

Flexural creep behaviour of jute polypropylene composites

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Abstract. Present study is about the flexural creep behaviour of jute fabric reinforced polypropylene (Jute-PP) composites. The PP sheet and alkali treated jute fabric is stacked alternately and hot pressed in compression molding machine to get Jute-PP composite laminate. The flexural creep study is carried out on dynamic mechanical analyzer. The creep behaviour of the composite is modeled using four-parameter Bürgers model. Short-term accelerated creep testing is conducted which is later used to predict long term creep behaviour. The feasibility of the construction of a master curve using the time–temperature superposition (TTS) principle to predict long term creep behavior of unreinforced PP and Jute-PP composite is investigated.

1. Introduction

Natural fibre-reinforced polymer composites are becoming more popular owing to their potential as an environment friendly option to conventional synthetic glass fibre composites. Natural fibre composites possess the merits such as easy availability, low cost, light weight and high specific stiffness. The natural fibre composite mostly find use in automobile and civil construction applications and therefore the need for time-dependent viscoelastic study of such composites.

For better interfacial adhesion in case of natural fibre polymer composites two options are generally adopted (i) using modified matrix by utilizing coupling agents and (ii) chemical treatment of natural fibres. The chemical modification of natural fibres is necessary for increased adhesion between the hydrophilic jute fibres and hydrophobic PP matrix. One of the most common and efficient methods of chemical modification is alkali treatment of fibres with successful results [1]. In the present study, jute fabric was subjected to alkali treatment.

The creep behaviour of polymer composite is an important study from the product durability point of view. The creep study involves long duration testing which is considered to be one of the biggest constraint. The Time-Temperature Superposition (TTS) principle involves short-term accelerated creep testing at different high temperatures. This facilitates in evaluating long-term creep behaviour in experimentally feasible time. The creep behaviour of natural fibre composite have been studied by various researchers; kenaf fibre/high-density polyethylene(HDPE) [2], bagasse/PVC and bagasse/HDPE [3], wood fibre/polypropylene composites [4, 5]. The time-temperature superposition (TTS) principle is employed for long-term creep study [6]. Inorder to implement the TTS principle successfully the constant stress applied during the creep test should be in linear viscoelastic region.

In the present study jute fibre in fabric form along with recyclable thermoplastic matrix that is polypropylene (PP) is used. The ASTM D2290 is the standard test method for flexural creep study.



The creep study of Jute-PP composite is carried out in the following manner:

- 1) Experimental creep data for the composite is obtained.
- 2) Modeling of the creep data obtained experimentally.
- 3) Predicting long-term creep behaviour based on short-term creep measurements.

2. Experimental

2.1 Materials and sample preparation

Polypropylene homopolymer in granules form (REPOL H110MA, Reliance Ltd, India.) of density 0.910 g/cc (MFR of 11g/10min; 230°C/ 2.16 kg) and commercial plain weave gunny jute fabric (370 g/m²) are used for the study.

Jute fabric is soaked in 5% NaOH solution for 12 hours at room temperature followed by washing with distilled water then neutralized with dilute acetic acid. The fabrics are dried at room temperature for 8 hours followed by oven drying at 100°C for 6 hours.

Polypropylene sheets of various thicknesses are prepared from PP granules by compression molding using a hydraulic press. The varying thickness of PP sheets control the jute weight fraction in the composite. The PP sheets and alkali treated jute fabrics are stacked alternately and hot pressed in compression molding machine at 165°C and 8 MPa for 20 mins to get Jute-PP composite laminate.

2.2 Dynamic Mechanical Analysis: Viscoelastic properties

Viscoelastic properties, such as storage modulus (E'), loss modulus (E'') and mechanical damping parameter ($\tan \delta$) as a function of temperature are measured using TA Instruments Q800 Dynamic Mechanical Analyzer. The measurements are carried out in single cantilever configuration using rectangular specimen of dimensions 35 x 12.5 x 3 mm over a temperature range of -50°C to 160°C, at a heating rate of 2°C/min. The samples are scanned at a fixed frequency of 1 Hz, with amplitude of 20 μ m. All measurements are performed under nitrogen atmosphere.

