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Investigation of microstructure and tensile properties of porous natural coir fibre for use in composite materials

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ABSTRACT

Natural coir fibres are studied for use as reinforcement in composite materials. In order to efficiently use the fibres and understand the composite properties, the microstructure and the mechanical properties of coir fibres are investigated in this study. X-ray microtomography in SEM (SEM-CT) and SEM image analysis are used to examine the fibre internal structure including the organisation of elementary fibres, microfibril angles and fibre porosity. Mechanical properties of coir fibres are determined by performing fibre tensile tests, in which an integrated optical strain mapping system is used to define fibre strain for producing more reliable values of *E*-modulus and strain at failure. The results show that technical coir fibres comprise plenty of elementary fibres and a lacuna at the centre. The elementary fibre is built up by two main cell walls which consist of bundles of microfibrils with a large misorientation with respect to the elementary fibre axis. Coir fibres appear to have a high porosity of 22 to 30%. The high microfibrillar angle in the coir fibres leads to the low stiffness in fibre direction and to high elongation to failure thanks to reorientation of the microfibrils under tensile loading.

1. Introduction

Natural fibres have recently attracted the interest as composite material reinforcement, thanks to their good mechanical properties in combination with lightweight. Additionally, they have environment-friendly characteristics such as low energy utilisation, renewability and biodegradability. As extracted from natural plants, natural fibres have a variation in mechanical properties which are mainly attributed to the microstructure and chemical composition of the fibres. To efficiently use natural fibres in composite materials, it is necessary to investigate the morphology, structure and mechanical properties of the fibres, which influence the properties of their composites.

The natural fibres used in composites are commonly defined as technical fibres. A technical natural fibre consists of several cells referred to as elementary fibres. The elementary fibre is mainly formed out of multiple cellulose–lignin/hemicellulose cell wall layers, in which crystalline microfibrils based on cellulose are connected into the cell wall layer by amorphous lignin and hemicelluloses (Bledzki and Gassan, 1999; Burgert, 2006; Fengel and Wegener, 1989; Lefeuvre et al., 2014). The cell wall layers can have different thickness, chemical organisation and orientation of the cellulose microfibrils (microfibrillar angle – MFA) (Burgert, 2006; Müssig and Stevens, 2010; Tomczak, 2007).

The mechanical properties of natural fibres depend on the organisation of the cell walls with regard to cell wall/lumen ratio and the cellulose MFA in the dominant cell wall layers. In relation with fibre cross-section, higher density fibres are stiffer and stronger than the lower density ones. The elastic modulus and strain at failure of fibres are also dependent on the MFA. A small MFA, in which cellulose microfibrils are oriented almost parallel to the axial direction, leads to a high modulus of elasticity, whereas the stiffness is substantially decreased for higher MFA's (Lefeuvre et al., 2014; Navi et al., 1995; Page et al., 1971).

The mechanical properties of fibres can be determined by tensile testing of either individual (single) fibres or fibre bundles. The single fibre tensile tests have been applied on a wide number of natural fibres including flax, hemp, jute, bamboo and coir (Baley, 2002; Martin et al., 2013; Osorio et al., 2011; Pickering et al., 2007; Silva et al., 2000), in which the fibre is usually glued onto a paper

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frame with a fixed test length. The test length varies from 1 mm in the study of Snell et al. (1993) to 50 mm reported in Silva et al. (2000), depending on the fibre structure. The latter method is dry fibre bundle tensile test, which was initially developed for characterisation of synthetic fibres (Chi et al., 1984; Coleman, 1958), and later applied on natural fibres (Trujillo et al., 2014). In most cases, it is not possible to use an extensometer for measuring fibre strain due to the small dimensions involved. For single fibre testing, Defoirdt et al. (2010) have presented a correction method for single fibre tensile tests to determine the real elongation of the fibre when only the registered displacement of the clamps is available. The method is however time consuming because a high number of single fibre tensile tests has to be carried out at various test lengths.

The aim of this work is to study the microstructure and the tensile mechanical properties of technical coir fibres. SEM and X-ray microtomography in SEM (SEM-CT) are used to examine the fibre internal structure and fibre porosity. The mechanical properties of coir fibres are then determined in tensile tests integrated with optical strain mapping. The relation between fibre structure and fibre properties will be discussed based on the results of the above analysis.

