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# Self-extinguishing polypropylene with a mass fraction of 9% intumescent additive II - Influence of highly oriented fibers

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## ABSTRACT

Recently, noticeably enhanced flame retardancy of multilayered self-reinforced composites, flame retarded with common ammonium polyphosphate based intumescent system, was described. In this paper the observed novel flame retardant synergism between intumescent additive system and highly oriented polymer fibers is further studied. The ignitability and combustion behaviour of flame retarded multilayer self-reinforced composites was compared to flame retardant compounds, prepared by simple melt compounding, of identical low additive contents, both when the heat was applied parallel (UL-94 tests) and perpendicular (cone calorimetric tests) with the direction of the embedded oriented tapes in self-reinforced composites. SEM and EDS analyses supported the different foaming process of the two types of samples to be understood, while the structure and character of the finally (after combustion) obtained charred layers were examined by compression tests. Considering the results of all the applied testing methods, the complex picture of the mechanism behind the enhanced flame retardant efficiency of flame retarded self-reinforced composites could be clarified.

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## 1. Introduction

The application of reinforced polymeric composites in construction and transportation industry is continually increasing. Due to the more and more strict environmental-focused legislation, however, these industries will be forced to replace their conventional (e.g. glass fiber reinforced) polymer composites by more environmental-friendly alternatives; the use of recyclable or biodegradable polymeric materials is urged. Such environmentally favourable examples are the easily recyclable self-reinforced composites (SRCs) [1,2,3]. It is claimed that 20-30% reduction in weight can be achieved by using SRC parts (from PE, PP, PET etc.) instead of conventional glass fiber reinforced parts of comparable mechanical properties [4]. At the same time the importance of flame retardancy of polymeric composites, especially in technical application fields, is indisputable. However, the flame retardation of such recently developed polymer composites is often not examined or solved yet.

When compared to conventional halogenated flame retardants, phosphorus-based flame retardants have several environmental advantages, such as lower smoke toxicity in case of accidental fire, therefore their use is favoured. Wide range of phosphorus-based flame retardant (FR) compounds is available for polypropylene (PP), among which the use of ammonium polyphosphate (APP) based intumescent flame retardant (IFR) system is fairly common [5]. The mechanism of such intumescent additive system (consisting of acid source, carbon source and blowing agent) has been already studied and reviewed comprehensively [6,7]. Accordingly, at least 15-20 wt% IFR is needed in order to achieve acceptable level of flame retardancy in polyolefins, however in most of the cases 25-35 wt% IFR is used. In contrast, it has been shown recently that much lower amount of IFR can be efficient in PP than expected if self-reinforced composites (SRCs) are formed [8], V-0 classification in UL-94 test is achievable with only 9 wt% of IFR additive. It has been demonstrated that the molecular orientation (i.e. shrinking ability) of the embedded highly stretched reinforcing PP tapes, when combined with IFR additives, plays the key role behind the observed outstanding fire retardant performance of the examined SRCs. The formation of a protecting charred layer of special structure, when an SRC is exposed to heat, was proposed as explanation in our former study [8]. In the present paper this phenomenon is examined further in order to contribute to deeper understanding of the observed synergism.

## 2. Materials and methods

### 2.1. Materials

Tipplen R 959A (produced by TVK Plc., Hungary) type PP (MFI = 45 g/10 min, 230°C/2.16 kg) was used as the matrix material for the prepared samples. As flame retardant additive, Exolit AP 766 (produced by Clariant Ltd., Germany) was added to the matrix material, which is a combined, ammonium polyphosphate (APP) based and charring agent containing IFR additive. In accordance with the product datasheet the phosphorus content of the additive is 23-25 % and its nitrogen content is 14,4-16,4 %. Plain-woven PP fabric (identification code: T-PPT-181, Stradom S.A., Poland), composed of highly stretched split PP tapes, was used as reinforcement. The reinforcing fabric has a melting temperature of  $T_m = 172.4^\circ\text{C}$  (determined by DSC), and a tensile strength of  $128.8 \pm 3.0$  MPa measured on a 50 mm wide strip and an areal weight of  $171.5$  g/m<sup>2</sup>.

