Materials and Design 51 (2013) 780-788

Contents lists available at SciVerse ScienceDirect

Materials and Design

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

Dynamic mechanical thermal behavior analysis of doum fibers reinforced polypropylene composites

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ARTICLE INFO

Article history: Received 7 February 2013 Accepted 27 April 2013 Available online 9 May 2013

Keywords: Natural fibers Composites Dynamic mechanical thermal analysis

ABSTRACT

Doum palm fibers are an environmentally friendly reinforcement in polymer composites. Their mechanical properties and abundance allow its use as an innovative material composite. In this paper doum fibers were alkali treated to clean their surface and enhance polymer fibers interaction. Tensile and rheological properties were investigated to see the effect of fibers content on the composites properties. Also, comparative composites were processed to assure a good wettability between fibers and the polymer with the use of a coupling agent as styrene–(ethylene–butene)–styrene three-block copolymer grafted with maleic anhydride (SEBS-g-MA). Results shows that tensile properties was enhanced when fiber were added to the polymer and has enhanced more with the use of coupling agent. A gain of 70% and 77% in the Young's modulus at 30 wt.% fibers content for the binary and ternary composites, respectively. And a gain of 18% in tensile strength at 10 wt.% fibers content for the ternary composites. Moreover, dynamic mechanical thermal analyses were carried out in order to compare the changes of the properties with frequency, fibers loading, temperature and compatibilizer.

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1. Introduction

Markets are increasingly oriented towards on environmental and the friendly products. There is a growing interest in the research to understand the structure and properties of natural resources as natural fibers [1-4]. The use of natural fibers as reinforcement in composite materials is justified as a way to exploit a local resource in industry [2,3]. Natural reinforcements have many advantages over mineral reinforcements due to the natural alignment of the carbon-carbon bonds within the structure of these materials, also this structure provided a significant strength and stiffness [5], low density, low cost, biodegradability, acceptable specific properties, better structure insulating properties, and low energy consumption during their growth or processing [4-6]. Theirs structure rich in cellulose make them easily biodegradable and recyclable [1]. The mechanical performance of natural fibers, has led researchers to develop techniques for the extraction and modification of cellulose (the main component of natural fibers) from such fibers [7].

Polymer composites based on natural fibers are an inexpensive material with minimal impact on the environment. However, the problem with the use of natural fibers is their low adhesion with the polymeric matrix [8–10]. Natural fibers are hydrophilic and possess polar functional groups, while most of the thermoplastic polymers (matrix) are hydrophobic and have non-polar functional groups. Many researchers have shown that the interaction between the thermoplastic matrix and the load distribution within the matrix are key factors to obtain materials with good properties [11]. In order to circumvent this problem and improve the affinity between the fibers and polymer matrix, use can be made of either chemical or physical modifications on the surfaces of fibers and/or the matrix [7]. In addition, cellulose, presents the advantage of having reactive sites that may interact with the polymer matrix in the presence of a coupling agent [3]. So, the use of coupling agents as a bridge between fibers and matrix can be beneficial in improving wettability of fibers with polymer chains [12]. Many varieties of natural fibers exist for the reinforcing function.

In this study, the Moroccan doum palm was used as fiber reinforcement in thermoplastic matrix, it was alkali treated to remove its non-cellulosic component and is compound using a twin screw extruder in a polypropylene matrix grafted with 8 wt.% of styrene–(ethylene–butene)–styrene three block copolymer grafted with maleic anhydride (SEBS-g-MA) as a compatibilizer. The effects of coupling agent and fibers loading on the tensile, and thermal properties such as, Young's modulus, tensile strength, complex modulus and loss factor of the composites, are investigated. Composites with 5, 10, 15, 20, 25 and 30 wt.%, will be extruded and used







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^{0261-3069/\$ -} see front matter \circledcirc 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.matdes.2013.04.092

in an injection molding machine to prepare dumbbells samples for conventional tests.

