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Performance of different intumescent ammonium polyphosphate flame retardants in PP/kenaf fibre composites

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ABSTRACT

The paper compares the flammability and degradation performance of polypropylene (PP)/kenaf fibre composites under three different intumescent ammonium polyphosphate (APP) flame retardants. Intermeshing co-rotating twin screw extruder melt mixing approach was used to prepare the composites. The initial morphological analysis reveals that twin screw melt compounding has provided an effective homogeneous dispersion and distribution of particles, irrespective of their different particle sizes and surface energies. The thermogravimetry analysis (TGA) proves that the presence of intumescent coating of APP1 improves the thermal stability and decomposition temperature over the others. UL-94V tests and cone calorimeter analysis show that the amount of APP content and the high aspect ratio of the particles are the key factors in obtaining homogeneous flame retardant blend, which tends to decrease sustained combustion and reduce the peak heat release rates of the composites. Morphological analysis of char residue further proves that the cross-linking density of the char provides better insulation for underlying material even though the initial swelling increases the degradation temperature through pronounced blanketing effect. Further analysis using different APP particles recorded competitive mechanical properties.

1. Introduction

Nowadays, the world concept of "go green" is the challenge for designers in many aspects, which warrants the identification of more environment friendly resources. In this regard, replacing synthetic fibres with natural ones in composite materials has to play a major role in manufacturing engineering [1–4]. The advantages, such as low cost, low density, high toughness, relatively high specific strength properties, low abrasiveness, low energy consumption in fabrication, and CO_2 neutrality of natural fibres, provide the researchers incentive to use these materials in new developments [5]. The most recent research efforts in areas, such as fibre matrix compatibility and fibre treatment in removing surface impurities, have led to enhanced mechanical properties, which is useful in a wide range of industry applications. However, poor thermal stability is a major drawback in using these materials, especially in transportation and aerospace applications [6–8].

Generally, natural fibres act as combustion sources in composites. If the fibre cellulose content is high, it tends to increase the flammability due to high levels of levoglucosan, but the amount of lignin content in the fibres leads to char formation after initial ignition and provides a thermal barrier. This phenomenon can be used to control the fire growth, by selecting good combination of materials (fibre and matrix), and obtaining effective homogeneous blend composites through optimum processing. Nowadays manufacturing applications demand high safety regulations, forcing worldwide researches to deal with finding new flame retardant additives to replace the existing ones, which are not highly effective with natural fibre based composites [9–11].

Halogen based flame retardants are more effective in polymer composites with their potency in quenching flames, but the environmental regulations significantly limit their use in today's flame retardant applications [12,13]. Phosphorus based intumescent flame retardants (IFR), constitute one way of addressing this issue besides using inorganic fillers, such as aluminium trihydrate or magnesium hydroxide, which always requires high quantities of filler to effectively protect a composite from fire [14]. The IFR system normally contains three components: an acid source, a carbonisation agent and a blowing agent. In a widely used IFR system, ammonium polyphosphate, pentaerythriol and melamine act as acid source, carbonisation agent and blowing agent, respectively [15,16]. The high efficiency of IFR system and the ability to produce low smoke and nontoxic gases lead them to wide usage

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in engineering applications. Even though, with these advantages of IFR system, high moisture sensitivity with poor compatibility cause problems in achieving homogeneous blend, when using IFR as flame retardant in polymer composites [17–19]. Extensive drying of fibres with the addition of efficient coupling agents can effectively reduce the moisture content and surface tension to provide desirable homogeneous composite during manufacturing [20,21] The main goal of this work is to investigate the flame retardant effectiveness of ammonium polyphosphate (APP) based IFR systems on PP/kenaf natural fibre composite [22–24]. Three different APP based flame retardants were chosen in this regard and their behaviour was analysed in 30 wt.% natural fibre composite. Twin screw melt compounding method was used to mix the immiscible blends under high shear rates to obtain flame retardant composites [25]. Detailed morphological analysis was carried out to show how the APP particles effectively dispersed within the composite to form homogeneous rigid blend.

2. Experimental and methodology

2.1. Materials

PP block copolymer (MFI = 8.0 g/10 min, 230 °C/2.16 kg) was purchased from Clarient New Zealand Limited, anhydride modified polypropylene polymer modifier (MAPP) (MFI = 120 g/10 min, 190 °C/2.16 kg) was purchased from DuPont New Zealand Limited. Kenaf bast fibres were supplied by Bruce Smith Limited, New Zealand. Three different grades of APP was used, APP1 (melamine formaldehyde surface coated ammonium polyphosphate ~20 μm, P_2O_5 = 68.5%, N = 17%) was supplied by Salkat New Zealand Limited, APP2 (fine particle ammonium polyphosphate <50 μm, P = 31–32%, N = 14–15%) and APP3 (intumescent ammonium polyphosphate <10 μm, P = 20%, N = 14%) were supplied by Chemcolour Industries New Zealand Limited. Irganox 1076, purchased from Clarient New Zealand Limited, was also used against thermo oxidative degradation of PP.