### 2.2. Preparation of composites

In order to compare the burning behavior of flame retarded simple PP compounds and multilayer SRCs, samples with the same composition, with IFR contents of 9 and 13 wt%, were prepared for both materials. The flame retarded compounds were prepared by straightforward mixing of the required amount of IFR additive with PP in a Brabender Plasti Corder PL 2000 type internal mixer at 190°C, with a rotor speed of 30 rpm, for 10 minutes. The blends were then hot pressed to form 4 mm thick specimens in a Collin P200E type laboratory hot press. The flame retarded SRCs were prepared through several steps. The first step was the compression moulding of 180 µm thick matrix layers with IFR content of 16.3 and 23.6 at 190°C in a Schwabenthan Polystat 300 S laboratory hot press. The multilayered SRCs were then manufactured by laminating 11 flame retarded matrix foils and 10 woven PP fabrics using the film stacking method [9,10]. The film-stacked packages were hot-pressed to form composites (in the same hot press) at a temperature of 162°C and a pressure of 6 MPa for 2 minutes. The SRCs had a total reinforcement content of  $45 \pm 1.5$  wt%. Thus each SRC (considering the total mass of the SRCs) had the same flame retardant content as the equivalent flame retarded simple PP compounds. (For example in the case of the SRC with an IFR content of 9 wt% 55% of the mass of the SRC was flame retarded film containing 16.3 wt% IFR, giving an overall IFR content of  $0.55 \times 16.3$  wt% ~ 9 wt%.) The mean thickness of the prepared SRCs was 3.78 mm.

### 2.3. Characterization methods

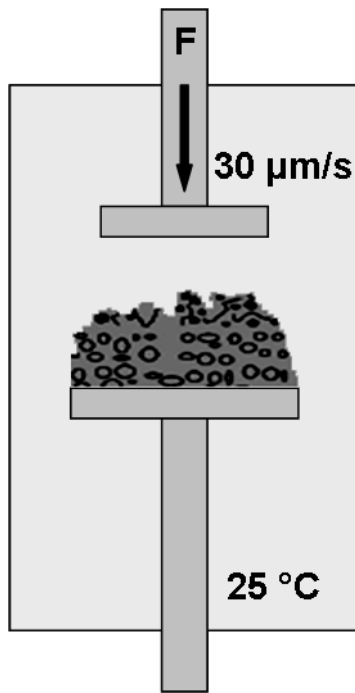
The flame retardant performance of the prepared samples was characterized by limiting oxygen index (LOI) measurements according to the ASTM D 2863 standard. The LOI value expresses the lowest oxygen to nitrogen ratio where specimen combustion is still self-supporting.

Standard UL-94 flammability tests (ASTM 1356-90 and ANSI/ASTM D-635/77) were also performed. UL-94 classification is used to determine dripping and flame spreading rates.

Mass Loss type cone calorimeter tests were carried out by an instrument made by FTT Inc, using the ASTM E 906 standard method. Specimens (100 mm x 100 mm) were exposed to a constant heat flux of 50 kW/m<sup>2</sup> and ignited. Heat release values and mass reduction were continuously recorded during their burning.

AR2000 Rheometer (TA Instruments, USA) with special adjustment was used for structural and mechanical characterization of chars obtained from round shaped specimen (diameter = 30 mm) burned at 500°C under cone heater. Compression tests were carried out with a constant squeeze rate of 30 μm/s while the changes in the normal force were recorded (**Fig. 1**). The diameter of the squeezing upper plate was 25 mm and the initial gap was 20 mm in all cases. As the heights of the examined chars were different, the registered normal force values were plotted against the percentage of deformation of the corresponding chars.

The cell structure of the charred foams was investigated under a field emission scanning electron microscope (SEM) (JEOL JSM-5500 LV). The foam samples were coated with a thin gold layer to prevent charge build-up on the surface. The used SEM apparatus is equipped with EDS (energy dispersive spectrometry) unit capable of performing elemental analysis of the obtained residues.



