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Research Article

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Flame retardant PP/PA6 blends: A recipe for recycled wastes

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Abstract: Disposal of plastics in the form of discarded scrap or waste is an environmental nightmare to the developed and under-developing countries. In this sense, modern technologies are every day growing; meanwhile recycling wastes are taking credit for higher performance to pave the way towards a cleaner planet. Recycling of polymer blends is associated with serious complexities in finding appropriate additives acting in each phase or at the interface of non-miscible polymers to attain materials with higher properties. A flame retardant system was designed and intended to play the role of typical recycled flame retardant polypropylene/polyamide (PP/PA6) blends by incorporation of two kinds of talc, sepiolite, and a phosphorus-based flame retardant. First, two types of talcs having different physical properties were added to PA6 in combination with melamine cyanurate (MC) or melamine pyrophosphate (MPP) to find the one acting as a barrier during combustion. Then, high-aspect-ratio talc that appeared more effective in the first-stage survey was then used in combination with MC, MPP and ammonium polyphosphate (APP) together with a compatibilizer for PP/PA6 blend. Then, thermogravimetric analysis (TGA), microcalorimeter of combustion (PCFC) and cone calorimeter measurements were performed to study thermal degradation and flammability behavior. The results are indicative of successful formulation of flame retardant system containing the aforementioned precursors to be intended to the recycled PP/PA6 blends.

Keywords: Recycling; PP/PA6 blend; Flame retardancy; Talc

1 Introduction

Demand for plastics has always been experiencing an ascending trend over the past century; meanwhile commercialization of engineering plastics has undergone a significant progress. By the time new grades of polymers have been under commercialization, recycling of plastics has become a mission of scientists and companies to obliterate environmental concerns [1, 2]. Disposal of plastics in the landfills and water resources has become the nightmare of the governments. The lack of information on recycling, cost-ineffectiveness of operations from economic standpoint, and generic inability to removal of contaminations or even screening wastes for separation of blended plastics were known as other barriers to efficient polymer recycling [3, 4]. Therefore, attempts have been made to enhance the performance of waste/recycled plastics as they are for particular applications by addition of appropriate additives/fillers [5]. In this regard, design and manufacture of flame retardant polymer materials is a dynamic area in which the performance of a wide variety of additives -both organic and inorganic- having physical or chemical actions against flame has given a particular dimension in the realm of recycling for the sake of higher performance [6, 7].

Polyamide 6 (PA) is an engineering plastic with superior characteristics, which has been frequently considered for automotive, textile, and other applications because of its high durability and strength [8]. Despite such beneficial aspects, PA6 is sensitive to moisture and temperature, and also it is notch-sensitive; hence, it is customary to blend it with commodity plastics known for their high ductility and impact resistance as well as their low sensitivity to moisture [9]. Polypropylene (PP) is one of these commodity plastics, also known for its good mechanical proper-

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ties [10], biocompatible nature [11], antibacterial characteristics [12], appropriate processability [13], and resistance against almost all solvents at ambient temperature- what give reason to consider PP for packaging, textile, and piping applications [14, 15], but PP suffers from poor fire reaction [16]. There has been continued interest in blending PP and PA6 over decades for their complimentary characteristics [17, 18] at laboratory scale but also at industrial one such as Orgalloys[®] from Arkema. According to the statistics, the blend of PP/PA is among the earliest commercial blends [19]. Since these plastics show complete phase separation due to their different nature, the use of a coupling agent/compatibilizer to tailor them into a compatible blend or even adding nanofillers like nanoclay for packaging industry [20].

