Materials and Design 57 (2014) 660–666

Contents lists available at ScienceDirect

Materials and Design

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

The influence of wood flour particle size and content on the rheological, physical, mechanical and morphological properties of EVA/wood cellular composites

Matheus V.G. Zimmermann [a], Taís C. Turella [a], Ruth M.C. Santana [b], Ademir J. Zattera [a,*]

[a] Program of Postgraduate Studies in Process and Technology Engineering (PGEPROTEC), University of Caxias do Sul, Rua Francisco Getúlio Vargas, 1130, 95070-560 Caxias do Sul, RS, Brazil
[b] Program of Postgraduate Studies in Mining, Metals and Materials Engineering (PPGE3M), Federal University of Rio Grande do Sul, Av. Bento Gonçalves, 9500, 91501-970 Porto Alegre, RS, Brazil

ARTICLE INFO

Article history:
Received 20 February 2013
Accepted 2 January 2014
Available online 10 January 2014

Keywords:
Cellular composite
Poly(ethylene-co-vinyl acetate)
Wood flour
Particle size

ABSTRACT

Cellular composites reinforced with vegetal fibers are an emerging class of materials combining good mechanical properties with reduced density and superior impact energy absorption, as well as thermal and acoustic isolation compared to other composites. This research aims to investigate the effects of different particle sizes and contents of wood flour (WF) on the properties of cellular poly(ethylene-co-vinyl acetate) (EVA)/WF composites. The cellular composites were foamed in a heat press using azodicarbonamide as blowing agent. The results indicate that decreasing the particle size of WF increases the viscosity of the composite, which restricts the expandability of the composite. The presence of WF in the cellular composite increases the nucleation of cells, providing a larger number of smaller cells with increased filler content. Optimal homogeneity was observed with WF B (100–150 mesh), but the highest mechanical properties of tear strength were observed with WF C (150–270 mesh).

1. Introduction

Wood plastic composites (WPC) represent an emerging class of materials that combine the favorable performance and cost attributes of both wood and thermoplastics [1]. These composites have been gaining increasing degrees of acceptance because of their favorable properties, such as lower cost, improved stiffness, lower density, lower abrasiveness and better processability compared to other fillers, such as inorganic fibers [2].

Although WF is gaining increasing acceptance as a reinforcement filler due to its significantly properties, the applicability of WPC is limited due to their lower ductility, reduced impact resistance, and higher density in comparison with those unfilled thermoplastics and natural wood [3]. Reducing the weight of WPC is another challenge for these materials. The density of WPC is almost two times that of solid lumber.

The central idea behind producing a cellular composite is to create a large number of bubbles or voids from a single-phase composite-gas mixture by introducing a blowing (foaming) agent during processing. The concept of creating cellular foamed structures has been shown greatly to reduce the weight, improve the impact resistance, and improve the thermal and acoustic insulating properties of cellular WPC [4,5].

Polymeric composites are materials consisting of two or more phases; usually, the polymer is the continuous phase (matrix), and the fiber is the reinforcing phase [6]. In cellular polymer composites or expanded polymeric composites, there is a third phase referring to the voids (cellular structure) created by the blowing agent in the polymer matrix, which are called cells [7].

The basic mechanisms involved in the production of cellular WPC are usually associated with the incorporation of a blowing agent during composite processing by extrusion, injection or a batch process. The blowing agent is dissolved or finely dispersed in the polymer, after which a large number of bubble nuclei are generated such that they are uniformly dispersed in the polymer matrix. Bubble nucleation is followed by bubble growth due to the diffusion of the dissolved or evolved gas into these nuclei and, finally, by stabilization of the grown bubbles [2]. The blowing agent can be physical, such as liquids with low boiling points or solid chemical compounds that decompose at processing temperatures, thus liberating gases, such as CO_2 and/or nitrogen [8].

In most cases, the addition of WF in cellular composites resulted in narrower cell size distributions and lower average cell dimensions. Since the introduction of a solid phase in the foaming process creates sites of lower surface energy, wood particles are

* Corresponding author. Tel.: +55 54 3218 2371; fax: +55 54 3218 2253.
E-mail addresses: matheus.vgz@gmail.com (M.V.G. Zimmermann), ruth.santana@ufrgs.br (R.M.C. Santana), ajzattera@ucs.com.br (A.J. Zattera).

0261-3069/$ - see front matter © 2014 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.matdes.2014.01.010

believed to act as nucleating agents that enhance heterogeneous nucleation [9–11].

The surface area of WF particles varies for different particle sizes, which can have effects on the viscosity, the cell nucleation, and the cell expansion behavior during foaming of WPC. Conversely, the surface area may also have an effect on the amount of volatiles generated during processing, which, in turn, affects all of the earlier mentioned parameters, and not necessarily synergistically. However, notably few studies can be found concerning the effects of WF particle size on the foaming of WPC in the literature [12].

Most published works relate to rigid expanded composites using polymeric matrices, such as polypropylene (PP), poly(vinyl chloride) (PVC) and high-density polyethylene (HDPE). However, there are few references in the literature that use poly(ethylene-co-vinyl acetate) (EVA) to produce cellular composites. One of most commonly used polymers in the manufacture of polymeric foams is EVA, a polymer that combines good mechanical properties and high flexibility with elastomeric characteristics, which can contribute to the production of a cellular composite with these characteristics. Cellular composites of EVA/WF, even partially crosslinked, are recyclable and have easy processability using various forming methods, such as hot press, batch process, injection and extrusion molding. Those composites may have huge potential for application in footwear products and/or construction industry. Given the shortcomings mentioned above, the objective of this study is to evaluate the influence of the size and content of wood flour on the properties of cellular composites of EVA.