**Figure 1** Compression test used for mechanical characterization of chars

### 3. Results and discussion

For better understanding of the role of highly stretched PP tapes in the fire retardancy the char forming process of intumescent flame retardant SRCs was compared to compounds, prepared by simple mixing of IFR additive with the molten PP, of identical compositions. The ignitability and combustion behaviours were compared both when the heat was applied parallel (UL-94 tests) and perpendicular (cone calorimetric tests) with the direction of the embedded oriented tapes in SRCs. SEM and EDS methods were selected for examination of the foaming process and char structure of the two types of samples, while the mechanical characteristics of the residual charred layers were compared by compression tests.

Clear evidence for significantly better flame retardant efficiency of IFR additive in multilayer SRCs comparing to bulk compounds was provided by **UL94** flammability tests and **LOI** measurements. As it is presented in **Table 1**, in contrast to the HB UL94 rating of the reference PP-IFR compounds V-0 classification of PP-SRCs could be achieved despite the same chemical compositions (IFR content of 9 and 13 wt%), and also noticeably higher LOI values were measured for the flame retarded SRCs than for the corresponding compounds. Although the local concentration of IFR outside the fibres is

higher (almost two-fold) compared to the homogeneous mixture it cannot explain the observed outstanding fire performance by itself, because, for example in the case of the PP-SRC-IFR9 sample, the IFR concentration in the matrix layer (16.3 wt%) is still significantly less than needed (at least 21 wt% IFR [8]) for reaching V-0 classification when preparing simple compounds. On the other hand, *Hornsby et al.* [11] studied the fire behaviour of multilayered composites consisting of fire retardant skin and unfilled core layers and did not find any noticeable enhancement in fire retardant behavior for the benefit of the multilayered structure. Therefore we concluded that in the case of the flame retarded self-reinforced composites another phenomenon (high degree of molecular orientation of fibers) must play the key role behind the observed significant enhancement in flame retardant behaviour.

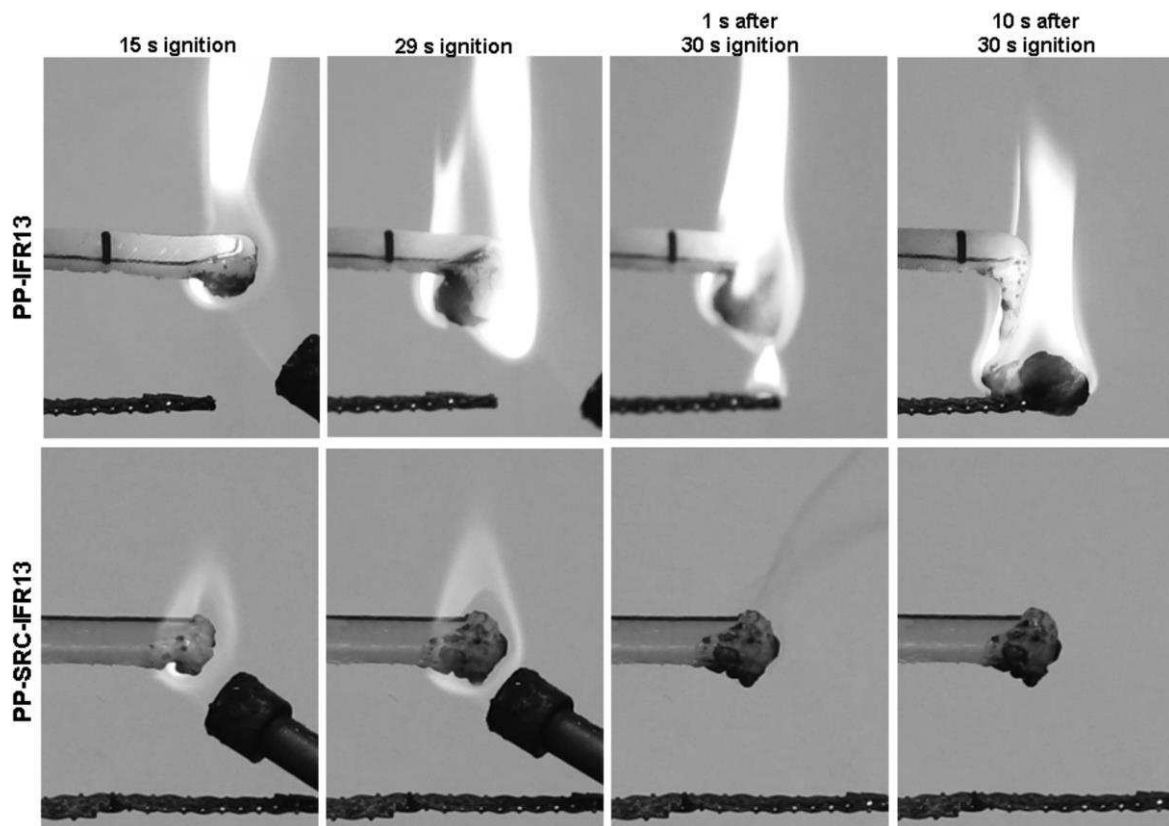
**Table 1 Comparison of UL94 ratings and LOI values of PP compounds and PP-SRCs with IFR contents of 9 and 13 wt%**

