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Flexural properties loss of unidirectional epoxy/fique composites immersed in water and alkaline medium for construction application

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ABSTRACT

Epoxy fique composites were evaluated for construction applications and compared with conventional wood used in construction. The composites studied were made with fique fibers treated using Na(OH) solution at 18 w/v%, untreated fique fibers were also used. The matrices were epoxy and epoxy with 5 wt.% of chemically modified C30B montmorillonite. Unidirectional composites of 90 mm × 20 mm × 4 mm were elaborated by pultrusion processing technique. The flexural properties loss occurred over 20 days of composites submitted to three types of environments: (i) water, (ii) saturated calcium hydroxide solution and (iii) mortar with w/c ratio of 0.45 and 540 kg/m^3 of cement, cured in a saturated solution of lime stone at 50 °C. Results showed that fiber treatment and montmorillonite addition improved the flexural modulus and strength of composites in 40% and 34% respectively. Moreover the flexural properties of composites before and after ageing resulted comparable or even better than conventional wood used in construction.

1. Introduction

Applications of natural fiber composites (NFCs) in construction are being investigated as a result of the increasing demand for environmentally friendly materials in construction. Applications like roof for housing [1–3], structural panels, unit beams [2,3], and door frames [4] have been investigated. Furthermore there are other applications in construction in which NFC could be a cheaper and environmentally friendly option. For instance NFC could be used to manufacture geocells used in treatments to control erosion of soil, or also in the manufacture of geotextiles used to separate, filter, reinforce, protect, and drain the soil. The knowledge of mechanical properties of NFC and their mechanical properties after exposition to both alkaline and humidity environmental or even mortar environmental is crucial for the acceptability of the NFC in construction applications. Since natural fibers may deteriorate in the highly alkaline environment of the cement matrix because dissolution of hemicellulose and lignin, affects the mechanical properties of composites [5,6].

Until now the researches about NFC, in construction [1–4,7], are focused principally on their processing and mechanical response, but their mechanical properties after exposition to alkaline or mortar environmental has not been studied. The use of polymeric matrix as a binder around the natural fibers provides protection to them. However, if the interface of composites is not good, or the matrix is not alkaline resistant, the composite will deteriorate. When the molecules of H_2O or $Ca(OH)_2$ enter in the NFC the following things could occur: (i) decrease in the mechanical properties of the matrix because, chains polymer hydrolysis, network swelling, relaxation and formation of hydrogen bonds between water and polymer molecules [8]. (ii) Deterioration of the interface, because H_2O or $Ca(OH)_2$ could migrate at the interface. In the case of calcium hydroxide, the growth of its crystals in the voids of the interface may cause a severe damage to the composite [9]. (iii) Fiber swelling and embrittlement by chemical and biological attack [10].

To improve their mechanical properties of NFC before and after exposition to humidity and alkaline environmentals, both modification of natural fibers and modification of matrix can be carried out. Alkaline treatment of natural fibers appears as low cost alternative compared with other commonly used treatments such as: acetylation or silanization. Alkaline treatment results in bleaching of the fiber removing impurities, waxy materials, lignin and hemicellulose [10–12]. Regards to matrix modification, homogeneous dispersion and exfoliation of montmorillonite comes out as an attractive alternative. It has been reported that addition of montmorillonite reduces the coefficient of moisture diffusion, increases their mechanical properties, and improves significantly the mechanical strength [13,14].

Unidirectional composites with the matrix and the fiber modified and unmodified were elaborated by pultrusion processing

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technique. The flexural properties loss was measured after 20 days of ageing of composites submitted to three types of environments: composites in distilled water (pH = 6.0); composites in alkaline solution (pH = 12.0), to simulate the pore solution of cement mortar; composites included in cement mortar, cured in a lime stone saturated solution.

2. Materials and methods

This investigation uses Colombian fique fibers, extracted from the leaves of the fique plant *Furcraea Andina*, which is the most cultivated natural fiber plant of Colombia. Fique fibers were washed and dried before alkaline treatment to eliminate impurities and waxes of its surface.

The epoxy resin used as matrix (**E**) was diglycidyl ether of bisphenol-A (DGEBA) with n = 0.15 and the hardener is triethylenetetramine (TETA) from Hunstman and provided by Distraltec S.A. Argentina. The montmorillonite Cloisite 30B (**C30B**) was kindly provided by Southern Clay Products, Inc, USA. The Cloisite 30B is a group of mineral silicates which have been chemically modified with a quaternary ammonium salt (methyl tallow bis-2-hydroxyethyl).