2. Materials and methods

2.1. Materials

Poly(ethylene-co-vinyl acetate), grade EVA 1825 with 18.8% vinyl acetate, was provided by Quattor S.A. (Brasken, S.A.). The coupling agent PEgMA, grade Polybond 3029, was provided by Chemtura Corp. The chemical blowing agent azodicarbonamide (ACA), activated with zinc oxide (ZnO), was provided by Inbra Industria Química Ltda. The crosslinking agent (dicumyl peroxide), grade DCP 40 SAP, and the lubricant (Lub), grade Retiflux, were supplied by Retilox Química especial Ltda. The WF was collected from the stem of a *Eucalyptus dunnis* tree. The wood was previously dried in an oven at 70 °C for 5 h and milled in a MARCONI, model MA 580 knife mill with different sieves sizes. The wood samples were separated in a particle size classifier to particle sizes of: WF_1 65–80 mesh (0.230–0.177 mm); WF_2 80–150 mesh (0.177–0.099 mm) and WF_3 150–270 mesh (0.099–0.053 mm).

2.2. Composite preparation

The samples abbreviations are described in Table 1. The compatibilizer agent PEgMA was added to EVA (2% w/w) using a SEIBT model ES35 single screw extruder, L/D 20 with a temperature profile of 90, 120, 140 and 140 °C and a screw speed of 60 rpm. The additives and fillers were incorporated into EVA + PEgMA using an open roll mill developed by the University of Caxias do Sul (UCS), preheated at 85 °C in the front cylinder and 65 °C in the back cylinder to prevent the adhesion of the band to the back roller. The mixing process entailed sequentially adding each formulation component in the following order: EVA + PEgMA (100 phr), WF, lubricant (1 phr), ZnO (1 phr), ACA (2 phr) and DCP (2 phr). The duration of the mixing step lasted between 10 and 12 min.

After mixing, the composite was formed into preforms using a thermal press SCHULZ at 115 °C with 5 tons of pressure for 5 min using a 140 × 160 × 3 mm mold. The average mass of the preform was 80 ± 2 g. The size and dispersion of the wood particles in the matrix composite can be seen in Fig. 1, which corresponds to the micrograph obtained by the MO of the surface of the unfoamed preform composite under study with 30 phr of wood.

Table 1
Designations and mixture proportions of the cellular composites (in phr).

Sample	EVA + PEgMA	WF_1 (65-80 mesh)	WF_2 (80-150 mesh)	WF_3 (150-270 mesh)
e-EVA	98/2	–	–	–
e-EVA/WF_1 (10)	98/2	10	–	–
e-EVA/WF_1 (20)	98/2	20	–	–
e-EVA/WF_1 (30)	98/2	30	–	–
e-EVA/WF_2 (10)	98/2	–	10	–
e-EVA/WF_2 (20)	98/2	–	20	–
e-EVA/WF_2 (30)	98/2	–	30	–
e-EVA/WF_3 (10)	98/2	–	–	10
e-EVA/WF_3 (20)	98/2	–	–	20
e-EVA/WF_3 (30)	98/2	–	–	30

WF – Wood flour; e-expanded.

The expansion process for the expanded composite (e-EVA/WF) occurred in a heated press. The preform was placed in a 150 × 170 × 12 mm mold for free expansion in the absence of applied pressure at 175 °C. The foaming time of the samples in the heat press was 40 min.

2.3. Characterization

2.3.1. Melt flow index – MFI

The MFI of the composites was measured without the addition of either a blowing agent or a DCP crosslinking agent. The experiment was conducted using a DYNISCO equipment Kayeness Test Systems, model D4001 Hv at a temperature of 190 °C and 2.16 kgf according to ASTM: D1238-13.

2.3.2. Rheometry

The EVA crosslinking parameters were obtained through an oscillating disk rheometer TECHPRO, model Rheotech OD + with a frequency of 1.67 Hz, strain amplitude of 1° and temperature of 175 °C. The test specimen weight was 4 g, and the tests were conducted according to ASTM: D2084-11. From the obtained rheometer curve, the following parameters were evaluated: maximum torque (M_H), minimum torque (M_L), pre-crosslinking time (t_{s1}) and crosslinking time (t_{90}).

2.3.3. Apparent density

The apparent density of the cellular composites was obtained by the ratio of the mass (g) and volume (cm^3) of the sample. The density of seven specimens of each sample was assessed according to ASTM: D1622-08. The density test was performed at a temperature of 21 °C.

2.3.4. Mechanical property of tear resistance

The mechanical property of tear strength of the composites was performed using a universal testing machine, EMIC DL2000, in accordance with the ASTM: D624-12. The test speed was 500 mm min^{-1} with a cell load of 20 kN. A type C tear strength model was used, and the test was performed on five specimens for each sample.

2.3.5. Morphology

The morphology of the cut surface of the cellular composites was evaluated by scanning electron microscopy (SEM – SHIMADZU SuperScan SS-550), previously coated with Au. The software used to measure the cell size was Image Tools for Windows, version 3.00. A universal stereoscope optical microscope also assessed