Polypropylene fibers containing dispersed clays having improved fire performance. Part II: characterization of fibers and fabrics from PP–nanoclay blends.

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Polypropylene fibres containing dispersed clays having improved fire performance Part II: Characterisation of fibres and fabrics from PP-nanoclay blends.

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ABSTRACT

In this second part of a series of studies on polypropylene (PP) fibres having improved fire performance and containing nanodispersed clays we build on the observations of the first part of this work (published in Journal of Applied Polymer Science), where the need to improve dispersion of nanoclays is the key to achieve significant improvement. This paper attempts to address this issue by reporting studies of the use of several different functionalised clays in the presence and absence of compatibilising PP copolymers and also to which the flame retardant, ammonium polyphosphate has been introduced. Compounded polymer samples have been characterised using XRD, optical and scanning electron microscopies and their melt flow indices (MFI). These compounded polymers have been successfully extruded into filaments using a single screw extruder and then knitted into fabric samples. Filament physical properties are reported together with burning behaviours of fabric samples using limiting oxygen index (LOI) and flame spread tests.

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1. **INTRODUCTION**

Improving the flame retardancy of polypropylene [1] and particularly fibres and textiles using nanoparticulate additives, is faced with the primary challenge of optimising the dispersion of otherwise hydrophobic particles such as montmorillonite clays, even when functionalised with relatively hydrophilic species as demonstrated in the first paper in this series [2]. While nanocomposites have attracted considerable interest as having the potential for reinforcement and flame retardants (FR) activity since 1997 due to their reported improved mechanical and fire properties [3-5], most of the published work to date in the nanocomposite fibre area has been on improving tensile and stiffness (as modulus) properties, in particular for nylon 6 [6-9] with melt processing variables [10-12] and even electrospinning [13] being of interest. Apart from the work of Bourbigot et al on nanocomposite polyamide 6 (PA6) fibres and the observed reduction in cone calorimetrically determined peak heat release rates (PHRR) [6], no other work apart from Part 1 of this series has been reported for polypropylene fibres. The success of Bourbigot et al. in achieving high levels of dispersion is a consequence of the relatively high polarity of the PA6 polymer which in the molten state behaves like a polyelectrolyte. No such advantage is observed with polypropylene and others have reported on the challenges that this polymer poses [14].

Dispersion can be improved by adding maleic anhydride-grafted polypropylene and, while this effect is concentration dependent, in order to keep the rheological properties necessary for extrusion into fibre, lower levels are preferred. Levels between 1-3% w/w of grafted PP along with sufficient compounding have been shown to improve the dispersion of the clay platelets in the PP matrix. In a parallel study we have studied the optimisation of nanoclay dispersion in polypropylene by observing the effect of different graft copolymeric modifications of PP [15]. It was found that diethyl-p-vinylbenzyl phosphonate (DEP) and maleic anhydride (commercially available as Polybond 3200) - grafted PP gave the best results for improving the dispersion of the functionalised clays (Cloisite 20A, Southern Clay Products, USA and Elementis Bentone 107 (E107),
Elementis Specialities Inc). A study by Garcia-Lopez et al has also reported that the use of Polybond 3200 gave superior interfacial adhesion between clay platelets and polymer, plus an improvement in mechanical performance compared with those samples where diethyl maleate had been used as the coupling agent [16]. In general, a compatibiliser with a low melt flow index (MFI) can improve the dispersion of the clay within the polymer matrix as can the feeding sequence of additives into the polymer. Direct compounding of all materials is very simple but this can interfere with the wetting of clay tactoids by compatibiliser and thus stop delamination of the clay platelets. However, adding PP to already-melted compatibiliser and clay dispersion part way down the screw of a twin screw extruder would seem to improve the exfoliation of the clay [17]. Also the residence time and shear rate are important in a well-dispersed mix, with high shear rates and long residence times, shearing the stacks of nanoclays to yield a delaminated structure [18].

In Part 1, we demonstrated that as, has been generally demonstrated in the literature for a number of polymers [19], the addition of a small quantity of clay to PP fibres and fabrics improves the fire performance properties by lowering PHRR values [2]. In Part 2, we extend the work and report the burning behaviour of knitted fabrics produced from polypropylene fibres comprising several dispersed nanoclays and compatibilising copolymers in the presence of ammonium polyphosphate (APP) as a model conventional flame retardant.

2. EXPERIMENTAL

2.1 Samples

2.1.1 Materials

Polypropylene, PP: Fibre grade polypropylene (PP) chips, Moplen HP561R, Basell Polyolefins

Grafted polypropylene:

Pb: Polybond 3200, Crompton Corporation; maleic anhydride modified homopolymer polypropylene with a maleic anhydride level 1% (w/w)

DEP: Polypropylene grafted with diethyl-p-vinylbenzyl phosphonate (DEP) with synthetic procedure described elsewhere [15].

Clays:
**20A:** Cloisite 20A, Southern Clay Products, USA; modified with dimethyl, dehydrogenated tallow quaternary ammonium chloride (2M2HT)

**30B:** Cloisite 30B, Southern Clay Products, USA; modified with methyl, tallow, bis-2-hydroxyethyl, quaternary ammonium chloride (MT2EtOH)

**E:** Bentone 107, Elementis : modified with dimethyl, dehydrogenated tallow quaternary ammonium ion.

**PCI:** Montmorillonite (Southern Clay Products, USA) modified with vinyltriphenyl phosphonium bromide by the method described elsewhere [20].

**Flame retardant:**

Ammonium polyphosphate (APP), Antiblaze MCM, Albermarle Corporation,

**Samples**

**Matrix 1:** This set of samples was designed to investigate the sensitivity of PP fibre properties with respect to maleated PP content (1%, 2%, 5%w/w). In each case of clay/compatibiliser combination the maximum component levels were determined by that above which excessive strength loss of fibres occurs. Matrix 1 samples comprised different levels of Polybond graft and E-107 clay given in Table 1.

**Matrix 2** is based on the performance of matrix 1 samples. 1 % w/w graft and 3% w/w clay levels have been used to prepare samples with different types of grafted PP and nanoclay types as given in Table 2.