From a fire retardancy standpoint, PA is a char-forming plastic with considerable chemical reactivity toward melamine and melamine cyanurate (MC) flame retardant (FR) additives that can properly be dispersed throughout the PA matrix to expedite volatilization at lower temperatures [21, 22]. On the other hand, PP can be easily degraded during the processing due to its excessive thermal oxidation [23]. The use of phosphorus-based FR additives, *e.g.* ammonium polyphosphate (APP) [24], and inorganic additives such as talc [25] and montmorillonite (MMT) [26], alone or concurrently [27], was recognized as a solution to low mechanical [28] and flame retardancy of PP [29]. Tang *et al.* elaborated mechanistically combustion of PP/PA compatibilized blends containing APP and MMT, and reported synergistic FR function between APP and MMT by highlighting the flame retardancy effect of PA on PP [30, 31]. Kinetic studies performed by Almeras *et al.* on PA/PP blends containing APP FR additive and poly(vinyl alcohol-co-ethylene) copolymer as coupling agent confirmed FR role of PA on fire retardancy of PP [30, 32]. Similar results were reported by Ma *et al.* for PP/PA blends compatibilized with carboxylated PP in the presence of organo-modified MMT through morphological, calorimetric, rheological, and char content analyses [33]. However, studies revealed that acceptable resistance against fire and thermal decomposition in such systems containing one kind of FR, *e.g.* magnesium hydroxide necessitates the use of high loadings up to 50%, which is sometimes detrimental to mechanical properties [34].

Technologically saying, PP/PA6 blends can be retrieved from the dismantling of vehicles as well as from end-of-life floorings and carpets. Their waste management is particularly a major issue when both plastics are associated in complex parts or multilayered materials. In this sense, the influence of three-layered silicates containing sepiolite, halloysite and organo-modified MMT in combi-

nation with APP on fire retardancy of PP/PA6 blends was compared in a previous work aiming to the direct reprocessing of these blends without separation. The results suggested that the combination of sepiolite and APP improves fire retardancy through the formation of a large amount of charred and expanded residue through the interaction between the used additives during the combustion. In this work, a FR system is designed on the basis of a typical recycled PP/PA6 blend and two kinds of talc additives, and melamine pyrophosphate (MPP) and MC are applied in formulation in the presence of a commercial compatibilizer through a two-stage recipe. In the first stage, two types of talc additives having different physical properties were added to PA6 in combination with MC or MPP to choose the more reactive one having higher barrier action against fire. Then, high-aspect-ratio or ultrafine talcs were identified and used in combination with APP, MC, MPP, and compatibilizer to PP/PA6 blend for fire retardancy measurements. Thermogravimetric analysis (TGA), PCFC and cone calorimeter measurements were performed to study thermal degradation and flammability behavior of the prepared blend samples, respectively.

2 Experimental

2.1 Materials

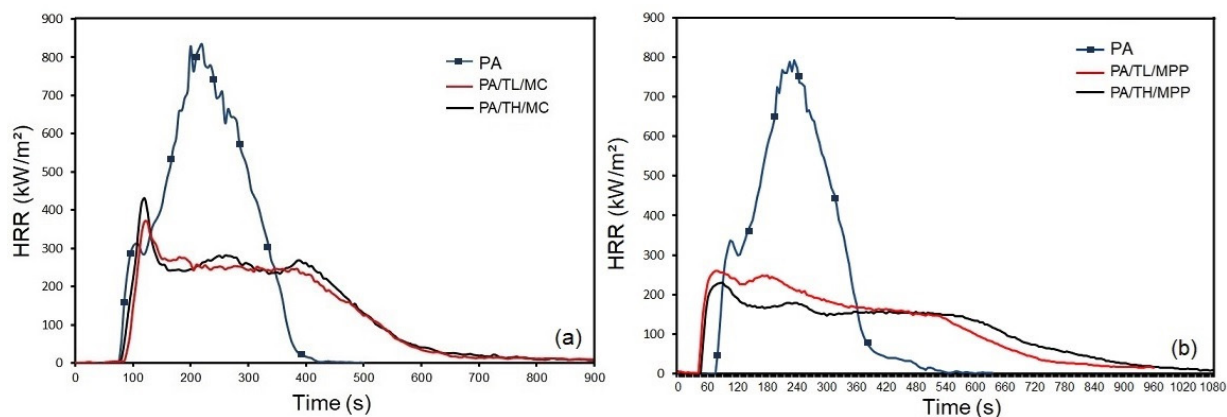
Polypropylene (PP) (SABIC[®]-576P) and polyamide 6 (PA 6) (Rhodia C216-Technyl[®]) were used as blend components. Styrene-ethylene-butylene-styrene grafted with maleic anhydride FG 1901X (SEBS-g-MA) was purchased from Shell Kraton. Other additives and flame retardants were melamine cyanurate, Melapur MC15 (MC) (BASF), melamine pyrophosphate (MPP) (Budit 311MPP (Buddenheim)), ammonium polyphosphate (APP) (Clariant AP 412), and two types of talc from Imerys, namely HAR T84 and Jetfine 0.7CA, hereafter referred to as TH and TL, respectively (Table 1).