2.1. Modification of fibers

The untreated fique fibers previously washed were treated with 18 w/v% NaOH solution at room temperature for 2 h, using a liquor ratio (fiber/solution w/v) of ¼ (**FQ18**). After the alkaline treatment, fibers were rinsed with distilled water until neutral pH. Both untreated (**FQ**) and treated (**FQ18**) fibers were dried in an oven overnight prior to composite manufacture.

2.2. Modification of matrix

The unmodified matrix (**E**), was elaborated with the prepolymer DGEBA previously mixed with 12 wt.% of TETA. The modified matrix (**EC30B**) was prepared by mixing the prepolymer DGEBA with 5 wt.% of C30B for 30 min by means of a mechanical stirrer, followed for 30 min of sonication. Finally, the TETA hardener was mixed for 5 min by means of a mechanical stirrer.

2.3. Manufacturing of composites

Unidirectional composites were elaborated by manual pultrusion with around 40 wt.% of fique fibers. To elaborate the composite, the fibers proceeded through a bath, where they were impregnated with the matrix. Then the matrix-impregnated fibers were preformed to the shape of the profile. After that, the composite material was passed through a steel die that was machined precisely to the final shape of the composite manufactured. Finally the composites were submitted to following curing cycle: 21 h at 80 °C, then 15 h at 120 °C and finally 3 h at 140 °C. Samples with dimensions 90 mm × 20 mm × 4 mm were cut from the pultruded plaques to develop the mechanical tests and water absorption. Composites were identified using the name of the matrix used followed by the name of the fiber used.

3. Methods

3.1. Attenuated total reflection Fourier transforms infrared spectroscopy (FTIR-ATR)

Infrared spectroscopy experiments were developed using a FTIR spectrometer Nicolet 6700 Series equipped with a single-reflection Attenuated Total Reflectance (ATR) accessory. A type IIA diamond crystal mounted in tungsten carbide of approximately 0.5 mm^2 sampling area was used. Infrared spectra were collected at 4 cm^{-1} resolution using 64 scans.

3.2. Thermogravimetrical analysis (TGA)

Thermogravimetric analysis was developed using N_2 atmosphere at a heating rate of 10 °C/min, using around 14 mg of sample.

3.3. X-ray diffraction (DRX)

Powder X-ray diffractometry were carried out. DRX patterns were recorded by a SIEMENS D5000 diffractometer equipped with an X-ray generator operating with a curve graphite filter at radiation wavelength (λ = 1.542 Å).

3.4. Field emission scanning electronic microscopy (FE-SEM)

Specimen surfaces were coated with a 30 nm gold layer and observed in a Field emission scanning electronic microscopy Zeiss Supra 40. Fiber and composite morphology were analyzed using FE-SEM operated in secondary electron mode under an accelerating voltage of 3 kV. The FE-SEM was performed on the polished and fractured surfaces of the composites.

Before FE-SEM analysis, surfaces of composites were studied using an optical microscope (Olympus PM63).

3.5. Energy dispersive X-ray spectrometer (EDX)

Energy dispersive X-ray spectrometer (EDX) attached to the SEM operated at 20 kV was used to characterize the diffusion of calcium hydroxide molecules in samples. All the composites were polished before the EDX tests.

3.6. Mechanical properties

Tensile tests of fique fibers were performed according to ASTM D3379 specification with a loading rate of 2 mm/min, using an Instron 4204 universal machine. About 20 fibers of 30 mm in length were tested. All tests were carried out at 25 °C.

Flexural tests of composites were performed in a Baldwin universal machine in three-point bending configuration of flexural stress, with 74 mm of support spam following the standard ASTM D790 M-93, Eq. (1) shows the bending equation. The specimen measures were: 90 mm × 20 mm × 4 mm and the crosshead speed was chosen in accordance with the standard.

$$\sigma = \frac{3PL}{2bd^2} \tag{1}$$

where σ is the stress in the outer fibers at midpoint; P is the load at a given point on the load–deflection curve L is the support spam; b is the width of the sample and d is the depth of the sample.

3.7. Flexural properties loss

The flexural properties loss occurred over 20 days of specimens stored at room temperature in three environments: (i) distilled water (pH = 6.0), (ii) saturated solution of $Ca(OH)_2$ (pH = 12.0) and (iii) cement mortar, cured in a lime stone saturated solution. The solutions were covered and the pH of the solutions was checked at regular intervals, solutions which had not retained the initial pH value were replaced. Changes in composites weight were followed over the time. After 20 days of ageing, samples were