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Effect of time at temperature for natural fibres

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Abstract

The manufacture of natural fibre reinforced polymer matrix composites is generally assumed to be limited to temperatures not exceeding 200°C. However, given the covalent bonding in the various constituents of natural fibres, they should retain properties to an extent dependent on the time at temperature. This study considers fibre mechanical properties after heating in air for various temperatures and times.

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Keywords: Jute; Natural fibres;Time-at-temperature

Nomenclature

RH	relative humidity
T_d	Degradation temperature
T_g	Glass transition temperature
T_m	Crystalline melting point

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1. Introduction

There is increasing interest in the use of natural fibres as the reinforcement for polymer matrix composites. Natural fibres may come from animal, plant or mineral sources, but the current principal interest is for bast (stem) fibres from plants [1-5]. Plant materials primarily contain cellulose fibres in a matrix of hemi-celluloses and pectins with increasing proportions of lignins arising as the plant ages.

Cellulose is a hydrophilic molecule, so it is essential for composite manufacturing processes that the reinforcement fibres is dried before use to prevent trapped moisture becoming voids in the composite matrix. ISO12215-4:2002 [6] requires that "The relative humidity in the moulding shop shall be maintained within the limit recommended by the material manufacturers". Armstrong and Barrett [7] recommend drying cycles (for aramid honeycomb) of 16 h at 40°C, 3 h at 100°C or 1 h at 160°C, with the materials then used within 1 h of drying. Davies [8] suggested that dried cores should be stored in a dedicated room at 50°C and <10% RH.

Dry cellulose is variously reported to have glass transition temperatures (T_g) in the range 84-250°C with T_g decreasing with increasing water content. Roig et al report that the T_g of cellulose is strongly dependent on the degree of crystallinity [9]. Szcześniak et al. reported colour changes at 180°C and significant weight losses (~70%) between 220°C and 300°C [10]. Cellulose normally decomposes in air before melting because of the extensive hydrogen bonding between the cellulose molecules. The melting temperature of cellulose is reportedly in the range 500-518°C (presumably under inert gas or in vacuo) [11].

Ligno-cellulosic fibres degrade at around 200°C in air [12-14], so there are few commercial thermoplastic matrix systems available for load-bearing applications at ambient temperature (i.e. below T_g to avoid creep) yet processed without degradation of the fibres ($T_m \approx T_g$+200°C). A notable exception is poly(lactic acid) (PLA) with T_g = 55-65°C, crystalline melting point, T_m = 120-170°C and degradation temperature, T_d, just above 200°C. PLA has a narrow process temperature window. PLA-matrix composites should be able to take stress at ambient temperatures without creep occurring.

Use of other polymer matrix systems might be possible if the time at higher temperatures is minimised (or if processing is under vacuum or in an inert atmosphere, although the process costs will inevitably be higher). This paper seeks to identify a process window for time and temperature where natural fibres can processed without significant degradation of their mechanical performance.

2. Experimental materials and methods

This study used fibres from a well-characterised single batch of quasi-unidirectional jute with an areal weight of 880 gm^{-2} [15-22]. Batches of fibre were heated in an oven to set temperatures (180-220°C step 10°C) for predetermined times (5, 10, 15, 30, 60 and 120 minutes). The individual fibres were mounted using epoxy resin on cards, based on the Grafil Test 101.13 for carbon fibres, and tested for apparent* filament tensile modulus and strength with a 50 mm gauge length. Five samples at each combination of temperature and time were tested and analysed. A number of filaments failed before they were ready for test and are not included in the analysis. Testing was undertaken in compliance with BS ISO 11566:1996 on an Instron single column test machine (serial number 3345K1669 calibration certificate E23032116110441 expiring March 2017, i.e. during the test series) with an Instron 10 N force transducer (model 2519-101, serial number 64650), with at 0.5 mm/min under ambient conditions. Fibre diameters were measured after failure and the analysis assumed circular cross section for the fibres. For true moduli and strength, the values should be increased using the fibre area correction factor* for (this batch of) jute fibres at 1.42 as established by Virk et al [20].