^{-2}$  with 5-end satin weave design was produced on a shuttle loom. 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 for pulverization. Green epoxy resin CHS-Epoxy G520 and hardener TELALIT 0600 were supplied by Spolchemie, Czech Republic. Sodium hydroxide (NaOH) was supplied by Lach-Ner, Czech Republic. Sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) and sodium hypochlorite (NaOCl) were supplied by Sigma-Aldrich, Czech Republic.

### 2.2. Chemical pretreatment

The untreated fabric was first washed with 2 wt% non-ionic detergent solution at  $70^\circ\text{C}$  for 1.0 h prior to surface treatments to remove any dirt and impurities sticking to the surface. The jute fabric and waste jute fibers were then immersed separately in 2% NaOH solution for 1 h at  $80^\circ\text{C}$  maintaining a liquor ratio of 15:1. Alkali treated waste jute fibers were further treated with 7 g/l NaOCl solution at room temperature for 2 h under pH 10–11 and subsequently antichlored with 0.1%  $\text{Na}_2\text{SO}_4$  at  $50^\circ\text{C}$  for 20 min. Both fabric and waste fibers, after chemical pretreatment, were then washed with fresh 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  $100^\circ\text{C}$  in an oven for 2 h.

### 2.3. Pulverization of jute fibers

Pulverization of chemically treated waste jute fibers was carried out using a high-energy planetary ball mill of Fritsch pulverisette 7. Pulverization 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 (Baheti and Miliitky, 2013). The sintered corundum container of 80 ml capacity and zirconium balls of 10 mm diameter were chosen for 1 h of pulverization. The ball to material ratio (BMR) was kept at 10:1 and the speed was kept at 850 rpm.

### 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 and then weighed amounts of pulverized jute fibers (PJF) under 1, 5 and 10 wt% were mechanically mixed with epoxy resin at room temperature until a homogeneous mixture was obtained. The prepared resin/PJF 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. Each composite laminate comprised 3 layers of jute fabric with orientation of each layer in the same direction. The composite layup along with Teflon sheets were sandwiched between a pair of steel plates and cured at  $120^\circ\text{C}$  for 1.0 h in a mechanical convection oven with predetermined weight on it to maintain uniform pressure of about 50 kPa (Mishra et al., 2014). 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 U (untreated), A-0% (alkali treated jute fabric with 0 wt% of PJF), 1%, 5% and 10% (alkali treated jute fabric with 1, 5, 10 wt% of PJF) respectively.

### 2.5. Characterization and testing

#### 2.5.1. Characterization of pulverized jute fibers

The untreated and treated jute fabric and fibers were analyzed by FTIR spectroscopy. A Thermo Fisher FTIR spectrometer, model Nicolet iN10, was used in this study. The spectrometer was used in the absorption mode with a resolution of  $4\text{ cm}^{-1}$ . Particle size distribution of pulverized jute particles was studied on Malvern Zetasizer nano series based on dynamic light scattering principle of Brownian motion of particles. Deionized water was used as dispersion medium and it was ultrasonicated for 5 min with BANDELIN ultrasonic probe SONOPLUS before characterization. Refractive index of 1.52 was used to calculate particle size of pulverized jute. In addition, morphologies of pulverized jute fibers 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.

#### 2.5.2. Characterization of composites

Short-term creep tests were performed in three point bending mode at temperatures  $40^\circ\text{C}$ ,  $70^\circ\text{C}$  and  $100^\circ\text{C}$  using Q800 Dynamic mechanical analysis (DMA) instrument of TA instruments (New Castle DL, USA) for 30 min. The static stress of 2.0 MPa was applied at the center point of long side of the sample through the sample thickness for 30 min after equilibrating at the desired temperature and creep strain was measured as a function of time. The static stress was selected after performing a strain sweep test, where the linear viscoelastic region was defined for each of the composites ensuring that the creep tests were conducted in the linear viscoelastic region. The TTSP was selected for short-term creep tests performed at various temperatures for jute/green epoxy composites incorporated with various contents of PJF. The temperature range was  $40\text{--}100^\circ\text{C}$ , in  $5^\circ\text{C}$  steps, and the isothermal tests were run on the same specimen in the temperature range. The 2.0 MPa stress was applied for 10 min at each temperature. In every measurement, the specimen was equilibrated for 5 min at each temperature, in order to evenly adjust for the correct temperature of the specimen. The dynamic mechanical properties of composites were measured in 3-point bending mode using the above same instrument. The testing conditions were controlled in the temperature range of  $35\text{--}200^\circ\text{C}$ , with a heating rate of  $3^\circ\text{C}/\text{min}$ , fixed frequency of 1 Hz, preload of 0.1 N, amplitude of  $20\ \mu\text{m}$ , and force track of 125%. The samples having a thickness of 4.5–5 mm, width of 12 mm and span length of 50 mm were used for both creep and DMA testing. Two replicate samples were tested for each test condition and average values are reported.

### 2.6. Creep modeling

The non-linear curve fit function of the OriginPro 9.0 software was used for modeling the creep curves and fitting the models to the experimental data. The minimum sum of squared deviation of experimental data from the creep models and correlation coefficient ( $R^2$ ) were selected as criterion (Meloun and Miliitky, 2011). The correlation coefficient value  $R^2$  is defined as model sum of squares divided by total sum of squares. A better goodness-to-fit is obtained when  $R^2$  is closer to 1.

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