2.3 Dynamic Mechanical Analysis: Creep behaviour

Flexural creep testing is carried out using TA Instruments Q800 Dynamic Mechanical Analyzer. A stress sweep at 1 Hz frequency at 35°C was done to determine the linear viscoelastic region of the composite. For stepped isothermal creep tests, all tests were started at 30°C upto 90°C at 20°C temperature increment. An isothermal soak time of 5 min was set and the specimen was subjected to constant stress of 1 MPa for a duration of 60 minutes at each temperature step. The creep tests were done in creep and creep-TTS mode in single cantilever configuration.

3. Results and Discussions

3.1 Stress Sweep test

An important consideration for the creep test is the magnitude of constant stress applied. The magnitude of stress used during the experimentation should lie in the linear viscoelastic region (LVR) for successful implementation of the TTS principle. The limiting value of stress is therefore determined using stress or strain sweep test. Figure 1 shows the storage modulus (E') vs stress plot for PP. Although the stress-strain relation appears to be linear, the drop in E' need to be observed to determine the LVR. The flat region in the range 0 to 1.5 MPa depicts the linear viscoelastic region where the storage modulus is observed to be a constant value.

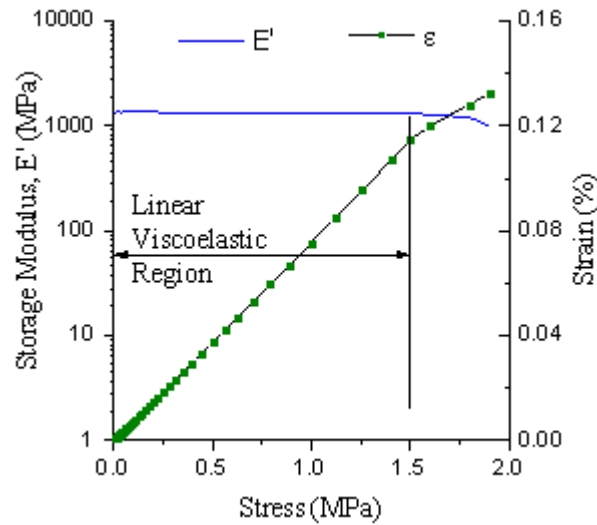


Figure 1. LVR of PP

The drop in E' value represents the initiation of non-linear region. For PP, the constant stress to be applied for creep study can be safely taken as 1 MPa as seen from Figure 1 which is well within the linear viscoelastic region.

3.2 Dynamic Mechanical Analysis: Viscoelastic properties

In order to determine the glass transition behaviour of PP and the Jute-PP composites dynamic mechanical analysis was carried out in the temperature range -50° to 160°C . For the Time-Temperature Superposition (TTS) principle for the long-term creep study to hold, it is essential to operate in temperature ranges where primary or secondary transitions do not occur in the polymer.

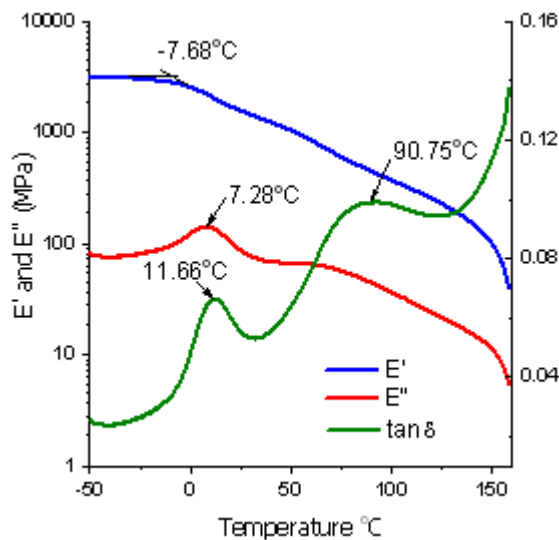


Figure 2. Viscoelastic properties of PP

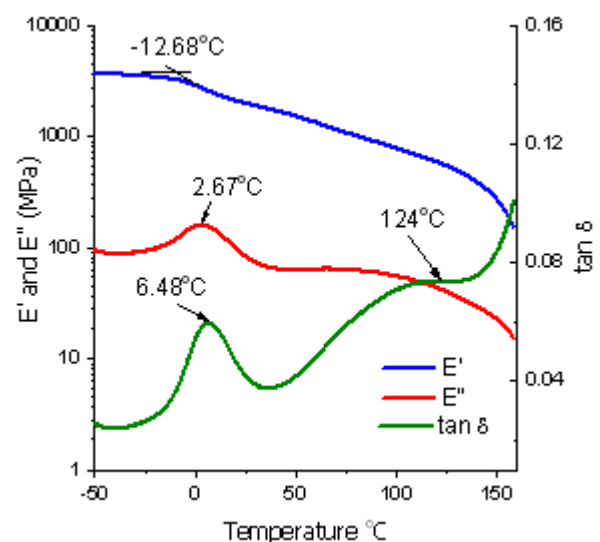


Figure 3. Viscoelastic properties of Jute-PP.

