Composites Science and Technology 82 (2013) 76–83

Contents lists available at SciVerse ScienceDirect

Composites Science and Technology

journal homepage: www.elsevier.com/locate/compscitech

Luffa cylindrica fibres/vinylester matrix composites: Effects of 1,2,4,5-benzenetetracarboxylic dianhydride surface modification of the fibres and aluminum hydroxide addition on the properties of the composites

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ARTICLE INFO

Article history:
Received 18 December 2012
Received in revised form 11 April 2013
Accepted 16 April 2013
Available online 25 April 2013

Keywords:
A. Polymer–matrix composites (PMCs)
B. Thermomechanical properties
C. Failure criterion
D. Dynamic Mechanical Thermal Analysis (DMTA)

ABSTRACT

In this work we used a sequence of organic extraction and chemical modification treatments in order to increase the compatibility between the mat fibrous of the *Luffa cylindrica* ripe fruit and the vinyl ester resin for composites preparation. As observed from SEM and suggested from XRD analyses, these sequential treatments did not degrade the *L. cylindrica* fibres preserving their potential use as a reinforcement. The FTIR analyses and the conductometric titrations showed evidences of interactions between the fibres and the dianhydride. A fibre mass ratio of 15% was used for composites preparation decreasing the density of the composites at ca. 45% when compared with the vinylester matrix. The TG analyses showed that the aluminum hydroxide addition in the matrix increases the thermal stability of the composites. The DMA showed that the fibres did not influence the matrix cure kinetics, and a biphasic system was formed during pre-curing under environmental conditions. The tensile and Charpy impact strength tests showed an increase of the strength of the composites when compared with the matrix. The best results were obtained for PMDA treated fibres/vinyl ester matrix composites, which showed an increase of ca. 30% for tensile strength and 250% for impact strength.

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1. Introduction

In the last years, there was an increased interest on the development of new materials using renewable natural sources as for example lignocellulosic fibres/polymer composites. The major advantages of the lignocellulosic fibres when compared to synthetic ones are the low costs and the density, the high specific properties, the moderated abrasivity during processing, the renewability, the biodegradability, and a great availability [1–6].

The polymer composites reinforced from natural fibres also present advantages such as low weight, reasonable strength and hardness, and important ecological and economic aspects [7,8]. Some lignocellulosic natural fibres/polymer matrix composites have been already described in the literature, as for example, polyester reinforced by Sansevieria cylindrica [9], hemp [10], sisal [11], and curauá [12]; epoxy resin reinforced by banana [13], bamboo [14], henequen [15] and flax [16]; formaldehyde resin reinforced by banana [17] and sugar cane [18], etc. Even if all these fibres are abundant mainly in tropical countries only very few studies have reported the use of fibres derived from fruits as coconut [19,20] and borassus fruit fibres [21,22] or fibres that form a natural fibrous tridimensional network for composites preparation. One recent example of the use of a 3D natural net for composites preparation is the work of Bouakba et al. [23]. These authors used fibres derived from *Opiuntus ficus-indica*, a variety of cactus spread over large parts of Mediterranean and North African countries, in order to prepare cactus fibre/polyester biocomposites.

Luffa cylindrica (Brazilian sponge gourds) is a subtropical plant derived from curcubitacea family, and is commonly found in China, Japan and other countries in Asia, and Central and South America [24]. *L. cylindrica* plant presents some advantages when compared to others tropical plants, as for example, sisal and curauá because its ripe fruit does not form individual fibres but a 3D natural fibrous mat, which present a potential use in order to obtain isotropic composites. Fig. 1 shows the fruit of the *L. cylindrica* plant. The ripe fruit has a thick peel and the sponge gourd, which has a multidirectional array of fibres comprising a natural mat, presents an inner fibre core and an outer mat core. Common sponges vary in length from around 15–25 cm to 1.20–1.50 m. The fibre sheaths are made by a zig-zag-type fabric with random orientation, and

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http://dx.doi.org/10.1016/j.compscitech.2013.04.012