Table 1: Properties of HAR (TH) and Jetfine talcs (TL)

Talc type	Median diameter Laser Diffraction (μm)	Median diameter Laser Diffraction (μm)	Specific Surface Area (m^2/g)
TH	10.5	2	19.5
TL	2.5	0.7	19.0

Table 2: Sample names and compositions

Sample code	PA6 (wt.%)	Talc HAR (TH) (wt.%)	Talc Jetfine (TL) (wt.%)	MC (wt.%)	MPP (wt.%)
PA6	100	-	-	-	-
PA/TH/MC	50	25	-	25	-
PA/TL/MC	50	-	25	25	-
PA/TH/MPP	50	25	-	-	25
PA/TL/MPP	50	-	25	-	25
PA/TH/TL/MC/MPP	50	25	-	12.5	12.5

**Figure 1:** Heat release rate curves of (a) samples containing PA/MC/talcs (b) PA/MPP/talcs, obtained from cone calorimeter test.

2.2 Samples preparation and characterization

The samples were prepared in a twin-screw extruder (corotative-Clextal BC 21) working at rotor speed of 150 rpm with a temperature profile of 210–230°C from feed to die zones. The extrudate was then pelletized and subsequently injection-molded (Krauss Maffei 55T). The thermal decomposition was investigated using a Perkin Elmer Pyris-1 TGA. All measurements were performed under nitrogen with a heating rate of 10°C min⁻¹. The sample weight was 10 ± 2 mg. Evaluation of the flammability properties was made using pyrolysis combustion flow calorimeter (PCFC) and cone calorimeter equipment produced by fire test technology (FTT). Cone calorimeter tests were carried out on 100 × 100 × 4 mm³ sheets using an incident heat flux of 35 kW·m⁻², according to ISO 5660-1 standard. For PCFC tests, the samples (1 to 4 mg) were heated under 1°C/s rate from 20°C to 750°C in a pyrolyzer and the degradation products were conducted to another chamber to be well mixed with oxygen. Combustion took place at 900°C.

3 Results and discussion

It is well known that individual spherical PA6 nodules are generally formed when PA6 is blended as minor component with PP [35]. This type of morphology can be useful for flame retardancy of PP. Spherical particles of PA6 can act as char former inside of PP. Therefore, the improvement of flame retardancy for PA6 in PP/PA6 blend is crucial, particularly to promote his charring. In a preliminary study, we investigated the effect of two types of talcs in combination with MC or MPP on the flame retardancy of PA6. It has been shown in several studies that the specific surface area, aspect ratio, particle size distribution, surface chemistry of a incorporated particle in a polymer can influence the barrier effect during combustion and therefore modify fire behavior [36]. Here, we chose two talcs having very high specific surface area to be incorporated into PA6. This preliminary examination allowed to select the talc more suitable for the second stage of this study. The characterizations have been performed using cone calorimeter test for this step.

The heat release rate (HRR) curves as a function of time are presented in Figure 1(a) for samples containing MC and MPP in Figure 1(b). The HRR peak (pHRR) in such

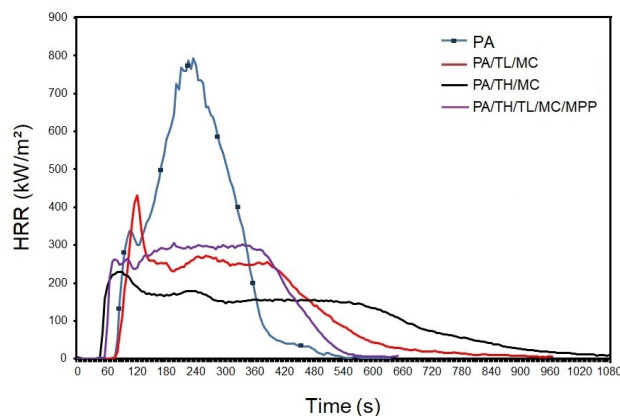
Table 3: Sample names and compositions

	PP (wt.%)	PA6 (wt.%)	SEBS-g-MA (wt.%)	MPP (wt.%)	MC (wt.%)	TH (wt.%)	APP (wt.%)
PP/PA/MPP/TH	24.25	24.25	1.5	25	-	25	-
PP/PA/MPP/MC/TH	24.25	24.25	1.5	12.5	12.5	25	-
PP/PA/APP/TH	24.25	24.25	1.5	-	-	25	25

