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Jute fibre/epoxy composites: Surface properties and interfacial adhesion

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ABSTRACT

Jute fibres were surface treated in order to enhance the interfacial interaction between jute natural fibres and an epoxy matrix. The fibres are exposed to alkali treatment in combination with organosilane coupling agents and aqueous epoxy dispersions. The surface topography and surface energy influenced by the treatments were characterized. Single fibre pull-out tests combined with SEM and AFM characterization of the fracture surfaces were used to identify the interfacial strengths and to reveal the mechanisms of failure.

1. Introduction

Natural fibres and their composites have attracted the attention of scientists worldwide because of their low density and attractive specific properties at low price, combined with recycleability and renewability. However, the application of natural fibres such as jute, flax, or hemp to composites has some drawbacks, e.g. the hydrophobicity of the fibres, the relatively poor thermal stability of the natural fibre composites and especially the poor compatibility towards a hydrophobic polymer matrix, resulting in weak interfaces and poor mechanical properties of the composites. Most of these drawbacks may be overcome by the use of surface modification [1–3].

In the as grown state, most natural fibres have a cellulose-rich core, whereas the outside is covered by cementing which includes waxes, fats, lignin, pectin and hemicellulose. This cementing prevents the formation of a strong interphase. The fibre surface is hydrophilic and therefore poorly suited for the use in hydrophobic polymer matrices. The aim is therefore to improve the interphase. In order to characterize the fibre matrix interphase, micromechanical methods which specifically probe the interfacial strength, are well established. There has been some research performed by the single fibre pull out test [4–6] or microbond [7] for estimating the apparent interfacial shear strength τ_{app} of natural fibre composites.

There are a number of approaches to rendering the fibre surface more compatible to the matrix. A first step is often an alkali treatment to reduce the cementing and increase the cellulose fraction [8–10]. This is often followed by fibre coating [1,2,11].

Alkali treated jute fibres were treated with oligomeric siloxane, which improved the adhesion in epoxy matrix, leading to improved mechanical properties [10]. Sydenstricker [5] subjected sisal fibres to a NaOH bath, followed by N-isopropyl-acrylamide treatment to improve the interfacial adhesion towards a polyester resin and the thermal stability of the composites. The influence of fibre surface modifications on the interfacial shear strength of the composites based on sugar cane bagasse fibres and polystyrene was assessed by Garcia-Hernandez et al. [6]. The interfacial shear strength followed the order: non-treated fibres < fibre treated with NaOH or silanized with 3-(trimethoxysilyl)-propylmethacrylate ≪ fibres grafted or coated with polystyrene. A significant increase in interfacial shear strength was achieved through a combination of treatments due to the synergistic effect of the surface treatment techniques. Alkali treatment of sisal bundles for the reinforcement of polyester or epoxy matrices was studied by Towo and Ansell [12], in mechanical as well as fatigue testing the treated fibres were superior to the untreated ones.

A different approach is the use of matrix modification, e.g. the addition of a small proportion of maleic anhydride grafted PP to a polypropylene matrix. In a previous work on jute fibre reinforced polypropylene matrix composites, the authors could show that matrix modification by maleic anhydride grafted polypropylene has significantly improved the mechanical performance and interfacial adhesion [1], as well as the thermal, dynamic mechanical behaviour and aging resistance [13]. A novel sensor material based on natural fibre composites can be prepared by coating the surfaces

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of jute fibres with multi-walled carbon nanotubes, forming jute/epoxy interphases which have electrically semiconducting properties and permit sensor applications for temperature, relative humidity, or stress/strain [14].

The objective of the present work is to investigate the effect of surface treatments on the jute fibre surface and jute/epoxy interface. The morphology and wetting behaviour of the jute fibre surface after different treatments with sodium hydroxide, silane coupling agents and an aqueous epoxy dispersion were characterized using Scanning Electron Microscopy (SEM) as well as Atomic Force Microscopy (AFM) and the Wilhelmy balance technique, respectively. The apparent interfacial shear strength τ_{app} of the differently treated fibres towards an epoxy matrix is determined with the single fibre pull-out test and the fracture surface is imaged by SEM and AFM.

2. Experimental

2.1. Materials

Jute yarn of 390 tex yarn fineness was obtained from Schilgen GmbH (Germany). As matrix, epoxy resin EPR L 20 and curing agent EPH 161 (Momentive Specialty Chemicals Germany) mixed with weight ratio of 100:25 was used.

2.2. Fibre surface treatments

2.2.1. NaOH treatment

The fibres were de-waxed in a mixture of ethanol and toluene with the ratio of 100:50, at 50 °C for 2 days, then washed with water and dried (4 h, 80 °C then 30 min 100 °C under vacuum).