Sample	UL-94 Classification*	LOI (vol%)
PP-IFR9	HB (22.8)	24
PP-SRC-IFR9	V-0	26
PP-IFR13	HB (19.4)	27
PP-SRC-IFR13	V-0	30

\*in parenthesis the horizontal burning rates (mm/min), if measurable, are indicated

For demonstrating the **burning behaviour** of the two types of flame retarded PP samples photographs, taken during horizontal and vertical burning of samples, are presented in **Figure 2** and **Figure 3**, respectively. It can be seen that, despite the same IFR content, the behaviour of the two types of flame retarded samples, exposed to flame, is completely different. During vertical test (**Fig. 2**), the PP-IFR13 specimen proved to be easily ignitable, and already after 15 seconds of ignition noticeable flame arouse. At this initial stage of burning the formation of a charred, but fairly loose foam structure is also visible, which, by virtue of its weight, gets to the bottom of the specimen and in time even falls down. As the foamed char continually loses contact with the specimen, over and over virgin, molten polymer gets to the pyrolysis zone and feeds the flame. In contrast, a weak, almost colourless flame can be observed during the ignition of the FR-SRC system. As the Bunsen burner is taken, the PP-SRC-IFR13 specimen extinguishes immediately. The appearance of the remaining burnt surface differs a lot from that of the non-reinforced sample. Survey of the shrunk structure of the charred residue helped the mechanism behind the observed flame-extinguishing behaviour to be

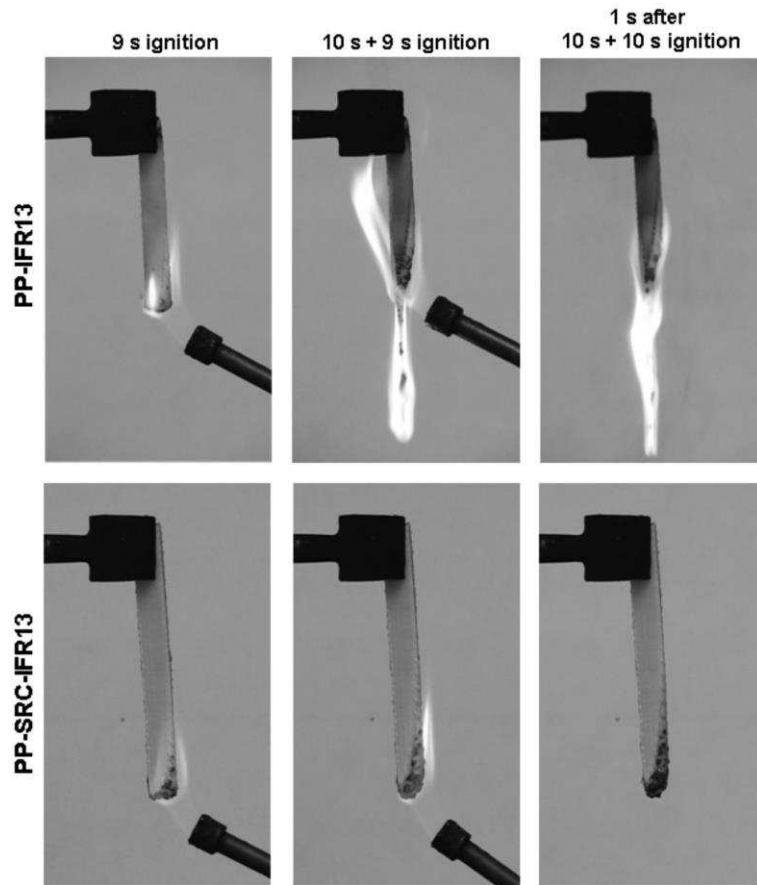
understood. It is proposed that the shrinkage of the heated highly-stretched PP tapes embedded in the matrix contributes to the formation of a compact surface and by this means hinders the availability of the fuel for flame.



