Composites: Part B 67 (2014) 472–480

Contents lists available at ScienceDirect

Composites: Part B

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

Extruded short wool fibre composites: Mechanical and fire retardant properties

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ARTICLE INFO

Article history:
Received 10 March 2014
Received in revised form 11 July 2014
Accepted 2 August 2014
Available online 9 August 2014

Keywords:
A. Fibres
B. Mechanical properties
C. Statistical properties/methods
E. Extrusion

ABSTRACT

Polypropylene (PP) and wool composite sheets were fabricated by continuous extrusion. Increases in mechanical properties of the composites, such as tensile modulus and strength, were achieved by adding wool and a suitable compatibiliser. Significant manufacturing parameters and desired formulations of the wool–PP composites to achieve the best possible mechanical properties were determined by Taguchi analysis. Wool characteristics within the extruded composites, namely fibre length, orientation and fracture behaviour, were also investigated by image analysis to find out their influence on the improvement of mechanical properties. Furthermore, positive effect of wool on improving fire retardancy of the composite has been identified by some preliminary flammability tests.

1. Introduction

As one of the favoured thermoplastic polymers in the plastic industry due to its good flexibility, transparency and low cost, polypropylene (PP) does not have high enough mechanical and fire retardant performance to satisfy the more advanced requirements of various applications. In order to overcome the modest properties, additional materials, such as glass fibres and halogen, have been added to PP. However, the synthetic reinforcement has led to environmental problems when land-filled or incinerated and an increase in density of the composite [1]. Therefore, natural fibres are increasingly considered as an alternative to the conventional reinforcing material for manufacturing composites because of their inherent advantages over the synthetic fibres namely, biodegradability, low density, high toughness, low energy recovery and CO_2 neutral when burned [2,3]. In particular, high specific strengths and stiffnesses of some natural fibres provide major benefits for mechanical properties. Research and development of composites using lignocellulose fibres have been conducted intensively and the composites have demonstrated predominant performances for mechanical [4,5] and other functional properties [6,7]. Meanwhile, animal fibres, also known as keratin fibres, have drawn less attention in the natural fibre composites research compared to the research done using lignocellulosic fibres. Keratin is a special type of fibrous protein found in wool, nail and feathers. This protein is durable, insoluble, chemically unreactive and pliable, thus protecting animals from extreme environmental conditions [8]. Moreover, keratin is distinguished from other fibrous proteins as its structure has high physical and thermal stability, created not only by the hydrogen bonds and van der Waals forces, but also by their high content of the amino acid cysteine [9]. In particular, the complex structure of the microfibril-matrix in wool plays a significant role in determining the mechanical properties [10]. In addition, relatively high sulphur (3–4 wt%) and nitrogen (15–16 wt%) content present in the amino acid groups contribute low flammability of wool [11]. In spite of the advantages, disposal of wool waste, especially coarse wool or poor quality raw wool from farm breeding, fibre by-product from textile processing, is estimated to be approximately three million tonnes per annum, raising serious environmental and economic concerns [12].

In order to reduce the waste and enhance the value of the fibres, wool fibre reinforced polymeric composites have been recently studied using thermoplastic polymers [13–15], thermosetting resin [16] and other types of matrix [17,18]. As the interfacial bonding created chemically between natural fibres and thermoplastic polymers directly influences the tensile properties of the composites, different treatments have been applied to enhance the interfacial adhesion and significantly improved properties of lignocellulosic fibre composites have been achieved [19,20]. Due to the fact that protein fibres are mixed hydrophobic/hydrophilic depending on sequence of amino acids, a fibre treatment is required to make these fibres compatible with a hydrophobic thermoplastic polymer [8,21]. The effect of short wool fibres and different fibre treatments, such as maleic anhydride modified PP (MAPP) and silane-based coupling agent on mechanical properties

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http://dx.doi.org/10.1016/j.compositesb.2014.08.002
1359-8368/

of compression moulded wool–PP composites has recently been reported [14,15]. Although these additives have been shown to have some positive influence on elastic moduli and thermal stability, tensile strength remained less than that of neat PP.

In this research, wool–PP composites using three grades of commercial PP with different weight contents of wool and MAPP have been manufactured by extrusion, which has not been used by others to fabricate wool based polymeric composites. Taguchi design-of-experiment (DoE) method and analysis of variance (ANOVA) were performed to statistically determine the significant factors contributing to the improvement of mechanical properties. This work aims at investigating the reinforcement effect of wool fibres on enhancement of mechanical and fire retardant properties with suitable manufacturing conditions.

2. Experimental details

2.1. Materials

Coarse wool fibres used as reinforcement in this research were supplied by Bloch & Behrens Ltd. (New Zealand) after removing impurities on wool fibre surface by a scouring process. Three grades of commercially available PP, denoted as PP-BI452, PP-HP400L and PP-H5300, were selected and the details of each PP grade are listed in Table 1. MAPP Licocene 6452 was provided by Clariant Ltd. (New Zealand) to be used as a compatibiliser.