**2.1.2 Compounding**

Polypropylene and additives were hand-mixed in a plastic container prior to compounding. A Betol BTS30 twin screw extruder (at Manchester Metropolitan University, Manchester) with a temperature profile over six heating zones between 179 and 190°C was used for compounding samples for matrix 1 (see Table 1). The samples were passed through the compounder twice to improve dispersion of the clay within the polymer matrix. For matrix 2 samples, the Thermoelectron Prism Eurolab 16 twin screw extruder at the University of Bolton was used. Compounded polymer samples were pelletised after cooling in the water bath.

**2.1.3 Melt extrusion into filaments / tapes**
Filaments were extruded from pellets using a laboratory-sized Labline MK 1 single screw melt extruder. The screw, driven by an electric motor, has an internal diameter of 22 mm. and three zones, a feed zone, a compression zone and a metering zone which feeds into a cavity transfer mixer for enhanced mixing. The polymer is fed into the hopper and melts in the barrel as it is carried along by the screw through a barrel temperature profile of 180 – 230°C. The polymer is then forced into the die head and extruded through a 40 hole spinneret. The filaments are passed over a set of six slow rollers, the first two of which are heated prior to traversing the faster drawing rolls. A draw ratio of 6:1 was selected to yield filaments of acceptable tensile properties but on occasion it was necessary to reduce this to avoid filament breakage.

Polymer tapes (40mm width, 0.6±0.2mm thickness) were extruded using a tape die primarily to provide samples more convenient for microscopic investigation while having experienced an extrusion cycle similar to that for filaments in addition to compounding cycles. Tapes were collected as they cooled with no drawing.

2.1.4 Fabric production

Because of the limited quantities of filaments available, production of small samples of weft-knitted fabric only was possible in order to test samples in fabric form. Filaments were knitted into fabric strips sufficient for flammability testing with a small, hand, circular knitting machine, gauge E7. Area densities are recorded in Table 3.

2.2. Material characterization

Melt flow index (MFI) values were determined using a Davenport (London) Ltd., melt flow indexer to determine the melt flow rate (MFR), which gives an inverse measure of viscosity. For polypropylene, measurements were taken at 230°C according to BS EN ISO 1133:200 to determine the effect of nanoclay on the melt viscosity of the polymer. Three replicates of each sample were undertaken (coefficient of variation = 5%) and the results averaged.
Differential scanning calorimetric (DSC) experiments were conducted using a Polymer Laboratories (PL-DSC) instrument under flowing nitrogen (10 ml/min) with a heating rate of 10°C/min from room temperature to 350°C. About 2.5 mg of sample was taken in each case and all the thermograms were then normalized to a specimen mass of 1.0 mg for comparison.

A Dynamic Analyser Rheometer RDA II (Rheometrics) with parallel plate geometry and a plate diameter of 25 mm was used to characterize nanoclay dispersion in the polymer matrix. Sample thickness was kept at about 1 mm and to ensure the full coverage of viscoelastic region, linear rheological measurements were performed at a frequency range of 0.1 to 100 rad/s. Elastic moduli (G’), loss moduli (G”) and complex viscosities (η*) were obtained at 240 °C. The temperature control was accurate to within ± 1 °C. Experiments were conducted under a nitrogen atmosphere in order to avoid oxidative degradation of the specimen.

X-ray diffraction (XRD) studies were carried out on both compounded polymer and selected filament samples using a Bruker D8 Advance X-ray Diffractometer with CuKα radiation (40kV, 40mA) monochromatised with a graphite sample monochromator over the 2-theta range 2-62° with a step size of 0.02° and a count time of 12.2 seconds per step. Analyses were performed on the collected XRD data for each sample to determine d-spacings using the Bruker XRD search match program EVA™.

Optical microscopy was carried out using a Nikon Labophot 2 optical microscope with image capture by a JVC TK-C1381 colour video camera.

Scanning electron microscopic (SEM) images were obtained using a Cambridge Stereoscan 200 SEM having a tungsten electron gun with accelerating voltage capacity 0.5-30kV and magnification ranges between 30X to 300,000X at 30 kV providing resolution down to 60Å. All images were obtained at 10kV in order to minimise target polymer damage.

2.3. Physical testing of filaments
Tensile testing was done on filaments using a Textechnic Statimat M Test with a gauge length of 100 mm, load cell 10 N and test speed of 300 mm/min. At least five different filaments from the same sample were tested and the results averaged. Where there were particularly large differences between specimens within a given sample set, a larger number of specimens was tested and after allowing for outliers to be discarded, the average value of of ten were reported.

2.4. Flammability testing of fabrics and cast films

Limiting oxygen index (LOI) values were determined on selected PP nanocomposite film samples with thickness of around 0.3~1.5 mm by using the standard ASTM 2863 test procedure [21].

Flame spread tests were undertaken using the test rig described in BS 5438 [22] used for flammability of textile materials. Due to limited sample sizes, sample holder, 190 x 70mm, used in Test 1 of this standard has been used. Samples of this same size were marked at 60, 120 and 180mm intervals. The first 10 mm of sample burning was not taken into account and so times of burning were recorded once the flame had reached a line drawn 10mm from the bottom edge against which the standard flame was applied for 10s as specified in the test. A video film was taken of the burning of each sample from which times to reach 60 ($t_1$), 120 ($t_2$) and 180mm ($t_3$) marks and/or to achieve flameout were noted. Three replicates of each sample were burnt and results averaged. The burning behaviour of each sample was observed and noted.

