



Compatibilization of toughened polypropylene/biocarbon biocomposites: A full factorial design optimization of mechanical properties



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ABSTRACT

The structure-property relationships of toughened polypropylene/biocarbon biocomposites prepared via melt extrusion are investigated with a focus on the effects of biocarbon particle size, functional polymer type and concentration. A full factorial design was utilized to study each factor's main effect and how their interactions with other factors affect the final mechanical properties of the biocomposites. The statistical analysis confirmed the synergistic interactions between the biocarbon and the functional polymer which improves the impact toughness of the composites by 120% while maintaining or improving the stiffness of the composite. Among the investigated factors, the type of functional polymers had the greatest impact on the properties by affecting the morphology of phases. Based on these analyses, the biocarbon with particle size range of 106–125 μm together with 5.0 wt% of maleic anhydride grafted polypropylene was selected as the optimum composite formulation providing the best stiffness-toughness balance among the tested samples.

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1. Introduction

There has been an increasing interest in polymer/biocarbon biocomposites within the last few years [1–7]. Unlike petroleum-based carbon black, biocarbon can be used at high concentrations in thermoplastics for reinforcement and coloring purposes, which makes it an attractive replacement for common fillers, such as CaCO_3 and talc, in various applications [2,5,8]. Additionally, the lower density of biocarbon compared to mineral fillers provides an opportunity to create a composite with higher biobased content and lower weight when it is substituting a conventional filler [9].

Elastomer toughened polypropylene (PP) based composites are one the most used composites in the automotive industry [10]. The performance of these composites is typically measured based on the stiffness and impact toughness as there is a tradeoff between

these two and an increase in one could result in a decrease of the other [11]. Therefore, the desired goal in such a composite system is to achieve a stiffness-toughness balance rather than focusing only on one property. Relatively high concentrations (up to 40 wt%) of mineral fillers such as CaCO_3 , talc, BaSO_4 , etc. have been routinely used to achieve stiffness-toughness balance. However, the move towards reducing greenhouse gases (GHGs) and the enforcement of new regulations (The Corporate Average Fuel Economy and EU regulations) for fuel efficiency requires lighter weight materials with lower carbon footprints [12]. Biocarbon could be a promising bio-filler since it is biobased and a relatively light filler.

In multicomponent systems, like elastomer toughened PP composites, each component is added to improve a specific property of the matrix. The elastomer phase improves the toughness, while the hard filler adds to the stiffness of the composites. However, the final mechanical properties are determined by the morphology of the phases which dictates the stress transfer through the composite [11,13]. In extreme conditions either the components are dispersed separately or the filler particles are encapsulated by the elastomer phase, but normally an intermediate morphology would form [14,15].

Pukánszky et al. [16] investigate the governing parameters on

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the phase morphologies of toughened PP CaCO₃ composites. Their results suggested that in a similar mixing condition (similar viscosity and shear rate), adhesion between the phases and particle size are the two most dominant parameters in determining the morphology of the composites. These factors have been investigated in other PP based composites and biocomposites and the findings were analogous to Pukánszky et al. study [13,17,18]. The adhesion between the phases can be manipulated by means of a compatibilizer. Generally, small amounts of functionalized PP are added to the system to enhance the interface between functional fillers and non-polar polymeric chains of PP. Functionalized rubbers can be added to improve the rubber-filler interactions. In the previous work of the authors [19], two types of biocarbon that were pyrolyzed at different temperatures were mixed with toughened PP. The results demonstrated that the rubber phase will tend to encapsulate the biocarbon particles regardless of biocarbon surface properties. The observed poor interface between the biocarbon particles and the matrix suggests further need for compatibilization.

Normally, an optimum concentration of a compatibilizer exists, any deviations from it would cause deterioration of properties [20,21]. The excess amount of compatibilizer would not improve the interface any further as it already reached its maximum concentration at the filler-polymer interface. Instead, this excess amount will form dispersed domains in the main matrix [20]. This can cause incompatibility between other components of the matrix (such as rubber) and reduce the properties. Compatibilizers are also more expensive than the base polymers and are preferred to be used in small amounts. Therefore, it is of prime importance to find the optimum level for compatibilizers in a system to achieve the highest possible properties.

