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A new eco-friendly chemical treatment of natural fibres: Effect of sodium bicarbonate on properties of sisal fibre and its epoxy composites

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ABSTRACT

Several researchers have shown how sisal fibres possess remarkable tensile properties that yield them good candidates as reinforcement in biocomposite materials.

This work aims to evaluate the effect of an eco-friendly and cost effective surface treatment method based on the use of commercial sodium bicarbonate (i.e. baking soda) on properties of sisal fibre and its epoxy composites. In particular, raw sisal fibres were treated with a 10%w/w of sodium bicarbonate solution for different periods (24, 120 and 240 h), at room temperature. Changes occurring in sisal fibres were characterized through scanning electron microscope, Fourier transform infrared spectroscopy, thermogravimetric analysis and helium pycnometer analysis.

The mechanical characterization of sisal fibre was carried out through single fibre tensile tests and a reliability analysis of the experimental data was performed. A mathematical model was also applied to investigate the relation between the transverse dimension of the fibres and their tensile properties. Interfacial adhesion of sisal fibre with an epoxy matrix was investigated using single fibre pull out technique. Moreover, to deeper investigate the effect of the proposed treatment, epoxy based composites reinforced with short randomly oriented sisal fibres were manufactured and characterized by means of quasi-static flexural tests.

The experimental results showed that 120 h is the optimum time for treating sisal fibre to achieve highest interfacial adhesion and mechanical properties with epoxy matrix.

1. Introduction

Over the last decades, natural fibres have received increasing attention as alternative to synthetic ones both from academic world and from various industries. There is a wide variety of different natural fibres, which can be applied as reinforcements of polymer (i.e. thermoplastics and thermosets) based composites.

Generally four main reasons make the natural fibres attractive: i.e. their specific properties, their price, their biodegradability and their recyclability. Particularly, their low density and high specific properties are nice benefit. Furthermore, they are renewable and have a CO_2-neutral life cycle, in contrast to their synthetic competitors (i.e. glass and carbon).

All natural fibres consist of long cells with relatively thick cell walls, which make them stiff and strong. In most plants, the cells are glued together into long thin fibres and their length on the plant length. The fibres may differ in coarseness, in the length of the cells and in the strength and stiffness of the cell walls [1,2]. The most common natural fibres are flax, hemp, jute, kenaf and sisal, due to their properties and availability. Furthermore, some scientific works analysed the feasibility of using less common natural fibres in the last decade (e.g. okra [3], isora [4], artichoke [5], arundo [6–8], ferula [9], althea [10], sansevieria [11] and buriti [12]).

Sisal fibre is one of the most widely used natural fibre in yarns, ropes, twines, cords, rugs, carpets, mattresses, mats and hand-crafted articles [13]. It is a hard fibre extracted from the leaves of the sisal plant (*Agave sisalana*) in the form of long fibre bundle. Nearly 4.5 million tons of sisal fibres are produced every year throughout the world, particularly in Tanzania and Brazil. A sisal plant produces about 200–250 leaves and each leaf contains

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1000 ± 1200 fibre bundles which are composed of 4% fibre, 0.75% cuticle, 8% dry matter and 87.25% water [14]. A leaf weighing about 600 g will yield about 3% by weight of fibre, with each leaf containing about 1000 fibres [15].

Similarly to all natural fibres, sisal one also possess some drawbacks such as high hydrophilicity with consequent high moisture absorption. Moreover, it is characterized by high variability of the properties that are strongly harvest-dependent, influenced by climate, location, soil characteristics, weather circumstances, fibre processing and their incorporation into composites.

The main drawback in use of natural fibres as reinforcement of hydrophobic polymeric matrices is the poor compatibility between fibres and matrix. These features result in a low ability to transfer stress from matrix to fibre in addition to dimensional changes of fibres that may lead to microcracking of composites, thus reducing their mechanical properties [16].

To improve the fibre-matrix adhesion, fibre surface modification is commonly carried out by physical or chemical methods both leading to reduction in moisture gain, as well as changes in the fibre surface.

Physical treatments (e.g. corona treatment [17,18], plasma treatments [19–21] and electron beam irradiation [22]) alter the surface properties of the natural fibres enhancing the mechanical bonding between fibre and matrix, with no alteration of the chemical composition of the fibres.

On the other hand, chemical modifications (such as silane, alkalization or mercerization and acetylation treatments) of natural fibres increase the fibre-matrix compatibility by introducing a third material [23].