3. RESULTS AND DISCUSSION

3.1. Structural properties characterisation

3.1.1. X-ray diffraction (XRD)

The XRD curves for matrix I samples, which contain E-107 clay without/with different levels of compatibiliser Polybond 3200 (Pb) are shown in Fig. 1. Although the XRD study was carried out in the range $2\theta = 2 - 60^\circ$ and the curves in Fig.1(a) for samples containing clay and different levels of Pb show significant differences between each other and from
the control polypropylene, only the regions between $2\theta = 2 - 10^\circ$ are analysed in detail and shown in Fig. 1(b). The intergallery d-spacings, analysed from these curves are given in Table 1. The curve for polypropylene (PP) is featureless in this region with no peak. Sample 1, PP-3%E shows a peak at $3.5^\circ$ with $d_{001}$-spacing at 2.53nm, which is slightly greater than the $d_{001}$ value of 2.43nm for E-107 clay (see Fig. 1(b) and Table 1). On adding 1% Pb (sample 2, PP-1%Pb-3%E), there is no increase in intergallery spacing, on the contrary there is a decrease to 2.48nm (See Table 1), which has further decreased with 2% Pb addition (Sample 3). Only after adding 5% Pb (sample 4, PP-5%Pb-3%E), does the d-spacing show an increase (2.98nm), indicating possible intercalation. However, as can be seen from Fig.1(b), the peak intensity decreases with increasing Pb level indicating that exfoliation may have occurred since specimen thicknesses were comparable. With 5% clay present in sample PP-5%E, the peak at $2\theta = 3.62^\circ$ ($d_{001} = 2.48$nm) is very intense and remains unaffected by 1% Pb addition shown for sample PP-1%Pb-5%E. This indicates that 1%Pb is not enough to improve nanodispersion of 5% clay. However and as will be discussed in the later section, the higher compatibiliser levels affect the processability of the compounded polymer into filaments and have a detrimental effect on latter’s physical and mechanical properties. Hence, for the matrix II samples, the compatibiliser and clay levels have been kept at 1 and 3%, respectively.

Cloisite clay 30B has $d_{001}$ spacing = 1.85nm [23] and when introduced into matrix II formulations with Pb and DEP compatibilisers in samples 7 (PP-Pb-30B) and 8 (PP-DEP-30B), the $d_{001}$ spacings have reduced to 1.34 and 1.33 nm, respectively (see Fig. 2(a)). This could be because sample preparation for XRD experiments was done by melt pressing the polymer chips at 210$^\circ$C and at this temperature organic modifier of the clay starts degrading thereby causing a collapse of the clay lamella [24]. On addition of APP into the formulation PP-Pb-30B-APP (sample 9), the clay peak apparently disappears. For the presence of APP to promote clay exfoliation is highly unlikely but its higher concentration (5% w/w) may have enabled its presence to selectively absorb incident x-ray radiation. This cannot, however, be confirmed as peaks are observed in samples PP-Pb-E-APP (see Fig. 2(b) and PP-DEP-PCl-APP (see Fig. 2(c)), although with higher d-spacings compared to respective samples without APP. Clearly the effect of adding poorly dispersed APP is not consistent across the three PP-clay-compatibiliser-containing
samples and it is most likely that any apparent changes to diffraction patterns may be related to differences in particle dispersions and sample heterogeneities present.

Sample 10 (PP-Pb-E) is similar in composition to sample 2, and hence, both have similar gallery spacings, with respective values being 2.48 to 2.49nm. Because matrix II samples were compounded using a different compounder than that used for the matrix I samples (see section 2.2), it was expected that the two sets of samples might have different dispersion levels. However, these results indicate that the different compounding histories have had little effect on dispersion levels although no data exists to be able to compare the actual shear rates present in each compounder. After inclusion of APP in the formulation PP-Pb-E-APP (sample 11), the gallery spacing apparently increases to 3.05 nm (see Fig. 2(b)).

The in-house modified clay (PCI) has a gallery spacing 1.55nm, which remains unaffected in the sample 12 (PP-DEP-PCI). However, the presence of APP in sample 13 (PP-DEP-PCI-APP), increased this to 1.62nm (see Fig. 2(c)).

While 20A clay has gallery spacing of 2.42nm [25], in sample 14 (PP-Pb-20A) this has reduced to 2.31 nm which might indicate some degree of exfoliation. The disappearance of the clay peak on addition of APP to the formulation PP-Pb-20A-APP (sample 15) is similar to that observed above for sample 9 where it is proposed that the APP interferes with the diffracting x-ray beam.

### 3.1.2. Differential Scanning calorimetry

Tables 1 and 2 show the endothermic peak maxima for many of the samples and it can be seen that the clay, graft and APP have little effect on the melting temperature of PP with temperatures ranging between 165 and 169 °C. Previously published work [2] gave a melting temperature of 164°C for PP control.

### 3.1.3 Optical Microscopy

Fig. 3 shows optical microscope images with the effect of graft-PP levels on the morphological structure of the extruded tapes. The spherulite structure is quite coarse in
Fig. 3(a) which has no graft-PP present in the formulation (sample 1, PP-3%E) and the clay particles are easily visible as black images and concentrated at the boundaries between the large spherulitic crystalline aggregates. Once the graft is added, even at the low level of 1% (see Fig. 3(b) for sample 2), the clay particles are smaller with better dispersion. While increasing the graft level to 2% does significantly reduce dispersion further (Fig. 3(c)), at 5% Pb level, sample 4 in Fig. 3(d) shows significant reduction in clay particle numbers and size.

In Figure 4, the effect of different clays at 3% level in the presence of different compatibilisers at 1% level can be seen. Elementis 107, E (Fig.4(c)) and Cloisite 20A (Fig.4(g)) clays appear to be better dispersed than Cloisite 30B (Fig. 4(a)) and PCl clays (Fig. 4(e)). It can be clearly seen from Figs. 4(b,d,f and g) that APP is not well dispersed and its presence has a negative effect on the dispersion of clay. This is contrary to the observation of XRD study where clay peaks either disappeared or the intergallery spacing appeared to increase thus confirming the view that APP particles interfered with any expected clay diffraction patterns.

3.2 Material physical properties - melt flow index and rheology:

It can be seen from Table 1 that the MFI of PP chips as they are received from the manufacturer is 25.9g/600s, however, all other samples reported here have gone through the twin screw extruder. In our previous communication [2] the MFI for PP after one passage through the twin screw extruder extruder has been reported as 36.0g/600s. The increase in MFI is a measure of polymer degradation induced by one extra thermal cycle [2]. With respect to matrix 1 formulations in Table 1, the introduction of 1% of the compatibiliser, Polybond 3200, has little effect on MFI when added to polypropylene containing 3% E-107 clay with an MFI of 38.4 for sample 1 and 39.0 for sample 2. However, increasing the compatibiliser level to 2% (sample 3) brings a reduction in MFI with a fall to 31.8 which then increases to 34.8 for sample 4 containing 5% compatibiliser although samples 5 and 6 also containing compatibilisers show reductions in MFI to 31.8 to 27.6 respectively. This reduction in MFI, if outside of experimental error, is indicative of an increase in viscosity which could be as a result of better dispersion of clay platelets within the polymer matrix. Table 2 shows the results on material properties from different clays plus the inclusion of flame retardant APP in samples 9, 11, 13 and 15. With the
exception of Cloisite 20A, the addition of APP to the mix reduces the MFI. It should also be noted that the choice of compatibiliser has an effect on MFI. Cloisite 30B was compounded with both Polybond and DEP in samples 7 and 8 with reductions in MFI value from 36 for singly-extruded PP to 32.4 and 27.0 g/600s, respectively.