**Figure 2 Horizontal burning test of PP compound (PP-IFR13) and SRC (PP-SRC-IFR13) with 13 wt% IFR content**

When the specimens are vertically ignited (**Fig. 3**), the shrinking operating against the force of gravity becomes more crucial for achieving prominent flame retardation. In the case of the compounded samples considerable elongation, accompanied with increasing area of easily inflammable surfaces, is characteristic for the burning specimen. Whilst, in the case of the layered composites, ignited parallel to the direction of orientation, significant vertical shrinkage is observable which hinders the elongation and the formation of virgin surfaces. In this case, the charring and FR action, corresponding to the flame retarding act of the applied IFR additive, concentrates to a smaller volume resulting in immediate fire extinguishing even at low additive level.



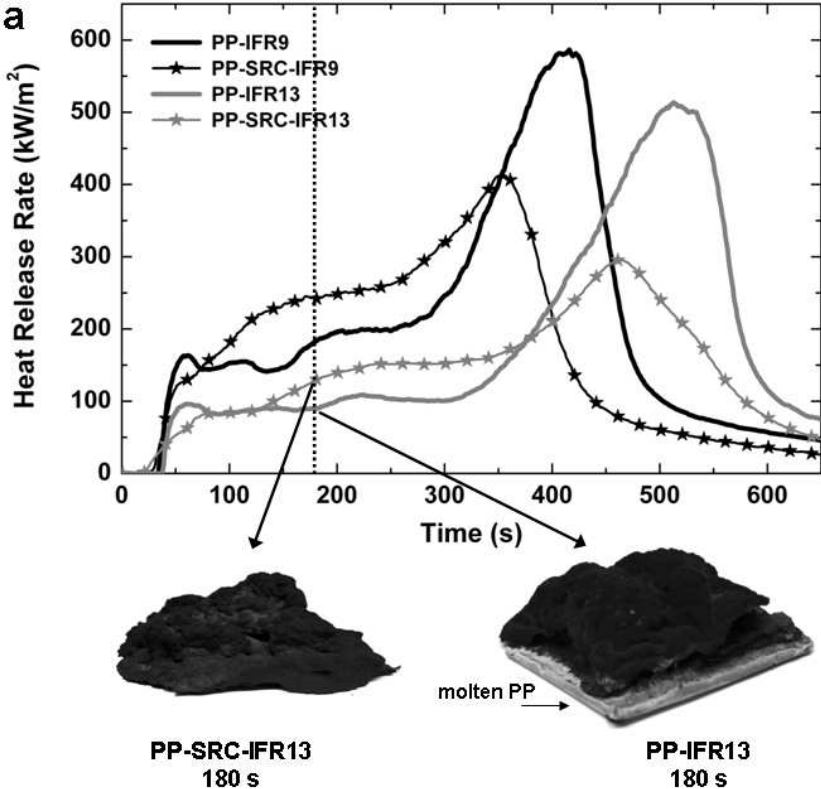


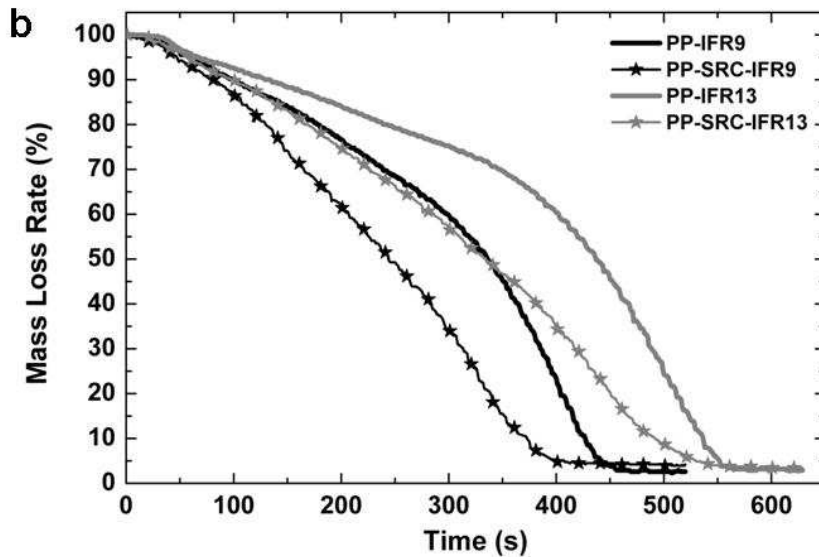
**Figure 3 Vertical burning test of PP compound (PP-IFR13) and SRC (PP-SRC-IFR13) with 13 wt% of IFR content**