The viscoelastic properties for PP as a function of temperature is shown in Figure 2. The glass transition temperature (T_g) is interpreted from the change in slope on the storage modulus (E') or the peak of the $\tan \delta$ plot. It is observed from Figure 4, the primary glass transition for PP occurs at a temperature of 11.66°C as depicted by peak of $\tan \delta$ curve and on the E' curve, the onset of slope change i.e. at -7.68°C. The secondary transition for PP is observed at higher temperature of 90.75°C.

Figure 3 illustrates the viscoelastic properties of 25% Jute-PP composite. Due to introduction of jute reinforcement there is a shift in PP T_g values. The safe temperature range to be used for the creep study for the TTS principle to be valid lies in the range of 11°C to 91°C. It is worth mentioning that the maximum service temperature for PP is around 100°C.

3.3 Dynamic Mechanical Analysis: Creep study

In a creep experiment, a constant stress is applied instantaneously to the specimen and the resulting strain is measured as a function of time. The creep compliance is obtained by dividing the time-dependent strain with the constant applied stress. Figure 4 shows creep-recovery behaviour of PP and 25% Jute-PP composite conducted at 50°C isothermal temperature for 30 minutes at 1 MPa constant stress. The creep time was set to 10 min while the recovery time was 20 min. The creep strain of jute-PP composite is observed to be lower than unreinforced PP which is mainly due to creep resistance offered by the jute fabric.

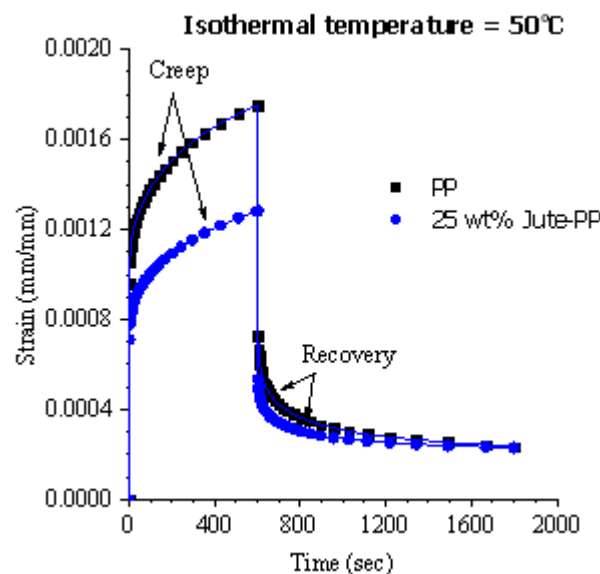


Figure 4. Creep-recovery curves of PP and Jute-PP composite.

Experimental data is represented by symbols and solid line represents the Burgers model

There are various mathematical models available for linear viscoelastic response[7]. The creep simulation for the experimental creep data can be carried out using a four-element Burgers model which is widely used to predict the creep behaviour. The springs and dashpots in the model represent the elastic and viscous behaviour respectively.

$$\varepsilon(t) = \frac{\sigma}{E_M} + \frac{\sigma}{E_K} \left(1 - \exp\left(-\frac{E_K}{\eta_K} t\right) \right) + \frac{\sigma}{\eta_M} t \quad (1)$$

where $\varepsilon(t)$ is the creep strain, σ is the constant applied stress, t is the time, E_M and E_K are the moduli of Maxwell and Kelvin springs respectively, η_M and η_K are the viscosities of Maxwell and Kelvin dashpots used in the Burgers model.