A recently developed rheological method [26] to characterise nanocomposite morphology has been used to study nanodispersions of clays present. Because a classic “card-house-like” structure of montmorillonite-based clays in polymer-layered nanocomposites imparts solid-like viscoelastic properties to polymer-clay nanocomposites any significant increases in dynamic viscosity measurements of polymer-clay composites at lower frequencies suggests nanodispersion of clay platelets [27]. Rheological properties, including complex viscosity, storage loss moduli obtained at lower frequency of 1.0 rad/s for most samples in matrix I and II sets are given in Tables 1 and 2 respectively. It must be noted that in the present study viscoelastic measurements for all samples were performed at 240 °C and since the viscoelastic behaviour of a sample at fixed temperature and strain amplitude is the characteristic feature of that material, the complex viscosity measurements are expected to be reproducible.

It can be seen from the data in Table 1 and Figure 5 that the complex viscosity for the control PP(1000 Pas) is reduced to 203 Pas in PP-2%Pb-3%E (203 Pas) sample and this may be attributed to the addition of Polybond which acts as a plasticiser in spite of the presence of 3% clay. This also suggests that 2% of compatibiliser is not sufficient to facilitate nanodispersion of clay platelettes. However, on increasing to 5% Polybond in the PP-5%Pb-3%E sample, the complex viscosity has significantly increased to 3081 Pas, indicating that the clay platelets are nanodispersed. This is consistent with the XRD results for PP-5%Pb-3%E which indicate possible intercalation of PP chains in the clay galleries. Combining the XRD results with rheological behaviour of PP-5%Pb-3%E , it can be concluded that PP-5%Pb-3%E is an intercalated nanocomposite.

On the other hand, data for the PP-1%Pb-5%E sample, which also exhibits similar viscoelastic behaviour to PP-5%Pb-3%E over entire range of frequencies tested (see Fig. 5) and suggesting improved dispersion of clay platelets, is contradictory to the XRD data which suggests that the there is no change in d_001-spacing of clay platelets. This discrepancy can probably be attributed to the deficiencies of XRD techniques where
preferred orientation effects can give the false perception that intercalation/exfoliation has not occurred [28, 29].

Rheological data in Table 2 and Figure 6 shows the effect of addition of different clays and of the flame retardant APP. In general, the viscosity of PP-clay composites increases with the inclusion of APP. However, the magnitude of increasing viscosity depends on the type of functionalisation of the nanoclay. It can be noted from Table 2 that compared to PP-DEP-30B, the complex viscosity of PP-Pb-30B-APP in the low frequency region (1 rads/s) has increased significantly from 492 to 1108 Pas. Assuming similar rheological behaviours of PP-Pb-30B and PP-DEP-30B samples on the basis that d-spacings for both the samples are almost similar (d_{001} = 1.34 nm), it can suggested that inclusion of APP in PP-Pb-30B-APP has facilitated dispersion of clay platelets. This is further reinforced by complete disappearance of peak in the X-ray diffractogram in Fig 2(a). Increase in viscosities of samples containing Cloisite 20A and PCI clays with APP is in agreement with the XRD data which suggests nanodispersion of clay. The presence of APP in samples containing Elementis 107 does not show significant change in viscoelastic properties compared to the one without APP.

Transmission electron microscopic studies of Cloisite 20A clay- and Polybond compatibiliser- containing samples, discussed in some detail in Part I of this study [2], showed that although these clays are not truly intercalated or exfoliated, dispersion is at nanolevel, i.e., particle size << 1 \mu m. Typical clay particle widths appeared to be < 0.1 \mu m with lengths up to 0.5 \mu m [2], hence, the clays are nanodispersed. While we have extended the number of clays and compatibilisers and included flame retardant in this current study with a major focus on studying the resulting filament properties, no further TEM work has been undertaken at this stage.

3.3. Physical properties of filaments and derived fabrics

The matrix I compounded polymers (samples 1 - 6) in Table 3, all containing E-107 clay, extruded quite well into filaments and, with the exception of sample 6, it was possible to maintain a draw ratio of 6:1. Although the draw ratio was maintained for samples 1 – 5, there is considerable variation in the linear density of fibres, ranging from 2.5 tex for sample 3 to 5.0 tex for sample 5. This indicates a great physical variability in the
filaments which is not unexpected from compounded polymers that were not dispersed at the nanoscale. Furthermore, while experimental errors associated with tensile value may be quite large with respect to changes in that value anticipated by changes in filament composition and history, there are general trends of change which are consistent and the discussion below is undertaken within this context. In Table 3, the physical property values for pure PP filaments are taken from our previously reported work [2]. An increase in modulus can be an indication of nanodispersion [2], therefore the addition of compatibiliser to the mix should improve the dispersion of clay and may increase modulus. This is seen to be the case when clay alone is added at 3% level (sample 1) and not at the higher 5% level (sample 5). However, the addition of 1% Polybond has no effect on the modulus of sample 2, but increasing the level to 2% increases the modulus from 4.4 to 5.9 N/tex. A further increase in the Polybond level to 5% shows a decrease in modulus to 3.7 N/tex. In a previous study, higher levels of compatibiliser were seen to reduce filament modulus, which was thought to be because of higher cross-link formation [2], but here the reduction does not occur until the compatibiliser level has reached 5%.

Tenacity values are much lower for samples 5 and 6 containing 5% clay than those with 3% clay. The introduction of the compatibiliser reduces tenacity at both clay levels. The samples containing 3% E clay have a reduction in tenacity from 36.8 to 25.0 cN/tex when 5% Polybond is present (sample 4). Similarly the addition of 1% Pb with 5% clay reduces the tenacity from 20.5 to 17.5 cN/tex in samples 5 and 6 respectively. Elongation-at-break values are also reduced on the addition of compatibilisers with values down from approximately 40% in pure polypropylene and sample 5 to around 12-19% for samples 1, 2, 3, 4 and 6.