The burning behaviour of the two types of flame retarded samples shows also noticeable differences when examined under the conditions of a **cone heater**. The obtained heat release rate and mass loss rate curves are presented in **Figure 4 a** and **b**, respectively. Considering **Figure 4 a** it can be seen that in the beginning the combustion of the compounded samples are accompanied with considerably lower heat release rates than those of the multilayered composites. However, after a while noticeable peak of heat release rate appears in all cases, which are by 30-40% lower in the case of the flame retarded SRCs. This can be explained by the different foaming processes and formed structure of chars. Highly expanded, but loose char forms from the compound containing IFR additive, which acts, due to its porous, multicellular structure, as efficient heat insulator at the initial stage of burning protecting the underlying molten PP from combustion. However, as this weak foam structure degrades, significant amount of PP gets into the burning zone abruptly, resulting in sharp peak of heat release. In contrast, the intensive expansion is hindered by the additive-free woven PP fabrics in the

case of SRC composites. When the multilayer composite is exposed to heat the shrinkage of the highly stretched tapes occurs simultaneously with the swelling of the flame retarded matrix layers. As a consequence, much denser, compact charred layer will be formed. As the heat insulation depends particularly on the thickness (a thicker layer gives more insulation), the formation of compact foams in the case of SRCs is associated with higher initial heat release rates.

In order to further investigate the different foam forming processes of the compounded and multilayer samples, the charred layers formed at the early stage of burning were examined. In the case of the flame retarded compound (PP-IFR13) large amount of molten PP was present (as expected) under the thick, swollen, charred layer after 180 s of combustion. In the case of the multilayer SRC (PP-SRC-IFR13), however, a completely charred full mass was obtained in the same period (only thin virgin polymer spots could be observed on certain areas of the char bottom). These observations are in accordance with the recorded heat release curves. The more pure polymer under the protecting layer, the more heat released abruptly (the higher the peak of heat release rate).



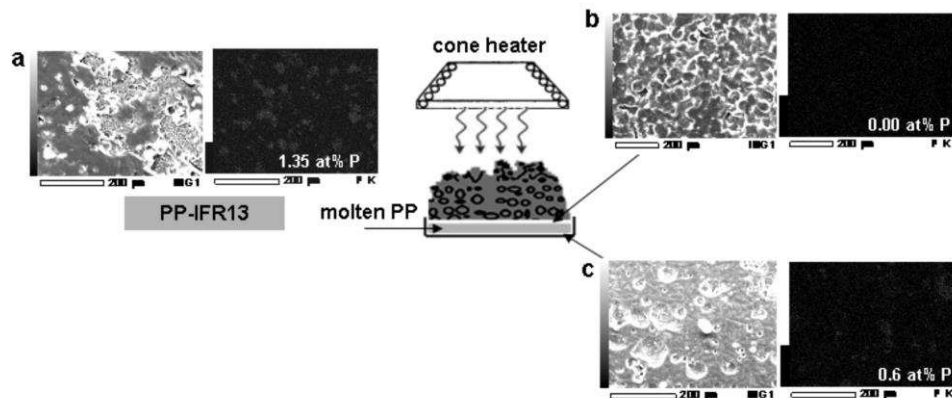


**Figure 4 a) Heat release rate curves (below charred residues obtained after 180 s of combustion) and b) mass loss rate curves recorded during the burning of PP compounds and SRCs with IFR contents of 9 and 13 wt%**