Dewaxed jute fibres were treated by NaOH 1%, 4 h, washed in water, neutralized with diluted acetic acid to remove the remaining alkali, rinsed again with water, and dried as above.

2.2.2. NaOH/(APS+ED) treatment

The 3-Aminopropyl-triethoxy-silane solution 1% (APS) was prepared by hydrolysis in water, then adjusted to pH = 4.5 with acetic acid. Next, epoxy dispersion (ED) was diluted with distilled water and added with the remaining water to achieve the desired concentration. NaOH treated jute fibres were immersed in the above solution for 1 h, the mixtures were drained in a two roller system (Mathis, Switzerland) and the fibres were dried as described above.

2.2.3. NaOH/PAPS treatment

The 3-Phenyl-aminopropyl-trimethoxy-silane aqueous solution 1% (PAPS) was prepared by hydrolysis in water at pH = 2.9. NaOH treated jute fibres were immersed in the solution for 1 h, drained with a two roller system (Mathis, Switzerland) and the fibres were dried as described above.

2.3. Methods

2.3.1. Topography and roughness

In order to investigate the fibre surfaces, the morphology and topography of fibre surfaces were studied by SEM (LEO 435 VP) and AFM (Veeco D3100, USA). Prior to the SEM observation at an acceleration voltage of 5–10 kV, the fibre surfaces were sputter coated with gold. Several images were recorded at different locations to verify the reproducibility of the observed features.

2.3.2. Wettability

The surface energy measurement were made using a Krüss K14 tensiometer (Krüss GmbH, Germany) in α-bromo-naphtalene, toluene 1,5-pentadiol and water. One end of a single elementary fibre was attached to the hook of an electro micro-balance (sensitivity ± 0.1 μg). The free end of the fibre was immersed into the liquid (final depth of immersion 1 mm, rate 1 mm/min, mean of 10 measurements). From the test, the force versus position loop was recorded and the force was converted to the contact angle (θ) using the Wilhelmy equation. The surface energy (γ^T) and its two components (non-polar or dispersive interaction, γ^d and the polar interactions, γ^p) of the fibre were then calculated to evaluate the wetting behaviour of the fibre surfaces.

2.3.3. Single fibre pull-out test

The interfacial adhesion strength of jute fibre/epoxy matrix micro-composites was evaluated by single fibre pull out test [15]. Using an embedding equipment designed and constructed at the Leibniz Institute, the model micro-composites were prepared by accurately embedding one end of the single elementary fibre in the matrix perpendicularly with a pre-selected embedding length (40–150 μm) at controlled atmosphere and temperature. The pull out test was carried out on a self-made pull-out apparatus with force accuracy of 1 mN, displacement accuracy of 0.07 μm and a loading rate of 0.01 μm/s at ambient conditions. The maximum force, F_{max}, required for pulling the fibre out of the matrix was measured. After testing, the fibre diameter, d_f was measured by

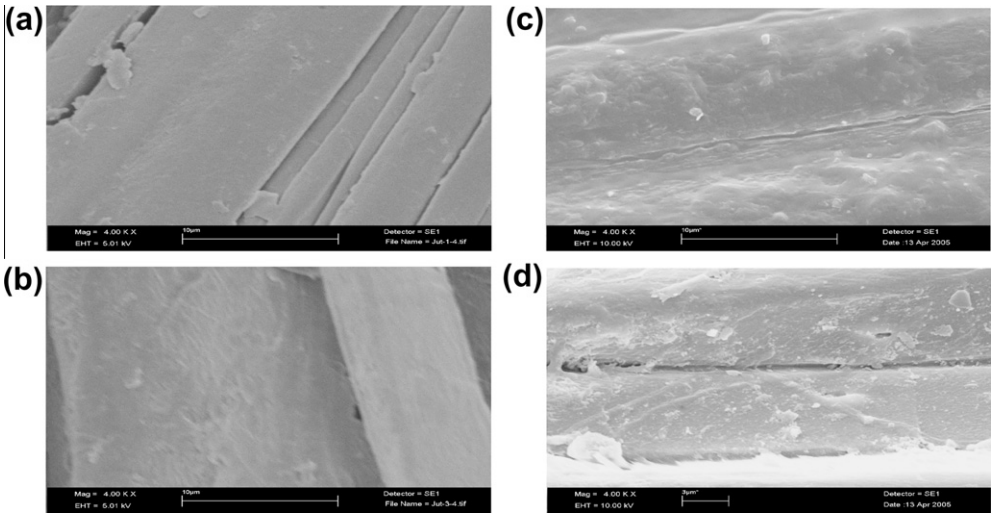

Fig. 1. SEM images of (a) untreated jute, (b) NaOH, (c) NaOH/(APS + ED), and (d) NaOH/PAPS treated jute fibres.