plot decreased about 50% for the samples containing talcs and MC compared to the pure PA6. For the samples containing talc and MPP, this decrease was ca. 70%. Time to ignition (TTI) was decreased for samples with MPP compared to pure PA. For the samples containing MC, TTI is remained approximately constant, while MC delays the ignition but the fall in pHRR was limited.

During the combustion, MC is mainly decomposed to melamine and cyanuric acid at about 300°C. The decomposition of MC is an endothermic process and contributes to the flame retardancy effect of such additive [37, 38]. At higher temperatures, the degradation of melamine dilutes the gas phase. Moreover, the degradation of cyanuric acid is lead to formation of cyanic acid which can react with amide group of PA. Therefore, MC is effective in PA phase in delaying the time to ignition. However, its char formation capacity is not as efficient as MPP. In the case of decomposition of MPP, melamine and polyphosphate are formed. The decomposition of polyphosphate compounds is similar to that of ammonium polyphosphate [39] and helps the charring of PA. Here, the formed char structure is reinforced by the talc particles. High aspect ratio (HAR) talc named as TH seems slightly more efficient than ultrafine talc for decreasing the pHRR (Figure 1b). Therefore, we chose TH for the next stage of this work. The comparison of HRR curves of samples containing TH/MPP and TH/MC revealed that MPP is more efficient than that of MC in combination with TH talc (Figure 2), in terms of decrease in HRR peak. However, THR values are not modified for samples containing FR systems (All values are around 115 MJ·m⁻²). The observation of the remaining char residue in cone calorimeter test, Figure 3, showed that TH talc is more efficient than TL talc in formation of a continuous char layer. The homogeneous char layer regulates the emission of melamine and volatile combustible as a physical barrier during the combustion.

In light of these results, new formulations based on PP/PA blend were prepared (Table 3). Talc HAR (TH) was combined with MPP and/or with MC. An additional formulation containing APP as a reference flame retardant was also prepared and the results were compared.

**Figure 2:** Comparison of heat release rate curves of PA, PA/TL/MC and PA/TH/MC, and PA/TH/TL/MC/MPP samples**Table 4:** Summary of TGA and DTG results, under nitrogen atmosphere

Sample code	T _{10%} (°C)	T _{max} (°C)	Residue (wt%)
PP/PA/MPP/TH	402	415-530	33
PP/PA/MPP/MC/TH	387	396-523	28
PP/PA/APP/TH	380	393-514	42

Figure 4 displays TGA thermograms of the samples prepared in the second stage of study and the results (T_{10%} and T_{max} corresponding to the decomposition temperature at which 10% and maximum degradation took place respectively and detected in DTG) are given in Table 4. TGA curves showed that the onset degradation temperature (T_{10%}) for PP/PA/MPP/TH and PP/PA/MPP/MC/TH samples are higher than that of PP/PA/APP/TH sample. The T_{10%} values were 402°C, 387°C, and 380°C respectively for PP/PA/MPP/TH, PP/PA/MPP/MC/TH, and PP/PA/APP/TH. The thermal stability of PP/PA/MPP/MC/TH sample was significantly decreased above 395°C compared to other samples, which can be related to the presence of MC in the system. All samples showed two main weight loss stages. The T_{max} was shifted to higher temperature in the case of PP/PA/MPP/TH, Figure 4b. The highest amount of char residue, 42 wt.%, was obtained for PP/PA/APP/TH sam-