A non-linear least squares regression analysis is carried out on the creep data to determine the four parameters of Burger's model. The goodness-of-fit was estimated by sum of squares due to error (SSE). A value close to 0 indicates that the fit is useful for prediction.

$$SSE = \sum_{i=1}^n w_i [\varepsilon(t_i) - \hat{\varepsilon}(t_i)]^2 \quad (2)$$

where $\varepsilon(t_i)$ represents the experimental strain observed at time t_i , $\hat{\varepsilon}(t_i)$ represents that predicted by the model and w_i represents the difference between two time samples. The constants of Burgers model obtained by curve fitting are shown in Table 1 below

Table 1: Four-parameter estimation of Burgers model

	E_M (MPa)	E_K (MPa)	η_K (MPa-s)	η_M (MPa-s)	SSE
PP	3982.5	1023.3	4219.9	960614.8	2.61E-07
25 wt% Jute-PP composite	7710.1	1320.8	4273.1	1281722.6	1.055E-07

3.4 Dynamic Mechanical Analysis: Accelerated Creep study

The strain (%) vs time plot is shown at various temperature (in the range 30 to 90°C.) for unreinforced PP and alkali treated 25wt% jute-PP composite in Figure 5 and Figure 6. An increase in the instantaneous deformation and creep rate is observed as the temperature increases. This is attributed to the fact that the mobility of the molecular chains increases at higher temperatures. This creep behaviour at higher temperature is shifted horizontally in order to predict creep behaviour at longer

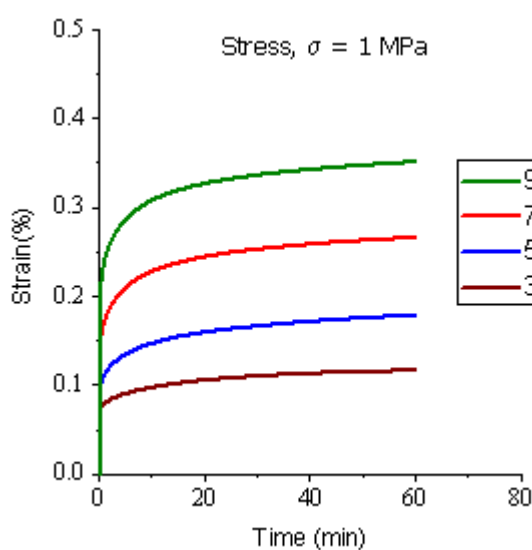


Figure 5. Creep behaviour of PP

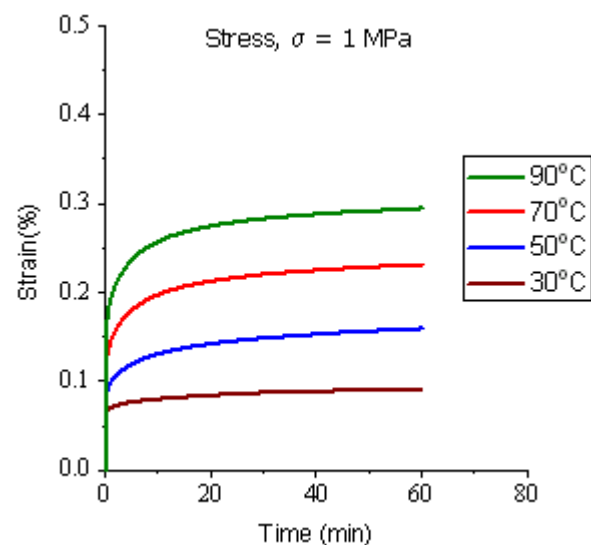


Figure 6. Creep behaviour of Jute-PP composite

duration. The underlying justification for this shifting process to predict long duration creep being the mobility of the polymer chains at higher temperature is equivalent to what would result at longer duration. Therefore, for every temperature, there is a certain characteristic shift-factor. One mathematical model that is used to relate the temperatures to the respective shift-factors is the Arrhenius model

$$\ln a_T = \frac{E_a}{R} \left(\frac{1}{T} - \frac{1}{T_o} \right) \quad (3)$$

where a_T is the horizontal shift factor, E_a is the activation energy, R is the universal gas constant, T_o is the reference temperature (K) and T is the temperature the test is run (K). The shift factors obtained need to be ascertained whether they conform to Arrhenius or the Williams-Landel-Ferry (WLF) model [8]. When a plot of shift factor a_T against the reciprocal of absolute temperature shows a linear relation, it indicates suitability of Arrhenius equation. The shift factors required for generating the master curves were modeled based on Arrhenius equation as seen in Figure 7. The activation energy E_a is tabulated in Table 2.