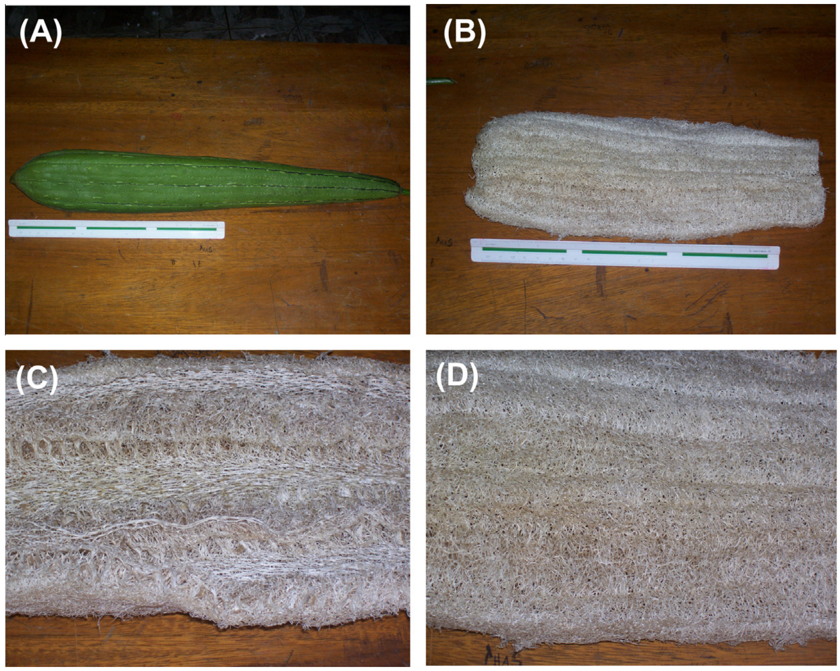

Fig. 1. *L. cylindrica* ripe fruit (A) and its natural mat fibrous (B) used for composite preparing: (C) inner and (D) outer core.

their overall layout of the tree-like fibril structure shows some similarities to the morphology present by the *O. ficus-indica* fibres [23].

The greatest limitation of the use of natural fibres as reinforcement in polymer composites is the low compatibility between hydrophilic natural fibres and hydrophobic polymer matrices. Physical and/or chemical treatments have been proposed in order to improve the compatibility between these interfaces [1,25–27]. In a recent paper published by our research group, we reported the potential use of a surface modification treatment of lignocellulosic fibres using 3,3′,4,4′-benzophenonetetracarboxylic dianhydride (BTDA) [1]. Based on the obtained results, one objective of this work is to optimise this kind of treatment and to evaluate the use of other dianhydride: 1,2,4,5-benzenetetracarboxylic dianhydride (PMDA) in order to modify the fibres surface.

The low resistance at high temperatures of natural fibres also limits their use for polymer composites preparation. In order to increase the thermal stability allowing processing of the composites at high temperatures aluminum hydroxide will be used like a polymer additive.

The vinyl ester resins are the addition product of an epoxy resin and an unsaturated carboxylic acid such as acrylic or methacrylic acids. There exists a great number of possible products depending on both the resin and the acid used, but the most utilised are that derived from a multi-methacrylate oligomer (typical bisphenol A-based), and styrene as a reactive diluent. They present reactive double bonds in their structure originating cross-linked networks from initiation by free radical mechanism. The choice of this resin for preparing a thermosetting matrix is mainly due to its high values of strength, durability, stability, corrosion resistance, and precuring at room temperature which accelerates the processing of the composites. Glass fibres/vinylester resin composites are used in primary structural components as for example: in marine and offshore (hulls, decks, ship, pier and bridge), and civil infrastructure (reinforcement in concrete) [28–31]. Other uses already described in the literature include vinylester composites reinforced by carbon [32] and jute fibres [33].

This paper aims the preparation and the characterisation of a new composite material prepared from a vinylester matrix, and reinforced by modified and unmodified fibrous mat of the *L. cylindrica* ripe fruit as an attempt to replace synthetic fibres, and the use of aluminum hydroxide as a polymer additive in order to enhance the thermal properties and to decrease the mass ratio of resin used. This composite material may be an interesting alternative mainly for Brazil in several applications due to the abundance of this fibre in this tropical country, its easiness of cultivation, high resistance to climatic changes, different soil types and short ripening time.

2. Experimental

2.1. Characterisation of the fibres of the L. cylindrica ripe fruit

The *L. cylindrica* plant used in this study was cultivated in southeast of Brazil. The fibrous mat from *L. cylindrica* ripe fruit was washed with distilled water, dried in an oven at 105 ± 5 °C for 24 h and stored in a desiccator. The characterisations were performed following standard methods (see Table 1). Each result represents an average value of 5 measurements of 5 different samples of *L. cylindrica* fruits.

2.1.1. Organic solvents extracting

The fibrous mat samples from *L. cylindrica* fruit were extracted in ethanol/cyclohexane (1:1, v/v) using a Soxhlet extracting system for 48 h, and dried in an oven at 105 ± 5 °C for 24 h. The organic extracted samples were stored in a desiccator.

2.1.2. NaOH and PMDA modification treatments

The organic extracted samples were treated in 2% NaOH solution under stirring at room temperature (mercerization) for 2 h, and washed in distilled water up neutral pH. After, they were dried in an oven at 105 ± 5 °C for 24 h and stored in a desiccator.

The PMDA surface chemical modification was carried out after the organic extracting and mercerization. The dried fibrous mat samples from *L. cylindrica* fruit were treated in PMDA (Sigma–Aldrich) solution following the procedure: 0.25 g of PMDA and 1.5 mL of triethylamine were added for each 10 g of fibre in

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