Composites Part B 75 (2015) 367–372

Contents lists available at ScienceDirect

Composites Part B

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

Mechanical, thermal and morphological characterization of cellulose fiber-reinforced phenolic foams

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ARTICLE INFO

Article history:
Received 13 October 2014
Received in revised form
24 December 2014
Accepted 29 January 2015
Available online 7 February 2015

Keywords:
A. Foams
A. Polymer–matrix composites (PMCs)
B. Mechanical properties
B. Thermal properties

ABSTRACT

In this work, the compressive mechanical properties, thermal stability and morphology of cellulose fiber-reinforced phenolic foams were studied. The cellulose fiber-reinforced phenolic foam showed the greatest compressive mechanical properties by incorporating 2 wt.% of the reinforcement. The compressive modulus and strength of 2 wt.% cellulose fiber-reinforced phenolic foam were increased by 21% and 18%, respectively, relative to the unreinforced material. The addition of the cellulose fibers to the phenolic foam slightly decreased the thermal stability of the material. The study on the morphology of the cellulose-reinforced phenolic foams via Scanning electron microscopy (SEM) indicated a strong bonding between the fibers and phenolic matrix. In addition, the incorporation of the cellulose fibers into the foam resulted in a decreased cell size and increased cell density of the material. The incorporation of 2 wt.% of cellulose fibers into the phenolic foam led to obtain the material with the best features.

1. Introduction

The use of phenolic foams for insulating applications has grown in the last few decades due to these materials show advantageous features compared to the other commercial polymeric foams. Phenolic foams are thermally stable in a broad range of temperature and exhibit excellent fire-resistance properties (low flammability, no dripping combustion and low smoke density) [1,2]. In contrast, these polymeric foams show relatively low mechanical performance and high brittleness. The incorporation of modifiers, inert filler and synthetic fibers into the phenolic foams are some of the investigated techniques to solve the mechanical deficiencies of this material [3].

In the last decade, the incorporation of reinforcements from natural resources such as jute, sisal, hemp, kenaf and wood fibers into polymeric materials to improve their mechanical performance has been widely studied [4]. Concerning phenolic foams, in previous work we incorporated wood flour and lignin nanoparticle into a phenolic foam to increase the compressive mechanical properties of the material [5,6]. However, the incorporation of alternative reinforcements obtained from natural resources such as the cellulose fibers into phenolic foams has not been reported yet. Cellulose is a macromolecule constituted by nonbranched chains of 1-4-linked-d-anhydro glucopyranose units and can be produced industrially as man-made and as regenerated fibers from natural resources [7,8]. The regenerated cellulose fibers are obtained from viscose and NMMO (N-methylmorpholine N-oxide) processes and show some advantages compared to other cellulose fibers such as high purities, good reproducibility of results and few defects [7]. Several works have demonstrated the suitability of cellulose fibers to improve mechanical properties of polymer composites. Bledzki et al. [9] obtained increased tensile and flexion mechanical properties by incorporating cellulose fibers into a polyoxymethylene matrix. Pöllänen et al. [10] found that the addition of cellulose fibers to a high density polyethylene matrix resulted in increased tensile mechanical properties of the material. Rojo et al. [7] reinforced a phenolic matrix with regenerated cellulose fibers. Regarding polymeric foams, Wan et al. [11] obtained increased tensile mechanical properties by incorporating cellulose nanofibers into a starch foam. Miléo et al. found that the addition of cellulose fibers from sugarcane straw to a polyurethane foam increased its flexural mechanical properties [12].

The present work aims to incorporate viscose cellulose fibers into a phenolic foam to improve its compressive mechanical properties. In addition, the effect of cellulose fibers on the thermal stability of the reinforced foams was studied via thermogravimetric

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http://dx.doi.org/10.1016/j.compositesb.2015.01.049
1359-8368/

analysis (TGA). Finally, the morphology of the foams was observed via Scanning electron microscopy (SEM) to study the effect of cellulose fibers on the cell size and density of the material.

2. Experimental

2.1. Materials and foam synthesis

The cellulose fibers incorporated into the phenolic foams were viscose fibers derived from *Eucalyptus* wood provided by Sniace with a length of 1.7–38 mm. The fibers were milled in a mill Retsch MM301 to a length of 100–1000 μm in order to obtain a great dispersion of the fibers in the material formulation mixture. The phenolic resol resin and hardener (ACE 1035) employed to prepare the foams were supplied by Momentive Specialty Chemicals. The surfactant (Tween® 40) and the catalyst (phenol-4-sulfonic acid) used for foam formulation were both supplied by Sigma Aldrich. The blowing agent (n-pentane) was provided by Panreac. The synthesis of the foams was carried out according to previous work [5,6]. All the studied foams were formulated with an apparent density of 160 kg/m^3, which is suitable for insulating and structural applications. The cellulose fibers weight fractions incorporated into the material were 2, 4, 6 and 8 wt.% based on the mass of the resin.