2. Experimental

2.1. Coir fibres

Coir fibres used in this research are long coir fibres (fibre length in the range of 200–300 mm) which were provided by Can Tho University – Vietnam. The fibres were mechanically extracted from husk shells of premature and mature coconuts (10–12 months on the plant) with a purely mechanical extraction process, which can keep the fibre as long as possible. There was not any retting or chemical treatment applied before extracting the fibres. The fibres are then soaked in hot water at 70 °C for 2 h, washed with ethanol, rinsed with deionized water and dried in a vacuum oven at 90 °C. These fibres were preserved in a conditioned room at 25 °C and humidity of 50% for later studies.

2.2. Investigation of the fibre microstructure using SEM and SEM-CT

For the analysis of the internal fibre structure and the fibre porosity, both SEM and SEM-CT were used for characterisation on the same fibre samples, and the results from the two methods were compared.

In the first method, SEM images of three different cross-sections were taken for each fibre. With the help of the software Leica QWin, the area of the fibre cross-section and the lumens were determined. These data were also used for the calculation of the fibre porosity based on the ratio between the porous area and the area of the fibre section.

The second method was X-ray tomography scanning of the fibre segments by SEM-CT, with the SkyScan micro-CT attachment for the XL30 SEM. Titanium was used as a target in combination with 30 kV voltage on the electron beam to generate X-rays. The entire fibre segment was scanned, and then a full volumetric image was obtained after reconstructing the scanned images by using the SkyScan NRecon software. With these sets of data, morphological measurement of the fibre in 2D and 3D was carried out with the help of the SkyScan CTanalyser software. In the tomography scanning, in order to obtain high resolution images, the X-ray beam is generated with low power (30 kV) and high current of 120 μA, in combination with long exposure time of 4000 ms.

Regarding the number of test fibres, it should be noted that the extracted technical coir fibres naturally present as such as in the coconut husk. They are quite similar and less variation in terms of fibre structure and fibre properties, as compared to other natural fibres (e.g. flax, jute and bamboo). In this study, ten fibre samples were characterised using the two methods.

2.3. Measurement of the fibre density

Coir fibre is a porous fibre which comprises a high number of lumens in the fibre structure. When the fibres are used in composites, the fibre volume fraction can be calculated by taking into account the whole fibre volume. However, only the solid material of the fibre will carry load during loading of the composite. Consequently, both the density of the whole coir fibre and that of the fibre solid fraction are important for the characterisation of fibre and composite properties. In this experiment, coir fibres were cut to different fibre lengths of 4, 2, 1 and 0.5 mm and also to grinded fibre of approximately 0.05 mm length (considered as solid particles). The samples were weighed to know the mass, and the sample volume was determined using a gas pycnometer (Beckman 930). Accordingly, the density of the samples could be calculated by using the measured mass and volume.

2.4. Single fibre tensile tests

Single technical fibres (which consist of a bundle of structurally bonded elementary fibres) were tested in tension on a mini universal test machine. Because of the small diameter of coir fibres (<0.5 mm), it is practically difficult to measure the fibre strain by using an extensometer, which is usually applicable for tensile testing of larger samples. Therefore, two methods were used to determine the fibre strain in this study.

In the first method, the test was performed on an Instron 5943 integrated with a camera system for optical strain measurement. Speckles were created on the fibre surface so that the camera system could map the fibre strain during tensile loading. It should be noted that the preparation of speckle pattern is very important since the detection and calculation of fibre displacements are based on the speckle pattern. The smaller size and higher density of speckles could provide more effective speckle pattern for the analysis. The recorded strain mapping data were analysed using Limess software and the calculated strains were then linked with the tensile load data to plot the stress–strain curve of the fibres. A 1 kN load cell was used for the test, and the crosshead speed was set at 1 mm/min. It should be noted that the load measurement accuracy of this new Instron machine is quite high (±0.5% of the reading down to 1/500 of the load cell capacity of 1 kN, hence ±10 mN on a total of 2 N) so this provides an accurate measurement even at the low loading forces used. At least 15 fibres were tested in this method.

The second method was based on correction of the fibre slippage and machine compliance. A variety of test span lengths (10, 15, 20, 25, 30 mm) were used for performing the tensile test on a home-made mini tensile machine. For each span length, a minimum of 15 fibres were tested. Based on the obtained data of load and displacement at different span lengths, a theoretical correction (developed by Defoirdt et al., 2010), which is described in the following paragraphs) for the fibre slippage and machine compliance was used to determine the correct strain of the fibre samples. The crosshead speed was set at 1 mm/min and a 200 N load cell was used in this study.

Concerning sample preparation, for both methods, the fibre sample was randomly selected and glued into a paper frame, as shown in Fig. 1. This keeps the fibre as straight as possible and assures a good gripping. Before fixing the fibres in the paper frame, the mass per length was measured for every fibre. The loaded cross-sectional area of the fibre, which was used to convert applied force

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