Table 2. Activation energy, E_a in Arrhenius equation

Activation energy, E_a (kJ/mol)	
PP	172.9
25wt% Jute-PP Composite	155.3

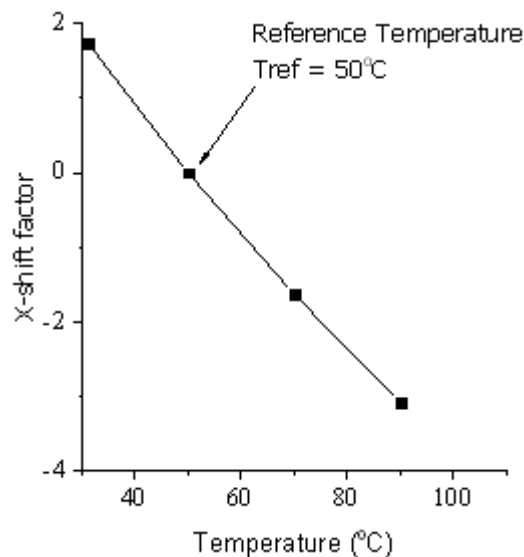


Figure 7. Shift factor for PP

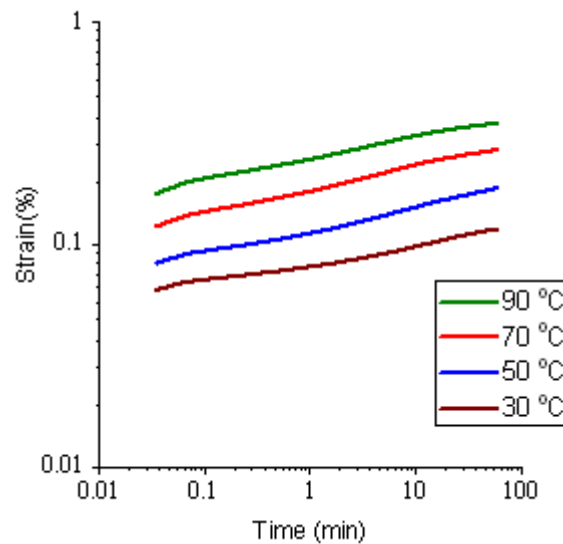


Figure 8. Log-Log plot of creep strain

The 60 minute duration creep data at different temperatures namely 30, 50, 70 and 90°C is plot on logarithmic scales as shown in Figure 8 in order to obtain the horizontal shift (time) factors.

The creep master curves are generated at a reference temperature (T_{ref}) of 50°C. The creep curve data above the $T_{ref} = 50^\circ\text{C}$ (i.e. 70 and 90°C) is shifted to the right of the 50°C reference creep curve and data below $T_{ref} = 50^\circ\text{C}$ i.e. 30°C data is shifted to the left on the log-log strain-time plot. The

generation of master curve was facilitated by use of Rheology Advantage software from TA instruments. As seen in Figure 9, the creep strains for 25wt% Jute-PP are 15% lower than unreinforced PP at 36500 minutes (approximately 25 days).

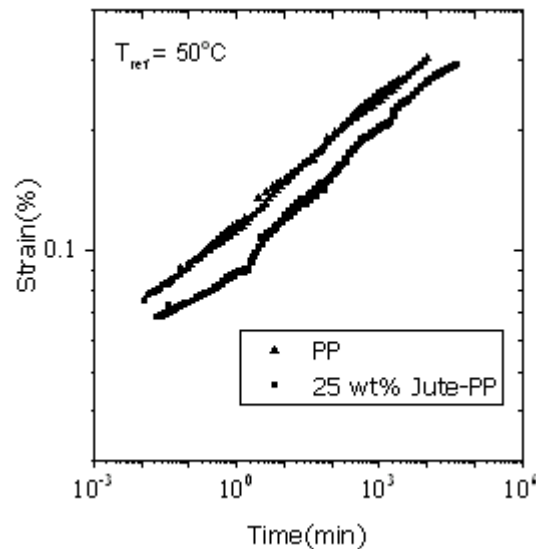


Figure 9. Master curve constructed from short-term creep data by horizontal shifting of creep data at different temperatures. The reference temperature is 50°C.

Conclusions

The creep behaviour of alkali treated jute-PP composite was investigated in this study. The DMA test revealed that the operating temperature range for PP as 80°C. However with the reinforcement of jute in PP exhibits increase in the operating range up to 117°C. The creep-recovery behaviour of PP and jute-PP composite was undertaken and the experimental trend was modeled with four-parameter Burgers model which gives reliable prediction. The TTS principle is applied to both jute-PP composite as well as unreinforced PP. It is observed that the alkali treated jute reinforcement enhances the creep behaviour of jute-PP composites.

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