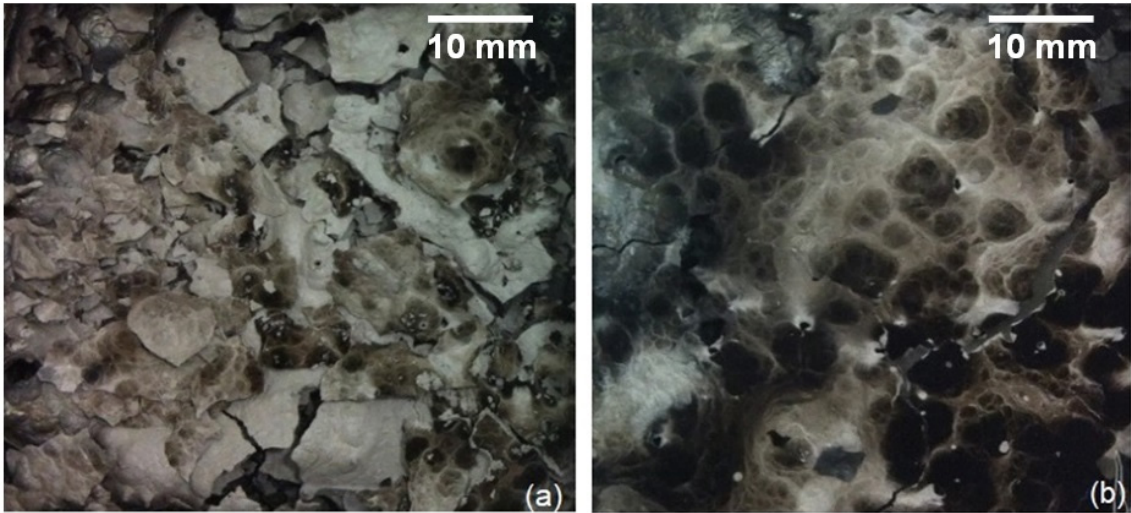


Figure 3: Photographs of the remaining char residue for (a) PA/TL/MPP; and (b) PA/TH/MPP in cone calorimeter test