These experimental filaments were very difficult to knit into fabric samples because of filament property variations. However, notwithstanding these difficulties, fabric samples were knitted with area densities ranging from 194 g/m² to 345 g/m² indicating that filaments with the highest linear density did not always knit into fabrics with the greatest area density. Some of the filaments proved so difficult to knit because of filament breakage that the knitting tension had to be reduced to ensure the production of some fabric samples, resulting in variation in their area densities.
Observation of the matrix II filament results in Table 4 in which different clays are present as well as APP show much more variable results than those in Table 3. More variation is shown between sample filament linear densities as well. The variation occurs because processing difficulties during filament formation, possibly caused by the relatively different particle sizes of some additives (eg for APP average particle sizes are quoted by the manufacturers to be 25-30µm), it was only possible to maintain a draw ratio of 6:1 for samples 12 and 15. Where variations were particularly large, 10 replicates were tested and any large outliers in the results discarded. It was found for most filaments that 5 replicates were suitable. The modulus is reduced significantly after addition of the flame retardant, APP, but the size of the reduction is dependent on the clay used in compounding. In filaments containing Cloisite 20A (samples 14 and 15), the reduction in modulus is negligible but in filaments containing Cloisite 30B (samples 7, 8 and 9) the reduction is larger (> 1.5N/tex) and outside experimental error. The most consistent results for tenacity are with the two samples containing Cloisite 20A (samples 14 and 15) which are quite unexpected as they have different draw ratios; hence and surprisingly, all the physical results are very similar for these two filament samples. The remaining samples show variation with results ranging from 7.6 to 30.9 cN/tex. Apart from Cloisite 20A-containing filaments (samples 14 and 15), tenacity values are significantly reduced with the addition of APP into the polymer mix. This is probably the result of APP particle size being too large for filament formation plus poor dispersion of all additives in the polymer matrix, although this issue did not compromise the 20A-containing filaments.

Elongation-at-break values are similar for most filament samples averaging at 13.5% ± 4% except for samples 9 and 11, both compounded with APP and sample 10, compounded with the compatibiliser Pb. The high value of sample 9 is a consequence of its lower draw ratio.

In Figure 7, SEM images of filaments show that there is little apparent difference between sample 1 (Fig. 7(a)) without compatibiliser and 2 with compatibiliser (Fig. 7(b)). Filaments and fibres are particularly difficult to observe under both the optical microscope and SEM because of their generally featureless circular shape and transparency. Despite this Figs. 7(c) and 7(d) show evidence of particles of APP within the filament which demonstrates that the APP has not dispersed well in the polymer matrix.
3.4. Flammability properties of films and fabrics

3.4.1 Limiting oxygen index of films
The samples that were submitted for LOI testing were tested as extruded tapes and the results are given in Table 5. All samples yield similar results which show that the clay has little effect on the burning properties of the PP samples. It would have been expected that samples containing APP would show some resistance to burning with higher LOI values although normal bulk PP samples require APP levels >15% before significant increases in LOI occur [1].

3.4.2 Flammability of knitted fabrics
In our previous work the cone calorimeter was used to determine the burning behaviour of PP/nanoclay films and fabrics [2]. While this is normally a very effective way of studying the burning behaviour, it is difficult to obtain accurate reproducible results from thermally thin materials such as fabrics and films. Therefore in this work a small scale flame spread test was developed to collect data such as burning time and mass loss as well as information fabric sample burning behaviour. A general observation of the knitted samples when introduced to a flame was an initial shrinking, ignition and burning of the sample vertically up the centre of the sample. The flame would then start to spread out and proceed up the side edges releasing burning drips as flames rose. As the burning PP dripped off the sample the remaining PP would start to twist and curl back to itself introducing the flame to the remainder of the sample.

Timings and mass losses for all the samples burnt in this study are shown in Table 5. Samples 1-6, where effects of clay and compatibiliser levels are assessed, show little difference in the time taken to reach the 60mm mark with values ranging from 3.0 – 4.6 seconds. The time taken to reach the second mark at 120mm varies between sample 4, which self-extinguished before reaching this mark to sample 2, which took an average time of 45s to reach the 120mm mark. Although many samples reached this mark, a few went on to burn up completely. The 100% PP sample burns completely in approximately 59s with much dripping and the remaining fabric twisting back on itself, spreading the flame further up the fabric strip. The introduction of the clay does seem to delay the spread of the flame with samples 5 and 6 containing 5% clay; these self-extinguish after 28 and 22 seconds respectively. Sample 4, PP-5%Pb-3%E, was the only sample that
failed to burn to the 120mm mark but still lost 63% mass, whereas sample 1, PP-3%E reached the 120mm mark in 28.9 seconds but only lost 20% mass. This demonstrates how little or how much the flame spreads across the width of the fabric strip. If the total mass loss is low, this indicates that the flame has just spread vertically up the centre of the fabric strip with little dripping occurring, but higher total mass loss values indicate that the flame not only burns vertically up the centre of the strip but also spreads horizontally or continues burning up the side strips of the fabric. It can be seen from Table 5 that only the control PP and the sample containing 1% Pb and 3%E burned to the 180mm mark. By conversion to rates of burning and plotting against burning length as in Figure 8, a better distinction between the individual performances may be gained. For matrix I fabric samples, 100% polypropylene and PP-1%Pb-3%E fabrics burn faster than the others followed by PP-3%E sample. The flame spread rates for samples containing compatibiliser are relatively slower with the slowest behaviour shown by PP-5%Pb-3%E sample. However, between 60 and 120mm, PP-1%Pb-3%E burns slower than others.

Samples containing different types of clays burned differently depending upon the clay type as can be seen from Table 5 and Fig 8(b) for matrix II fabric samples. All samples containing only clay and compatibiliser except PP-Pb-20A (sample 14), namely PP-Pb-30B (sample 7), PP-DEP-30B (sample 8), PP-Pb-E (sample 10) and PP-DEP-PCl (sample 12) self-extinguished beyond 60mm but before 120 mm. The best results in terms of lowest burning rates are shown by samples PP-Pb-30B and PP-DEP-PCl, which had burning times to 60 mm of 7.8 and 7.0 seconds respectively. The total mass loss for these samples is also very low (4 and 1.5%), which shows that they did not burn extensively and little dripping occurred.