Maleic anhydride grafted PP (MAPP) has been shown to be an effective compatibilizer for PP-biocarbon composites [3]. Ikram et al. [22] used Taguchi statistical design of experiment to investigate the effect with and without MAPP on the stiffness and strength of PP/Wood/Biocarbon composites. The results confirmed the effectiveness of this compatibilizer on the tensile and flexural properties of the composites, but its effect on the impact toughness of the biocomposite was not reported. Interestingly, no studies have reported on how the type and different concentrations of a compatibilizer affects the morphology, and the performance of elastomer toughened biocarbon biocomposites to the best of our knowledge. This is certainly due to the fact that utilization biocarbon in polymer composites is relatively a new field.

The purpose of this study is to examine the effect of biocarbon particles size, compatibilizer type and concentration on the mechanical properties of the toughened PP composites and how these changes are related to the morphology. In this work, PP based and PE based compatibilizers were used at three different concentrations. The compatibilizers were added to the elastomer toughened PP/biocarbon composites and notched Izod impact strength, and tensile modulus were measured as the response. It is hypothesized that MAPP would provide a better interaction between the PP and biocarbon, generating a separate dispersion of phases, while the encapsulated structure would be formed in the case of MAPE. Particle size as one of the important factors was considered in the statistical design. Since it is known that these parameters interact with each other, a full factorial design of experiments was used to make sure that all the interactions between the factors are considered in the statistical model. Variation of compatibilizer concentration together with compatibilizer type and the biocarbon particle size would allow predicting an optimal range of compatibilizer concentration to achieve the best balance of properties.

2. Materials

Pyrolyzed miscanthus grass biocarbon (BC) was produced at Competitive Green Technologies, Leamington, ON, Canada. The pyrolysis temperature of this biocarbon was ~630 °C and the material kept in the furnace for 15 min. The output charred fibers were then hammer milled to ~400 µm (1/64 in.) by the company.

Commercially available injection molding grade polypropylene (PP), (trade name 1335Z) was used as the matrix (Pinnacle Polymers LLC, LA, USA). The melt flow index (MFI) at 230 °C/2.16 kg and density of PP were 35 g/10 min and 0.9 g/cm³ according to the materials datasheet, respectively. Polyoctene ethylene copolymer (POE), a product of Dow Chemical Company (trade name Engage 8137), was used as the elastomeric phase of the matrix. The MFI at 190 °C/2.16 kg and density of POE were 13 g/10 min and 0.866 g/cm³ respectively. A maleic anhydride grafted PP (MAPP), and PE (MAPE) copolymer was added to produce preferential adhesion between the particles and polymers. The MAPP and MAPE used in this study were Fusabond P353 and M603 a product of DuPont (Wilmington, DE) respectively.

3. Experimental

3.1. Specimen preparation

As received biocarbon batch was sieved to two particle size ranges of 106–125 and smaller than 20 µm using a Ro-tap sieve shaker (W.S. Tyler, OH, USA) fitted with appropriate Tyler sieves. The large particle size range (106–125 µm) underwent a second sieving cycle through a forced air sieving machine (Air-jet 200, Retsch, Germany) to remove any particles smaller than 106 µm from this range.

To achieve results independent of the size distribution of the particles (as much as possible by the sieving method) and maintaining a meaningful difference between the sizes, the sieves were chosen from American Standard Test Sieve Series so that the obtained particle size range had one order of magnitude difference in the size range while retaining a similar distribution range (20 µm).

Toughened PP/biocarbon biocomposites were prepared by melt processing at 190 °C, in DSM Xplore micro compounder with length over diameter ratio (L/D) of 18 (DSM Xplore, the Netherlands). The screw speed was set to rotate at 100 rpm in the co-rotating configuration for 120 s during the mixing stage. The compound then transferred to DSM Xplore 12 cc injection unit to mold the test specimens. The injection, packing and holding cycles were set to perform at 0.4, 0.8 and 0.8 MPa for 4, 6 and 10 s respectively. Mold temperature was controlled at 40 °C throughout the injection process. Table 1 shows all the formulation and corresponding designation used in this study.

3.2. Characterization

Tensile properties of the composites were measured by Instron universal testing machine (Norwood, MA). Type IV specimens were tested as per the ASTM D638-14 protocol with a test speed of 50 mm/min at room temperature and 50% relative humidity. The impact strength of the samples was measured in accordance with ASTM D256-10. The samples were notched 48 h prior to testing. The tests were conducted on a TMI monitor impact tester (Testing machine Inc. DE, USA) with five ft. lb pendulum at room temperature.

Fracture surface morphologies of samples were observed by scanning electron microscopy (SEM), Phenom ProX (Phenom World

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