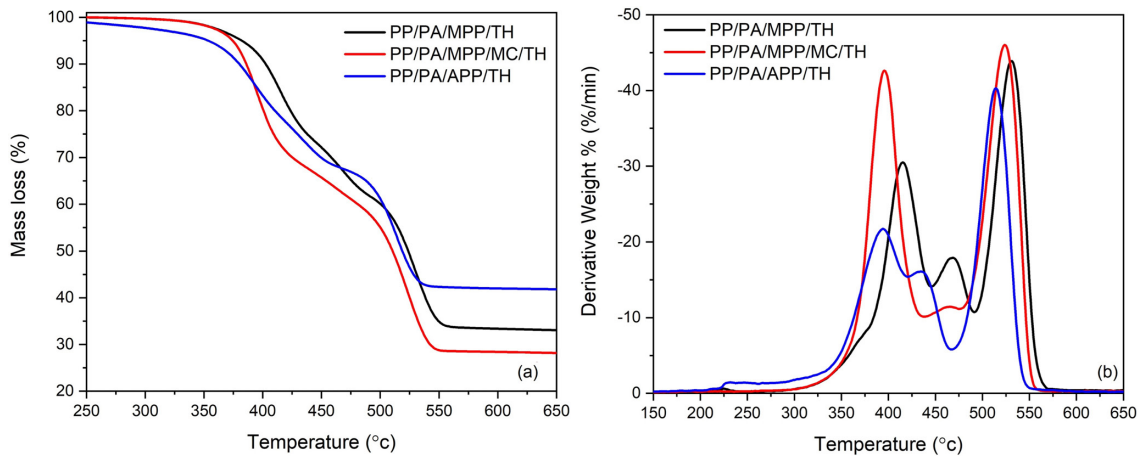


Figure 4: TGA and DTG thermograms of PP/PA blends containing TH, MC, MPP and APP

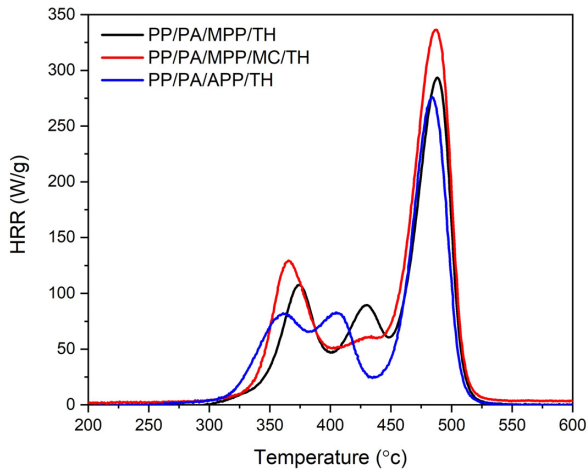


Figure 5: HRR curves of all samples studied here obtained from PCFC tests

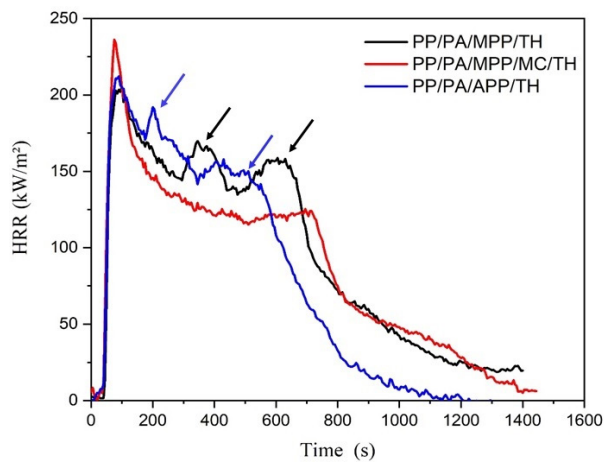


Figure 6: HRR curves, obtained from cone calorimeter test

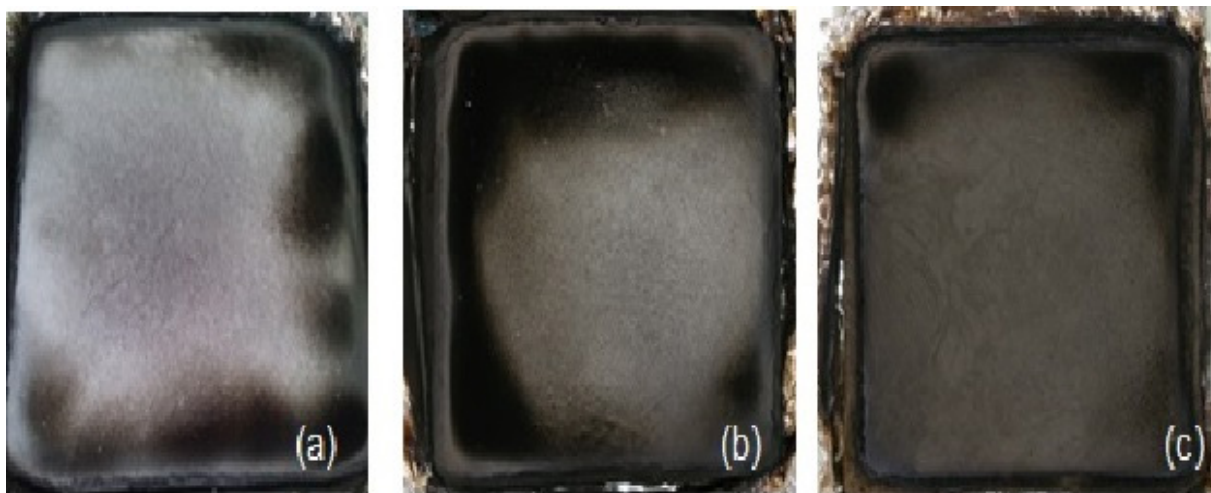


Figure 7: Digital photographs of the residues for PP/PA/MPP/MC/TH (a), PP/PA/MPP/TH (b), and PP/PA/APP/TH (c) samples from cone calorimeter test

ple, against 33 wt.% and 28 wt.% for PP/PA/MPP/TH and PP/PA/MPP/MC/TH samples, respectively.

The flame retardancy of composites was investigated by PCFC and cone calorimeter tests. The heat release rate curves obtained from PCFC tests are presented in Figure 5. The pHRR was slightly higher for PP/PA/MPP/MC/TH compared to two other samples, but the difference was not so significant. Moreover, the THR values (18 kJ/g) were approximately the same for all samples. The comparison of PCFC and DTG curves showed that an earlier degradation of APP had also an impact on HRR curve of PP/PA/APP/TH sample. Moreover, in the case of PP/PA/MPP/TH sample, the second step of degradation in TGA (around 430°C) did not liberate a high energetic gas and therefore in PCFC there was a small shoulder that meant a low heat release rate. The first peak in HRR curve of PP/PA/APP/TH can be explained by the formation of a char layer from APP during the pyrolysis in PCFC. This fact could be also supported by the results of TGA under nitrogen, Table 4. A high char quantity means that a more important quantity of polymer has not been completely pyrolyzed, instead transformed into a less-combustible product.