It was assumed that the migration of the flame retardant components [12] plays crucial role in the char formation process and is greatly responsible for the observed differences in combustion behaviours. **EDS spectrometry** was used to establish the phosphorus (P) content in the representative parts of the samples. As it is presented in **Figure 5 a**, 1.35 at% P could be detected on the surface of the compounded PP sample with 13 wt% IFR content. After 180 s combustion (in Cone Calorimeter at 50 kW/m<sup>2</sup>) significant amount of molten polymer remained under the formed thick char foam. The distribution of P atoms on the upper and lower surface of this (solidified) PP layer is presented in **Figure 5 b** and **c**, respectively. It can be noticed that on the upper surface, which is directly connected to the charred foam, no P could be detected at all and on the bottom surface, being contiguous with the Al sample holder, only 0.6 at% P was measured. Considering these results, it was concluded that the applied heat flux initiated migration of the FR additives towards the upper surface.

By the enrichment of the active species an effective heat protecting layer can develop on the top of the compounded sample, however, the flame retardant content of the underlying polymer becomes poor (insufficient). As a consequence, when the protecting shield gets damaged, the remaining molten polymer will get burnt abruptly, accompanied with the appearance of a sharp peak of heat release rate. When considering the mass loss rate curves, recorded during the cone calorimetric tests (**Figure 4 b**), it can be established that about 60-70% (indicated by the increased rate of mass reduction) of the original mass of the compounded samples is referred to be combusted during this accelerated burning period.

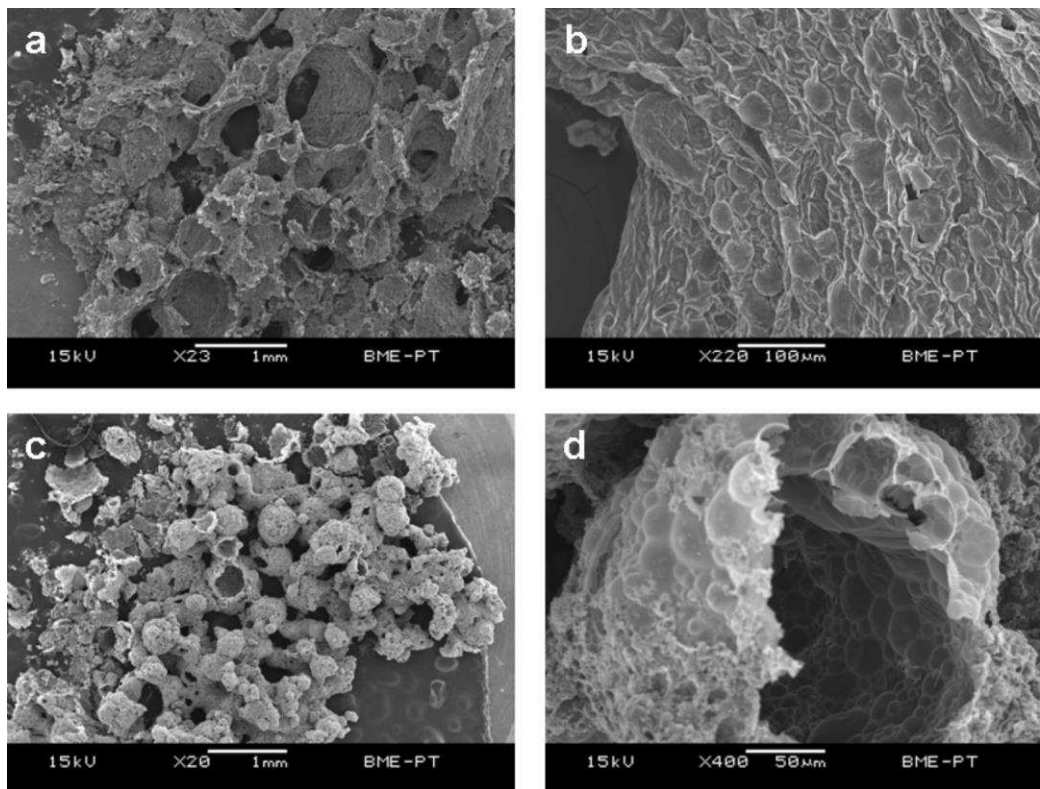
In the case of the SRCs no considerable amount of molten polymer was observed at the beginning period of burning (and during char formation), and in the cross section of the developed char homogeneous P distribution could be observed. Therefore it was concluded that the migration of the IFR additives throughout the entire bulk of the multilayered sample is hindered by the embedded additive-free PP fabrics. As a consequence, the whole mass of the sample participates simultaneously in the charring and foam-forming process resulting in somewhat higher initial rates of heat emission but suppressed peak of heat release rates. In the case of the SRCs there is no polymer layer with insufficient additive content, thus, as we have noticed in our preliminary study [8], the total heat emission is reduced and higher amount of residual char remains after combustion when compared to the compounded samples (in which the migration is unobstructed) of identical IFR contents.