A deep research on a cost effective treatment is needed because one of the main attractions of the market of biocomposites is the competitive price of the natural fibres. To this regard, it is worth noting that all the chemical treatments performed until now are more or less expensive and/or harmful to environment. Large amounts of hazardous chemicals are usually involved and chemical waste must be appropriately handled and disposed of. Due to these limitations the use of chemical treatments becomes less attractive.

Moreover, the improvement of the fibre-matrix by means of chemical treatments is usually obtained at the expense of tensile properties of the fibres. This decrease may be due to substantial delignification and degradation of cellulose chains during treatment, which has usually a lesser effect on the extension at break of these fibres [24,25].

Kabir et al. [26] studied the influence of alkaline, acetyl and silane treatments on the tensile properties of hemp natural fibres. The experimental results showed that the tensile strength of the chemically treated fibres decreases compared to untreated fibres. In particular, alkalization reduces the fibre strength, additional acetylation could recover some of the strength loss and silane treatment leads to an increase of the failure strain of the fibres.

Alsaeed et al. [27] used different concentrations (i.e. 3%, 6% and 9%) of NaOH solution to pre-treat date palm fibres for 24 h at room temperature, showing that the increase in NaOH concentration worsens the tensile properties of the natural fibre. On the other hand, it was found that higher concentration of NaOH enhances the surface characteristics of the fibre by removing the waxy layer from the surface and the fibre-matrix interfacial adhesion. Hence, the authors stated that 6% NaOH is the optimum concentration which provides acceptable fibre strength and surface characteristics.

At the best of our knowledge, a simple and eco-friendly treatment based on the use of an aqueous solution of sodium bicarbonate, without adding any other compounds, has not yet been carried out to improve the properties of natural fibres and their adhesion to polymers. However, sodium bicarbonate was already used as addition to a chromium sulfate solution to treat okra fibres [28] and coir fibres [29,30].

This work aims to investigate the effect of this new, eco-friendly and cost effective treatment based on sodium bicarbonate solution on the properties of sisal fibre and its epoxy composites. For this purpose, the morphology and chemical composition of sisal fibre were investigated through scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. Moreover, the mechanical characterization of sisal fibre was carried out through single fibre tensile tests, analyzing the results through statistical analysis. Finally, in order to investigate the effect of the proposed treatment on the interfacial adhesion of sisal fibre with an epoxy matrix, single fibre pull out tests were performed.

Finally, to deeper investigate the effect of the proposed treatment on the properties of sisal/epoxy composites, single fibre pull-out and quasi-static flexural tests were carried out.

2. Material and methods

Sisal plants were collected in a plantation of northern Sicily and the extracted fibres were hand-washed and dried at room temperature for 48 h.

Portions of the cleaned fibres were soaked in 10 wt% $NaHCO_3$ solution for 24 h, 120 h and 240 h at room temperature, then washed with distilled water and dried in an oven at 40 °C for 24 h. The sisal fibres investigated will be identified with the sample codes RAW, T-24 h, T-120 h and T-240 h, respectively.

As well known, an aqueous solution of sodium bicarbonate is mildly alkaline due to the formation of carbonic acid and hydroxide ion:

$$NaHCO_3 + H_2O \rightarrow Na^+ + HCO_3^- \quad (1)$$

$$HCO_3^- + H_2O \rightleftarrows H_2CO_3 + OH^- \quad (2)$$

Considering that the OH groups present in the fibres correspond mostly to the alcoholic hydroxyls (weak acids), it can be proposed that the interaction is similar to that which happens during a traditional mercerization treatment [25,31,32].

$$\text{Fibre-OH} + \text{Na OH} \rightarrow \text{Fibre-O}^-\text{Na}^+ + H_2O \quad (3)$$

To perform pull-out tests, an epoxy resin system obtained mixing a diglycil ether of bisphenol-A (DGEBA) epoxy monomer (SX 10, supplied by Mates Italiana Srl) with its own amine based curing agent (100:30 mix ratio by volume) was used. To carry out the quasi-static flexural tests, composites with fibre weight fraction equal to 5% (1 cm long randomly oriented fibres) were manufactured by using the above epoxy system as matrix, for each treatment condition.

2.1. Density measurement

The density of sisal fibres was measured using gas intrusion under helium gas flow with a Pycnomatic ATC Thermo Electron Corporation equipment pycnometer. For each sample, ten measures were carried out and average values of density were recorded.

2.2. Fibre tensile test

A population of 50 samples for each condition was prepared and tested in tension, according to ASTM standard [33] using an UTM by Zwick-Roell model Z005, equipped with a load cell of 200 N. The strain rate was set equal to 2.5 mm/min and gage length to 30 mm.