2.2. Preparation of flame retardant composites

Ground PP, MAPP and APP were dried over-night at a temperature of 80 °C. Kenaf bast fibres (30 wt.%) were dried in an oven at 70 °C for 3 days and then dry-mixed with PP, MAPP (3 wt.%) and APP (20 wt.%) mixture using a Phas-o-mec high intensity mixer at 19 Hz for 2 min. All blends were compounded in an intermeshing co-rotating Scientific Twin-Screw Extruder (Type LTE26-40), having screw diameter of 26 mm and L/D ratio of 40. Temperature profile of 165–185 °C was used along the extruder barrel, from feed section to die section, with a screw speed of 150 rpm and 2 kg/h throughput. Final compound was ground into pallet form using Moreto grinder and dried overnight, before making test specimens using Boy 50A injection moulding machine with a heat profile of 165–180 °C from feed zone to die zone.

2.3. Characterisation of flame retardant composites

Thermal analysis was carried out in a thermogravimetric analyser (Shimadzu TGA 50), using constant heating rate of 10 °C/min under inert atmosphere with an argon gas flow rate of 80 ml/min. Flammability properties were investigated using a cone-calorimeter test apparatus (Fire Testing Technology Limited, UK), and UL-94V standard test (ASTM D3801). Cone calorimeter test was conducted at a horizontal sample position with an external heat flux of 50 kW m^{-2}, following ASTM E1354 standard. Finally, the morphological analysis of tensile fractured samples and after burn samples was carried out using scanning electron microscopy (SEM) (Philips FEI XL30S FEG) and environmental scanning electron microscopy (ESEM) (FEI Quanta 200F).

3. Results and discussion

3.1. Morphology analysis under SEM

3.1.1. Morphological features of PP/kenaf/APP composite

The SEM image Fig. 1a, shows the APP2 particles before compounding. Even though the particles are in different size and shape, the lateral dimensions are ranging within few microns. During the preparation of effective flame retardant homogeneous composite with immiscible blends of PP/kenaf and APP, the form of the solid particles and the viscosity of the polymer play a major role with the presence of the compatibilizer. When high percentages of solid additives (20 wt.% of APP and 30 wt.% of kenaf fibre) incorporated into polymer matrix material their particle size, moisture content, cohesive strength and external surface energies has to be well balance within the mixture to effectively distribute them throughout the composite. In this regard, the high shear twin screw extruder provides an effective dispersion and distribution to promote homogeneity in the composite as shown in Fig. 1b, though it leads to rupture and reduction to primary particles. The presence of extruder kneading elements create re-agglomeration and exfoliation of APP particles within the matrix and thereby improve the miscibility and concentration of the blend providing high aspect ratio, finally leads to effective flame retardant composite with improved mechanical bonding strength.

3.1.2. Morphological features of fractured samples

Surfaces of the fractured samples were observed under SEM, Fig. 2, to identify dispersion and distribution of APP particles within PP/kenaf composite. The kenaf fibres are well distributed within PP matrix, Fig. 2a, and with the presence of compatibiliser (MAPP) it is often possible to bring homogeneity to the composite by introducing covalent bonds across the fibre interface [26]. Surface coated APP grade (APP1) composites show better dispersion of fibres with low void content, Fig. 2b, compared to other APP composites. This may be due to the synergistic effect from surface coating, which leads to reduce interfacial incompatibility by reducing interactions. Furthermore, it can enhance the surface treatment (over MAPP) of fibres, and thereby fibres tend to well disperse within the matrix, thus improving overall performance of the composite. On the other hand, well dispersed fibres tend to undergo more damage, such as splitting, during high shear processing (under twin screw extrusion), that may potentially degrade their effective properties even in a homogeneous blend. Other intumescent APP (i.e. APP3) has shown a different structure in compatibility leading to larger amount of long straight fibres in the composite, Fig. 2d. The void content present shows that it has not achieved blend homogeneity as such compared to other APPs, but the presence of long aligned fibres improves the composite properties over PP/kenaf composites. Over the other blending methods [10,27] more uniformity, simplicity and large scale production capability are with the twin screw extrusion process, which can be beneficial for long term applications of different manufacturing techniques. These morphological analyses also proves this twin screw melt mixing approach is one of the best approach in obtaining homogeneous flame retardant composite with minimum defects.

3.2. Thermal properties of flame retardant composites

Thermal decomposition of polymers, fibre composites and the influence of these with three different intumescent APP flame