It is well known that in PCFC the barrier effect is not evidenced because of the small amount of sample (2-4 mg), as well as the separation of pyrolysis and combustion phenomena [40]. Thus, the chemical effect can only be monitored in gas phase. Therefore, all samples were also analysed by using cone calorimeter test, which is considered to be one of the most important equipment for fire behaviour testing and capable of simulating a developing fire scenario [41]. All samples were tested in cone calorimeter and the obtained curves are plotted in Fig-

ure 6. The TTI was almost 50 s irrespective of ingredients. The main peak of HRR was observed for all samples around 90 s. The value of pHRR was 200 kW/m² for PP/PA/APP/TH and PP/PA/MPP/TH samples, against 240 kW/m² for PP/PA/MPP/MC/TH sample. It seems that the presence of MC was led to an increase in pHRR in the beginning of combustion process. However, the HRR value of PP/PA/MPP/MC/TH sample was significantly decreased compared to those of PP/PA/APP/TH and PP/PA/MPP/TH samples. The presence of two small peaks of HRR for these samples (as indicated by the arrows Figure 6) indicates that the char layer formed was not as efficient as in the case of PP/PA/MPP/MC/TH. Therefore, the HRR curve was remained lower for this sample after 110s. A more efficient char quality probably prevented the release of gases during the combustion and therefore a better fire behavior was consequently observed. The digital photographs of remaining char residues at the end of cone calorimeter test are presented in Figure 7. The surfaces of all samples are smooth without cracks, which means formation of an efficient char quality for all samples. Therefore, a better quality of char in the case of PP/PA/MPP/MC/TH sample is probably related to the interaction between talc and flame retardants, and the formation of more resistant structures during the combustion compared to the other samples.

4 Conclusions

Flame retardant systems were developed based on PP and PA6 polymers through a two-stage recipe as typical recycled flame retardant PP/PA6 blends. Two kinds

of talcs having different physical properties were examined. Additives were first added to PA6 in combination with melamine cyanurate (MC) or melamine pyrophosphate (MPP) to specify the one acting as a barrier in combustion process. The high-aspect-ratio talc with more effectiveness was used in combination with MC, MPP and ammonium polyphosphate (APP) together with a compatibilizer in the second step. Thermogravimetric analyses in terms of $T_{10\%}$ and T_{max} revealed two main weight loss stages. $T_{10\%}$ values of 402°C and 387°C assigned to PP/PA/MPP/TH and PP/PA/MPP/MC/TH stood higher than 380°C for PP/PA/APP/TH. By contrast, thermal stability of PP/PA/MPP/MC/TH sample significantly dropped at temperatures above 395°C due to the presence of MC in the system. According to cone calorimetry data, the pHRR of PP/PA/MPP/MC/TH was the highest, while THR value of ca. 18 kJ/g was unconditionally observed for all samples. The comparison of PCFC and DTG curves showed an earlier degradation of APP that affected HRR of PP/PA/APP/TH. For PP/PA/MPP/TH sample, the first peak in HRR curve of PP/PA/APP/TH was ascribed to formation of a char layer in pyrolysis in PCFC, as supported by the results of TGA under nitrogen. The second step of degradation in TGA (around 430°C) did not liberate a high energetic gas, as there was a small shoulder in PCFC signifying a low heat release rate. Cone calorimetry revealed a TTI of about 50 s irrespective of formulation, whereas the main peak of HRR was ca. 90 s. The value of pHRR of 200 kW/m² for PP/PA/APP/TH and PP/PA/MPP/TH samples with respect to 240 kW/m² for PP/PA/MPP/MC/TH sample was indicative of enhanced pHRR in the presence of MC in the early stage of combustion process. However, the HRR value of PP/PA/MPP/MC/TH sample significantly dropped compared to those of PP/PA/APP/TH and PP/PA/MPP/TH samples, where bimodal HRR curve was an evidence of efficient char layer formed in PP/PA/MPP/MC/TH, which prevented the release of gases during the combustion and hence a high resistance against fire was achieved.

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