Samples containing APP (samples 9, 11, 13 and 15) burned up to the 60 mm mark quickly over the range 3.5 – 4.7 s and when observed burnt quite readily and vigorously, although the flame did flicker quite significantly, which was not observed in other samples. This phenomenon was probably due to the poor dispersion and low level of APP. Poor dispersion would mean the flame would flicker when it reached a particle of APP but the level was not high enough to extinguish the flame. It can be seen from Table 5 that generally most of the samples 7-15 have lower mass loss than samples 1-6, which can be explained by the better dispersion of clay in samples 7-15 which were compounded in a different twin screw extruder from the earlier matrix I samples.
4. CONCLUSIONS

It is observed that the nanoclay is difficult to disperse in extremely non-polar polypropylene, which however can be improved by adding compatibiliser like maleic anhydride-grafted polypropylene. This study has shown that with increasing compatibiliser, the clay dispersion improves although higher compatibiliser levels affect the processability of the compounded polymer into filaments, having detrimental effect on latter’s physical and mechanical properties. The nanoclays are not truly intercalated or exfoliated, however, optical microscopic evidence shows an improvement in dispersion at the microlevel. This observation is consistent with the transmission electron microscopic study of selected samples discussed in Part I of this study [2], where there was no evidence of intercalation or exfoliation, but the dispersion at nanolevel could be observed.

With regard to flammability and as expected when present alone, LOI values are not changed following addition of clay and even in presence of low levels (5%w/w) of the flame retardant, ammonium polyphosphate. However, the presence of the clay changes the burning behaviour and fabric becomes self-extinguishing. In presence of ammonium polyphosphate, burning behaviour became more erratic with no significant improvement, which is due to poor dispersion of this flame retardant in the mix.

In conclusion, our study shows that while true nanocomposite structures could not be attained, some degree of nanodispersion was achieved and both micro- and nanodispersed polymers were processable and could be extruded into filaments, which were sufficiently strong to be knitted into fabrics having slower burn properties than the pure polymer.

Acknowledgements
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REFERENCES

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23. www.nanoclay.com, Southern Clays Corp. MSDS sheet Cloisite 30B

25. www.nanoclay.com, Southern Clays Corp. MSDS sheet Cloisite 20A.


Captions for Tables

Table 1 Effect of clay and graft levels on material properties : matrix 1 sample composition, XRD, MFI, DSC and dynamic viscometric results of polymer chips

Table 2 Effect of different clays on material properties : matrix 2 sample composition, XRD, MFI, DSC and dynamic viscometric results of polymer chips

Table 3 Effect of clay and graft levels on the matrix I sample fibre and fabric properties

Table 4 Effect of different clays, compatibilsers and flame retardant on the matrix II fibre and fabric properties

Table 5 Flammability properties of films and fabrics
Captions for Figures

Figure 1. XRD curves for matrix 1 samples 1-6 showing the effect of clay and graft levels on dispersion

Figure 2. XRD curves showing reduction in Cloisite 30B, E-107, PCl and 20A clay peak intensities for matrix II samples

Figure 3. Optical micrographs (x200)

Figure 4. Optical micrographs (x200) of tape samples showing the effects of different clays and presence of the flame retardant APP

Figure 5. Effect of compatibilser levels on complex viscosity

Figure 6. Effect of clays and flame retardant on complex viscosity

Figure 7. SEM images of selected extruded filaments

Figure 8. Burning rates for a) matrix 1 and b) matrix II fabric samples.
Table 1 Effect of clay and graft levels on material properties: matrix 1 sample composition, XRD, MFI, DSC and dynamic viscometric results of polymer chips

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Sample</th>
<th>Sample composition w/w</th>
<th>Graft Pb, % (w/w)</th>
<th>E-107 % (w/w)</th>
<th>XRD d-spacing nm*</th>
<th>DSC Endo. peak maxima °C</th>
<th>MFI g/600 s</th>
<th>Rheological properties at 1 rad/s and 240°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Storage modulus (G’) Pa</td>
</tr>
<tr>
<td>1</td>
<td>PP-3%E</td>
<td>0</td>
<td>0</td>
<td>-</td>
<td>-</td>
<td>164</td>
<td>25.9</td>
<td>1384</td>
</tr>
<tr>
<td>2</td>
<td>PP-1%Pb-3%E</td>
<td>0</td>
<td>1</td>
<td>3</td>
<td>2.53; 1.27</td>
<td>166</td>
<td>38.4</td>
<td>**</td>
</tr>
<tr>
<td>3</td>
<td>PP-2%Pb-3%E</td>
<td>1</td>
<td>3</td>
<td>3</td>
<td>2.48; 1.25</td>
<td>167</td>
<td>39.0</td>
<td>**</td>
</tr>
<tr>
<td>4</td>
<td>PP-5%Pb-3%E</td>
<td>2</td>
<td>3</td>
<td>5</td>
<td>2.45; 1.26</td>
<td>165</td>
<td>31.8</td>
<td>158</td>
</tr>
<tr>
<td>5</td>
<td>PP-5%E</td>
<td>2</td>
<td>5</td>
<td>5</td>
<td>2.98, 2.46; 1.22</td>
<td>167</td>
<td>34.8</td>
<td>413</td>
</tr>
<tr>
<td>6</td>
<td>PP-1%Pb-5%E</td>
<td>1</td>
<td>5</td>
<td>1</td>
<td>2.43; 1.24</td>
<td>167</td>
<td>27.6</td>
<td>1716</td>
</tr>
</tbody>
</table>

Note: * $d_{001}$ for clay E-107 = 2.43  
** = Test not performed
Table 2: Effect of different clays on material properties: matrix 2 sample composition, XRD, MFI, DSC and dynamic viscometric results of polymer chips