2. Materials and methods

2.1. Materials

The used Polypropylene as matrix, was supplied by ExxonMobil Chemical, it has a density of 0.9 g/cm³, and melting temperature of 165 °C. Raw doum fibers (Chamaerops humilis) with an average length of 25 cm were collected from rural areas of Morocco. And before further use fibers were grinded in a precision grinder (FRIT-SCH Pulverisette 19) equipped with a 1 mm sieve size, Fig. 1a, shows the length and width distribution of the grinded fibers and Fig. 1b shows the morphology of the doum fiber. Chemicals used in this study to treat doum fibers were sodium hydroxide (NaOH, 98%) from Sigma-Aldrich (France) and Acetic acid (CH₃₋ COOH, 99-100%) which is from Riedel-de Haën. The used coupling agent has been supplied by Shell and is named Styrene-(ethylenebutene)-styrene triblock copolymer grafted with 1.4 to 2 wt.% maleic anhydride (SEBS-g-MA) (Kraton FG-1901X).

daverage = 128µn (b)

2.2. Treatment of doum fibers

To enhance fibers/matrix surface adhesion, whole fibers down were subjected to an alkali treatment to clean their surface [13]. Crushed fibers are first washed thoroughly with water then kept for 48 h in a 1.6 mol/l sodium hydroxide aqueous solution [14]. The fibers are then removed from the caustic solution and treated with acetic acid (100 ml) to neutralize the remaining caustic [15], after which they were air dried for 24 h. This treatment removes a certain amount of lignin, waxes and oils that covers the external surface of fibers wall [16].

2.3. Fabrication of composites and test specimens

Polymer composites reinforced with various amounts of doum fibers were prepared by compounding neat polypropylene and the alkali treated doum fibers at various concentrations (5, 10, 15, 20, 25 and 30 wt.%) using a co-rotating twin screw extruder machine (model ZSE-18) equipped with 7 heating zones. The extruder screws were configured to ensure a better dispersion-distribution of the doum fibers within the polypropylene matrix. The temperature profile in the various zones along the extruder was set at 200, 200, 200,200, 180,180,180, and 180 °C [17-19] from the hopper to the die. The co-rotating screws were operating at 125 rpm (rpm) while a side feeder screw for feeding fibers was set at 40 rpm.

The strands coming out of the extruder were cooled in a water bath and pelletized (Thermo Fisher, Stone, UK) into pieces of 2-3 mm length [20]. All specimens for tensile and rheological tests were molded using an Engel e-Victory injection molding machine. The temperature was set at 200, 200, 200 and 180 from the barrel to the nozzle while the temperature mold was set at 45 °C [20].

3. Characterization

3.1. Structural characterization

3.1.1. X-ray diffraction analysis

X-ray diffraction (XRD) characterization of doum fibers was carried out on Bruker D8 Discover, using the Cu Ka radiation $(\lambda = 1.54184 \text{ nm})$, and a GADDS detector. The scanned angular range (2θ) was from 1° to 36° at a step size of 0.02°, at a working voltage and current of 45 kV and 100 mA, respectively.

The use of XRD counts offers an easy way to evaluate the crystalline index of fibers, which calculates from Eq. (1) [17]:

$$I_{\rm XRD} = (I_{002} - I_{\rm amp})/I_{002} \tag{1}$$

where I_{XRD} is the crystalline index; I_{002} is the maximum intensity of the 002 lattice diffraction plane at a 2θ angle between 22° and 23° ; $I_{\rm amp}$ is the intensity diffraction at an angle 2θ close to 18° representing amorphous materials in plant fibers.

3.1.2. ATR-FTIR analysis

Fourier Transform-Infrared spectra were recorded on an ABB Bomem FTLA 2000-102 spectrometer (ATR: SPECAC GOLDEN GATE). The spectra were obtained with an accumulation of 16 scans and with a resolution of 4 cm^{-1} .

3.2. Thermal stability

The thermal stability of the neat Polypropylene, fibers and composites were evaluated by thermogravimetric analysis (TGA) using a Q500 instrument from TA Instruments. Roughly 10 mg of each sample was placed in a platinum pan and heated under air from room temperature to 600 °C at a heating rate of 10 °C/min to yield

Fig. 1. (a) Doum fiber distribution; and (b) doum fiber morphology.



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