2.2. Compression tests

Compression tests of the foams were performed according to ASTM D1621 standard using a universal testing machine Zwick/Roell Z030 [13]. The samples were cut with a keyhole saw Bosh PST 900 PEL and polished using a polished Buehler MetaServ® 3000 to obtain cubic specimens of 25.4 mm of side. The specimens were placed between stainless steel plates and a load with a crosshead rate of 2.5 mm/min was applied to obtain stress–strain curves. The compressive modulus (E) and compressive strength (σ_c) were determined from the slope of the lineal step of the strain-stress curve and the maximum strength of the curve for strain $\leq$10%, respectively. A minimum of five specimens were tested.

2.3. Thermogravimetrc analysis (TGA)

Thermogravimetric analysis was performed using a Mettler Toledo TGA/DSC1 TG. The samples were placed in an alumina crucible of 70 μL and heated at 10 °C/min over a temperature range of 30–900 °C. The experiments were performed under a nitrogen atmosphere at a flow of 20 mL/min. The mass of foam and cellulose fibers analyzed were 5 ± 0.5 mg and 10 ± 0.5 mg, respectively. The obtained thermograms lead to determine $T_{10\%}$, $T_{25\%}$ and $Ash_{900°C}$ of the materials. $T_{10\%}$ and $T_{25\%}$ were determined as the temperature at which the foams showed a 10% and 25% of mass loss in the step of thermal degradation, respectively. $Ash_{900°C}$ was obtained as the remaining mass (wt.%). All the thermal parameters were calculated in dry base, i.e. considering the initial mass of the material as its mass at 135 °C. A minimum of three samples were tested.

2.4. Scanning electron microscopy (SEM)

The morphology of the materials was observed via SEM. The samples of the foams were introduced in liquid nitrogen and fractured. The fracture surfaces were subjected to a gold sputtering and observed using a JEOL JM-6400 microscope operating at 40 kV. The foams cell diameter was determined by visual inspection from the images obtained via SEM. The foams' cell density (N_F, cm^3) was determined assuming the materials showed an isotropic distribution of spherical cells as follows [14,15]:

$$N_F = \left(\frac{n}{A}\right)^{3/2} \quad (1)$$

where n is the number of bubbles in the micrograph of area A, in cm^2. A was 0.0365 cm^2.

3. Results and discussion

3.1. Compressive mechanical properties of reinforced phenolic foams

The compressive modulus and strength of the unreinforced (PF) and cellulose fiber-reinforced phenolic foams (CRPFs) are shown in Fig. 1. Compressive modulus and strength of the phenolic foam increased by incorporating 2 wt.% of cellulose fibers into the material and decreased by incorporating higher reinforcement weight fractions. Therefore, the greatest mechanical properties were obtained for the 2 wt.% reinforced phenolic foam. The compressive modulus for PF was 56.46 MPa and for 2% CRPF 68.26 MPa, an increase of 21% compared to PF. The compressive strength for the PF was 1.629 MPa and for 2% CRPF 1.920 MPa thus, the strength for the 2 wt.% CRPF increased by 18% relative to PF. Ribeiro da Silva et al. [16] obtained an increase in mechanical properties of a polyurethane foam by incorporating 1 wt.% of rice husk ash into the material. However, the addition of higher amounts of the rice husk ash to the foam resulted in decreased mechanical properties of the material. This fact was due to a worse disposition of the cells in the foam structure caused by the presence of the rice husk ash.

The compressive mechanical properties for several reinforced-phenolic foams reported and the results obtained in this study are summarized in Table 1. The increases in compressive

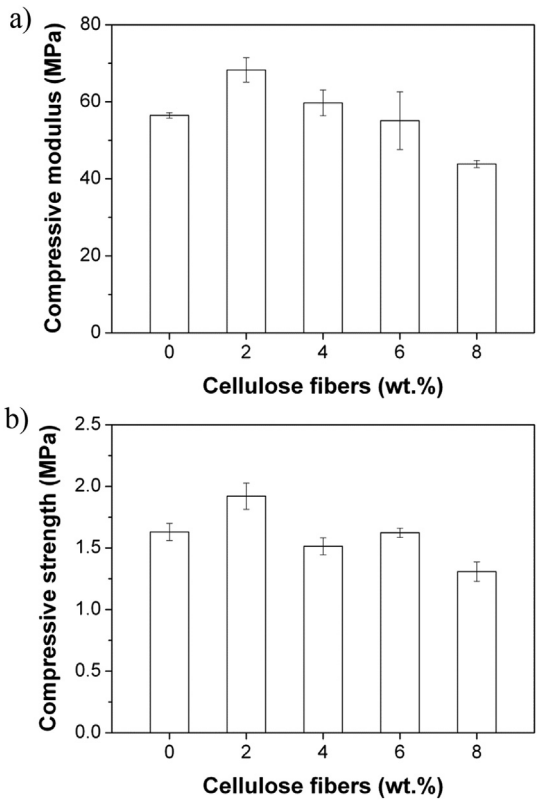

Fig. 1. Compressive mechanical properties for PF and CRPFs with 2, 4, 6 and 8 wt.% of cellulose fibers: a) Modulus (MPa) and b) Strength (MPa).