2.2. Manufacturing of composite samples

A continuous extrusion process was used to fabricate the composite sheets based on short wool and PP. This process has not been utilised in any previous studies to manufacture wool/thermoplastic polymer composites. Prior to being extruded, the coarse wool fibres were cut by a granulator (GRV variant series, Italy) to prepare short fibres and then dried at 75 °C for two days to reduce the moisture content to less than 5%. After dry-mixing of wool, PP and MAPP, a Brabender® twin screw extruder, LTE 26 (screw length L/screw diameter D = 32), was operated for melt blending at a screw speed of 170 rpm with a temperature profile of 175 °C to 179 °C across 10 heating zones. A WOYWOD Plasticolor® 2200 pellet screw feeder was set up to feed the dry-mixed materials at a constant rate during the melt-blending. A composite sheet, having around 100 mm width and 0.6 mm thickness, was then fabricated using a single screw extruder (L/D = 32) with a slit die (100 mm × 0.8 mm) at a processing temperature of 180 °C and a screw speed of 8 rpm. The extruded composite sheet was subsequently passed directly through a pair of calendering rolls to achieve uniform thickness and a good surface finish.

2.3. Material characterisation

2.3.1. Microscopic characterisation

Dimensions of the coarse wool fibres were measured using an optical microscope (Leica MZ16, Germany) and an image analysis program (Image J, National Institute of Health, USA). To determine the distribution of wool within the composite and the interfacial adhesion between the fibres and PP, environmental scanning electron microscopy (ESEM) was performed on the cross-sectional areas of tensile specimens of the wool–PP composites with an FEI Quanta 200F Field Emission Gun SEM machine.

Table 1
Specifications of commercial polypropylenes.

PP grade	Manufacturer	Density (g/cm^3)	Melt flow index (MFI) (g/10 min)	Application
BI452 copolymer	Samsung	0.9	8.0	Injection
HP400L homopolymer	Lyondell Basell	0.9	5.5	Injection
H5300 homopolymer	Honam	0.9	3.5	Extrusion

2.3.2. Thermogravimetric analysis

Thermal decomposition of neat PP and wool–PP composites was measured using a TGA-50 (Shimadzu, Japan). Samples of around 7 mg were heated from room temperature (~20 °C) to 700 °C at 10 °C/min under inert atmosphere.

2.3.3. Cone calorimeter

Heat release rate (HRR) of neat PP and wool–PP composites under extremely high heat transferring condition were measured by a cone calorimeter (Fire Test Technology, UK) according to ASTM E1354. All samples were preconditioned for at least 48 h at 23 °C and 50% relative humidity, and tested in the horizontal position under an external heat flux of 50 kW.

2.4. Mechanical testing

2.4.1. Composites and neat PPs

Tensile tests were performed on an Instron 5567 (10 kN load cell) universal testing machine (UTM). According to ASTM D882, the tensile moduli (chord modulus between 0.05% and 0.25% strain) and strengths were measured at a crosshead speed of 12.5 mm/min and the extension under tension was measured by a video extensometer. Flexural testing was conducted using a 3-point bending rig on the Instron 5567 UTM based on ASTM D790. The span length of 24 mm and crosshead speed of 16 mm/min were used, as required by the testing standard.

2.4.2. Single wool fibre tensile tests

The tensile stiffness and strength of a single wool fibre were tested on an Instron 5567 UTM with a 10 N load cell (Interface Model SMT 1-10) according to ASTM-D3822. Twenty fibres were selected randomly and then tested using the cross-head speed of 30 mm/min. Load-extension graph of each test was recorded to obtain the properties.

2.4.3. Interfacial shear strength between fibre and PP

Interfacial shear strength (IFSS) between a single fibre and PP was measured by a microbond testing method [22,23]. A PP droplet was created by tying a knot of thin PP filament around the single wool fibre in order to obtain short embedded length. The knotted PP around the fibre was put in an oven at a temperature of 185 °C for 10 min and then cooled down to ambient temperature. Each of the samples was monitored under an optical microscope to identify the symmetrical shape of the microdroplet and measure the dimensions of the sample, such as embedded length of PP and fibre diameter, prior to the test. Twenty samples were then tested using an Instron 5567 UTM with a 5 N load cell (Interface Model SMT 1-5). So, the wool fibre critical length could be obtained by Eq. (1) following Bowyer and Bader model [24,25] for aligned and discontinuous fibres.

$$L_\varepsilon = \frac{E_f \varepsilon_c D}{2\tau} \qquad (1)$$

where E_f is the Young's modulus of fibre, D is the fibre diameter, ε_c is composite strain and τ is the interfacial shear strength.

2.4.4. Tensile properties

Tensile properties of the extruded short wool–PP composites were predicted by a modified rule of mixture, as defined by Piggott [26,27]. In order to apply the effects of short fibre orientation and length distribution towards the tensile properties of the composites, fibre orientation efficiency factor and length correction factor