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Sample</th>
<th>Sample composition</th>
<th>Sample composition</th>
<th>Sample composition</th>
<th>XRD d-spacing (nm*)</th>
<th>DSC Endo. peak max. °C</th>
<th>MFI g/600s</th>
<th>Rheology properties at 1 rad/s and 240°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Graft 1% (w/w)</td>
<td>Clay 3% (w/w)</td>
<td>FR 5% (w/w)</td>
<td></td>
<td></td>
<td></td>
<td>Storage modulus (G''), Pa</td>
</tr>
<tr>
<td>7</td>
<td>PP-Pb-30B</td>
<td>Pb 30 B</td>
<td>-</td>
<td>1.34</td>
<td>168</td>
<td>32.4</td>
<td>**</td>
<td>**</td>
</tr>
<tr>
<td>8</td>
<td>PP-DEP-30B</td>
<td>DEP 30 B</td>
<td>-</td>
<td>1.33</td>
<td>**</td>
<td>27.0</td>
<td>90</td>
<td>483</td>
</tr>
<tr>
<td>9</td>
<td>PP-Pb-30B-APP</td>
<td>Pb 30 B APP</td>
<td>-</td>
<td>166</td>
<td>25.2</td>
<td>9</td>
<td>1108</td>
<td>1108</td>
</tr>
<tr>
<td>10</td>
<td>PP-Pb-E</td>
<td>Pb E107</td>
<td>-</td>
<td>2.49; 1.26</td>
<td>169</td>
<td>27.6</td>
<td>166</td>
<td>827</td>
</tr>
<tr>
<td>11</td>
<td>PP-Pb-E-APP</td>
<td>Pb E107 APP</td>
<td>3.05</td>
<td>**</td>
<td>24.6</td>
<td>151</td>
<td>981</td>
<td>994</td>
</tr>
<tr>
<td>12</td>
<td>PP-DEP-PCI</td>
<td>DEP PCI</td>
<td>-</td>
<td>1.55</td>
<td>**</td>
<td>33.6</td>
<td>60</td>
<td>12</td>
</tr>
<tr>
<td>13</td>
<td>PP-DEP-PCI-APP</td>
<td>DEP PCI APP</td>
<td>1.62</td>
<td>**</td>
<td>30.0</td>
<td>13</td>
<td>3487</td>
<td>3490</td>
</tr>
<tr>
<td>14</td>
<td>PP-Pb-20A</td>
<td>Pb 20A</td>
<td>-</td>
<td>2.31</td>
<td>165</td>
<td>26.4</td>
<td>28</td>
<td>150</td>
</tr>
<tr>
<td>15</td>
<td>PP-Pb-20A-APP</td>
<td>Pb 20A APP</td>
<td>-</td>
<td>166</td>
<td>31.2</td>
<td>137</td>
<td>951</td>
<td>1108</td>
</tr>
</tbody>
</table>

Note:  
* $d_{001}$ for clays: 30B = 1.85; E-107 = 2.43; PCI = 1.55; 20A = 2.42 nm  
** = Test not performed
Table 3 Effect of clay and graft levels on the matrix I sample fibre and fabric properties

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Sample</th>
<th>Fibre properties</th>
<th>Fabric area density, g/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Linear density, tex</td>
<td>Modulus, N/tex</td>
</tr>
<tr>
<td>1</td>
<td>PP*</td>
<td>5.4</td>
<td>3.9±0.5</td>
</tr>
<tr>
<td>2</td>
<td>PP-3%E</td>
<td>3.4</td>
<td>4.4±0.6</td>
</tr>
<tr>
<td>3</td>
<td>PP-1%Pb-3%E</td>
<td>4.5</td>
<td>4.4±1.0</td>
</tr>
<tr>
<td>4</td>
<td>PP-2%Pb-3%E</td>
<td>2.5</td>
<td>5.9±2.8</td>
</tr>
<tr>
<td>5</td>
<td>PP-5%Pb-3%E</td>
<td>2.8</td>
<td>3.7±1.4</td>
</tr>
<tr>
<td>6</td>
<td>PP-1%Pb-5%E</td>
<td>3.8</td>
<td>2.8±0.5</td>
</tr>
</tbody>
</table>

* = Values for PP from Ref 2
### Table 4. Effect of different clays, compatibilisers and flame retardant on the matrix II fibre and fabric properties

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Sample</th>
<th>Draw ratio</th>
<th>Linear density, tex</th>
<th>Modulus (N(tex))</th>
<th>Tenacity, cN/tex</th>
<th>Elongation-at-break, %</th>
<th>Fabric area density g/m²</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>PP-Pb-30B</td>
<td>5:1</td>
<td>4.4</td>
<td>2.7±0.3</td>
<td>14.8±2.0</td>
<td>9.5±1.7</td>
<td>333</td>
</tr>
<tr>
<td>8</td>
<td>PP-DEP-30B</td>
<td>5:1</td>
<td>4.8</td>
<td>2.8±0.6</td>
<td>17.5±3.5</td>
<td>13.1±6.0</td>
<td>302</td>
</tr>
<tr>
<td>9</td>
<td>PP-Pb-30B-APP</td>
<td>3:1</td>
<td>11.7</td>
<td>1.2±0.4</td>
<td>7.6±2.0</td>
<td>296.0±120</td>
<td>279</td>
</tr>
<tr>
<td>10</td>
<td>PP-Pb-E</td>
<td>5:1</td>
<td>5.8</td>
<td>2.6±0.6</td>
<td>9.9±2.6</td>
<td>41.9±25</td>
<td>4.8</td>
</tr>
<tr>
<td>11</td>
<td>PP-Pb-E-APP</td>
<td>5:1</td>
<td>4.5</td>
<td>1.9±0.9</td>
<td>15.9±5.0</td>
<td>64.5±30</td>
<td>325</td>
</tr>
<tr>
<td>12</td>
<td>PP-DEP-PCl</td>
<td>6:1</td>
<td>3.6</td>
<td>2.4±0.4</td>
<td>30.9±2.3</td>
<td>13.1±5.0</td>
<td>258</td>
</tr>
<tr>
<td>13</td>
<td>PP-DEP-PCl-APP</td>
<td>3:1</td>
<td>7.7</td>
<td>2.6±0.6</td>
<td>13.6±1.6</td>
<td>8.4±1.3</td>
<td>463</td>
</tr>
<tr>
<td>14</td>
<td>PP-Pb-20A</td>
<td>3:1</td>
<td>3.7</td>
<td>3.3±0.6</td>
<td>22.6±4.2</td>
<td>16.3±4.0</td>
<td>253</td>
</tr>
<tr>
<td>15</td>
<td>PP-Pb-20A-APP</td>
<td>6:1</td>
<td>3.7</td>
<td>3.2±0.8</td>
<td>23.9±2.7</td>
<td>17.4±2.2</td>
<td>260</td>
</tr>
</tbody>
</table>
Table 5. Flammability properties of films and fabrics

