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Processing parameters and characterisation of flax fibre reinforced engineering plastic composites with flame retardant fillers *



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ABSTRACT

This work studies the possibility of compounding natural fibres (flax) into engineering plastics (PA6 and PB6) and comparing the results with counterpart glass fibre composites. The problem in compounding is the difficulty to compound the fibres with such polymers of high melting temperatures without decomposing the natural fibre thermally. Preliminary experiments are tried to define the possible processing window using the kneader namely temperature, compounding time and shear rate. Fibre content is tried in range of 0–50 wt.% with 10% step. The mixing temperature covers the range around the melting temperature 'Tm' [Tm–20, Tm+20]°C. The use of pre-melting temperature in compounding would utilise the energy evolving by fibres mutual rubbing. Compounding time is optimised at the minimum level. Shearing rate is tried at 25, 50, 75 and 100 rpm. Optimum conditions are defined to be 210–230 °C and 200–210 °C for PBT and PA6 respectively. Shearing rate is also defined to lie within 25–50 rpm.

Two different additives of non-organic mineral and organic phosphate flame retardants are tried with the prepared composites either alone or in combination with each other. The loading of flame retardants is limited to 20 wt.% in order to leave a space for natural fibres as well as the polymer and to keep in turn the overall composite mechanical properties. A mix of 1:1 ratio between the both types of retardants is needed to reach V0 flame retardation level. Mechanical properties are even improved 30% in E-modulus and 4% in strength with respect to composites without flame retardants. However, the injection moulding is reported to be difficult because of the high viscosity and the parameters should be optimised regarding the desired flame retardance level and the required mechanical properties as well as keeping the fibres not damaged.

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1. Introduction

Application of natural fibres into the polyolefin plastics like polypropylene and polyethylene has been already deeply studied under different aspects; namely improving the fibre to matrix adhesion [1,2], flow and rheological behaviour [3,4] process optimisation [5,6] and even modelling of strengthening mechanisms [7,8]. These composites are even commercialised and still exploring fields of new applications [9,10]. However engineering plastics reinforced with natural fibres represent a more difficult challenge because of the higher melting point of these plastics. This requires higher processing temperatures. For example, it is a common practice to inject PBT and PA6 at least by 250° and 230 °C respectively. These values are recommended to be increased by 20 °C in case of reinforced grades. This high temperature is supposed to result in the natural fibre decomposition firstly in hemicelluloses at 250 °C then in cellulose region starting from 300 °C [11]. That is why this topic of reinforcing engineering plastics with natural fibres rare in literature except trials [11–13].

These trials start with mixing Nylon polyamide PA6 with high purity cellulose fibres [12] as well as wood fibres [13,14]. These previous trials in 2001 lead to a patent later [15]. The patent [15] focuses on the design of extrusion process parameters where the polymer granulates are fed at the primary feeder of the extruder. Then the fibres are side fed to the extruder where low temperature profile along the extruder in Arcaya et al. [16] compounded flax, jute, pure cellulose and wood pulps with PA6 and PA6,6.

 $^{\,^{*}}$ The results of this work have been presented at the 4th Conference of Natural Fibre Composites, Rome 17–18 October 2013.

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Mixing temperature is kept again 5 °C above the physical melting point measured by Differential scanning calorimetry DSC. Mechanical properties are reported to improve but unexpectedly, nothing about fibre structure damage specially or burning features with PA6,6 is mentioned. Wu et al. [17] prepared compoite made of polybutyleneterephthalate (PBT) and sisal fibres using blade rotor at mixing conditions of 220–230 °C, 50 rpm and 20 min. Mechanical properties are not reported [17]. As observed, the literature does not show intensive trials regarding the engineering plastics composites with natural fibres the next 10 years. This is attributed to the difficulty of the compounding process conditions which should be strictly controlled to avoid natural fibre decomposition. These conditions are namely temperature and time as well as the shearing rate.

The Successful implementation of natural fibres within engineering plastics will open new market areas for the application of natural fibre composites due to their low cost and low density as well as their process-ability in the available injection and extrusion machines [7,8].

2. Preliminary thermal characterisation and material selection

Compounding of natural fibre with an engineering plastic has utmost a narrow processing window which allows relatively safe processing without natural fibres damage as well as keeping the polymer in the molten phase. So it is required to select natural fibres with high thermal stability and resistance to decomposition. Fig. 1 shows the thermal gravimetric analysis (TGA) of some commonly used natural fibres namely flax, hemp, sisal and Jute. The temperature range under consideration is within 100-300 °C. The change at 100° depends on the amount of water absorption level which is not important for this study because the fibres will be executed to drying step before compounding. On the other side, the processing temperature should not reach high temperatures as 300 °C. As shown the weight derivative curves in Fig. 1, jute has the higher thermal stability and resistance to degradation but its trend to decompose after the onset temperature is very fast. Thus suggests unstable situations after crossing the onset decomposition temperature which in other words means more gas evolution. From the other side, it is obvious that flax has the least change in weight which means more stable behaviour even at the expected high processing temperature. This means that the degradation features, where lignin decomposes, is kept at minimum level in case of flax natural fibres. Away from the quantitative TGA results, some qualitative measures of thermal degradation can be also noticed like the degree of fibres' dark coloration and fibres' smell like burned wood.

This study deals with commercial flax fibres of relatively good thermal resistance. Compared to its weight at 100 °C, flax fibre



Fig. 1. TGA of flax, hemp, sisal and jute (air atmosphere, 10 k/min).



Fig. 2. Processing window of natural fibre with engineering plastic (example of PBT and flax natural fibre).

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