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Sample</th>
<th>Film LOI (vol%)</th>
<th>Fabric</th>
<th>Time to 60mm mark (s)</th>
<th>Time to 120mm mark (s)</th>
<th>Time to 180mm mark (s)</th>
<th>Flame out (s)</th>
<th>Total mass loss (%)</th>
<th>Burning behaviour</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PP</td>
<td>20.1</td>
<td></td>
<td>3.0</td>
<td>37</td>
<td>59</td>
<td>59</td>
<td>80</td>
<td>Burns up the middle then flames spread to sides, flaming drips</td>
</tr>
<tr>
<td>1</td>
<td>PP-3%E</td>
<td>*</td>
<td>3.1</td>
<td>29</td>
<td>-</td>
<td>37</td>
<td>20</td>
<td></td>
<td>Curls and drips, self-extinguishes</td>
</tr>
<tr>
<td>2</td>
<td>PP-1%Pb-3%E</td>
<td>20.3</td>
<td>4.6</td>
<td>45</td>
<td>49</td>
<td>76</td>
<td>70</td>
<td></td>
<td>Burns up the middle, flames spread to the sides while sample twists and curls</td>
</tr>
<tr>
<td>3</td>
<td>PP-2%Pb-3%E</td>
<td>20.2</td>
<td>3.6</td>
<td>22</td>
<td>-</td>
<td>40</td>
<td>31</td>
<td></td>
<td>Edges burn and twist, self-extinction before 180mm mark</td>
</tr>
<tr>
<td>4</td>
<td>PP-5%Pb-3%E</td>
<td>20.3</td>
<td>3.8</td>
<td>-</td>
<td>-</td>
<td>29</td>
<td>63</td>
<td></td>
<td>Flames and drips, self-extinction</td>
</tr>
<tr>
<td>5</td>
<td>PP-5%E</td>
<td>20.6</td>
<td>3.8</td>
<td>17</td>
<td>-</td>
<td>28</td>
<td>48</td>
<td></td>
<td>Burns, melts and drips</td>
</tr>
<tr>
<td>6</td>
<td>PP-1%Pb-5%E</td>
<td>20.5</td>
<td>3.0</td>
<td>14</td>
<td>-</td>
<td>22</td>
<td>33</td>
<td></td>
<td>Stops burning when sides burn through and accompanying drips fall</td>
</tr>
<tr>
<td>7</td>
<td>PP-Pb-30B</td>
<td>20.0</td>
<td>7.8</td>
<td>-</td>
<td>-</td>
<td>27</td>
<td>14</td>
<td></td>
<td>Self-extinguishes</td>
</tr>
<tr>
<td>8</td>
<td>PP-DEP-30B</td>
<td>*</td>
<td>4.0</td>
<td>-</td>
<td>-</td>
<td>22</td>
<td>4</td>
<td></td>
<td>Burns up centre but self-extinguishes</td>
</tr>
<tr>
<td>9</td>
<td>PP-Pb-30B-APP</td>
<td>20.9</td>
<td>4.7</td>
<td>24</td>
<td>-</td>
<td>52</td>
<td>40</td>
<td></td>
<td>Burns erratically and drips</td>
</tr>
<tr>
<td>10</td>
<td>PP-Pb-E</td>
<td>19.9</td>
<td>4.3</td>
<td>-</td>
<td>-</td>
<td>33</td>
<td>40</td>
<td></td>
<td>Shrinks away from flame but extinguishes</td>
</tr>
<tr>
<td>11</td>
<td>PP-Pb-E-APP</td>
<td>20.3</td>
<td>4.4</td>
<td>-</td>
<td>-</td>
<td>26</td>
<td>19</td>
<td></td>
<td>Drips, twists and burns to approx half way</td>
</tr>
<tr>
<td>12</td>
<td>PP-DEP-PCl</td>
<td>*</td>
<td>7.0</td>
<td>-</td>
<td>-</td>
<td>15</td>
<td>1.5</td>
<td></td>
<td>Melts away from flame then self-extinguishes</td>
</tr>
<tr>
<td>13</td>
<td>PP-DEP-PCl-APP</td>
<td>*</td>
<td>4.7</td>
<td>-</td>
<td>-</td>
<td>28</td>
<td>13</td>
<td></td>
<td>Self-extinguishes after burning erratically</td>
</tr>
<tr>
<td>14</td>
<td>PP-Pb-20A</td>
<td>20.2</td>
<td>5.4</td>
<td>36</td>
<td>-</td>
<td>44</td>
<td>50</td>
<td></td>
<td>Melts, twists, drips then self-extinguishes</td>
</tr>
<tr>
<td>15</td>
<td>PP-Pb-20A-APP</td>
<td>20.5</td>
<td>3.5</td>
<td>-</td>
<td>-</td>
<td>24</td>
<td>33</td>
<td></td>
<td>Flame burns up fabric, twists, melts then self-extinguishes about half way up</td>
</tr>
</tbody>
</table>

* = Test not performed
Figure 1. XRD curves for matrix 1 samples 1-6 showing the effect of clay and graft levels on dispersion.

a) 

b) 

Figure 1. XRD curves for matrix 1 samples 1-6 showing the effect of clay and graft levels on dispersion.
Figure 2 XRD curves showing reduction in Cloisite 30B, E-107, PCl and 20A clay peak intensities for matrix II samples
Figure 3. Optical micrographs (x200) of tape samples showing the effects of graft-PP and graft levels on dispersion of clay.
a) Sample 7; PP-Pb-30B

b) Sample 9; PP-Pb-30B-APP

c) Sample 10; PP-Pb-E

d) Sample 11; PP-Pb-E-APP

e) Sample 12; PP-DEP-PCl

f) Sample 13; PP-DEP-PCl-APP
Figure 4. Optical micrographs (x200) of tape samples showing the effects of different clays and presence of the flame retardant APP.

g) Sample 14; PP-Pb-20A

h) Sample 15; PP-Pb-20A-APP
Figure 5 Effect of compatibiliser levels on complex viscosity
Figure 6 Effect of clays and flame retardant on complex viscosity
Figure 7. SEM images of selected extruded filaments
Figure 8. Burning rates for a) matrix 1 and b) matrix II fabric samples.