**Figure 5** EDS imaging of the P content of a) the prepared PP-IFR13 compounded sample, b) upper layer of the molten PP and c) lower layer of the molten PP (obtained after 180 s combustion in cone calorimeter)

The charred residues obtained at the end of the combustion were noticeably of different character. The foam corresponding to the flame retarded compounds seem to be soft and flexible, while that of the SRCs were more compact and rather rigid. The structure of the two types of flame retarded samples was examined by SEM. At lower magnification it is visible that the char corresponding to the PP-IFR13 sample (**Figure 6 a**) consists of funnel-like formations and holes formed probably during the release of the gaseous degradation products. In contrast, the char of the PP-SRC-IFR13 sample (**Figure 6 c**) consists of hollow spherical formations. When applying greater magnification it can be seen that the walls of both the funnels (**Figure 6 b**) and spheres (**Figure 6 d**) are built up from spherical cells, however, while these constitute a tough, multicellular honeycomb structure in the case of the SRC (**Figure 6 d**), they are limp and apparently lack of toughness in the case of the compound (**Figure 6 b**). The observed loose foam structure, formed during the intensive

gas release and instantaneous foaming process of the compounded samples, is probably in correspondence with the abrupt change of the corresponding heat release rates at 40 s in **Figure 4 a**. On the other hand, the additive-free reinforcement layers in the case of the SRCs can hinder the abrupt gas escape and expansion, resulting in foam of mainly closed cell structure. Considering the corresponding heat release rate curves in **Figure 4 a**, it can be observed that the char formation process is prolonged compared to that of the compounded samples.



**Figure 6 SEM images of the charred residues obtained after the cone calorimeter tests of PP-IFR13 (a and b), and SRC-IFR13 samples (c and d)**

**The mechanical resistance** of the charred residues obtained after combustion of round specimen (with a diameter of 30 mm) in the cone calorimeter (set to 50 kW/m<sup>2</sup> heat flux) was examined through compression tests. The tests were carried out in a rheometer with a plate-plate geometry on the basis of the method used earlier for characterization of charred foams formed from intumescent coatings [13]. In **Figure 7**, the recorded normal force values were plotted against the deformation percentages

of each charred residues. It can be seen that the main trends of the obtained curves are similar: initial increase in normal force, corresponding the first cracking of the foam structure; then slightly growing average normal force needed for deformation; and finally the compaction stage. However, unambiguous differences can be established between the compression behaviour of the chars of the two types of flame retarded samples. It can be noticed that in the case of the SRCs the step of the initial increase in normal force is much higher, the fluctuation around the characteristic average normal force is considerably larger and the compaction phase starts at significantly lower stage of deformation than those of the corresponding counterparts at both examined additive content.

It can also be observed that before compaction the steepness of deviations are characteristic for each sample. For characterisation of the toughness and strength of the foam structures by a general index, these characteristic values (steepness of the curves) were calculated and presented in **Table 2**. The calculated indexes could be interpreted as the characteristic resistance of char layers or cell structures of the formed foams against deformation. The chars corresponding to the SRCs proved to be much more rigid than those of the compounded samples (in accordance with the SEM images). The more dense character of these chars is also demonstrated by the much lower deformation percentages corresponding to the starting point of compaction (**Figure 7**). These observations are also in accordance with the measured higher amount of charred residues obtained after cone calorimeter tests in the case of the SRCs, as already published earlier [8].

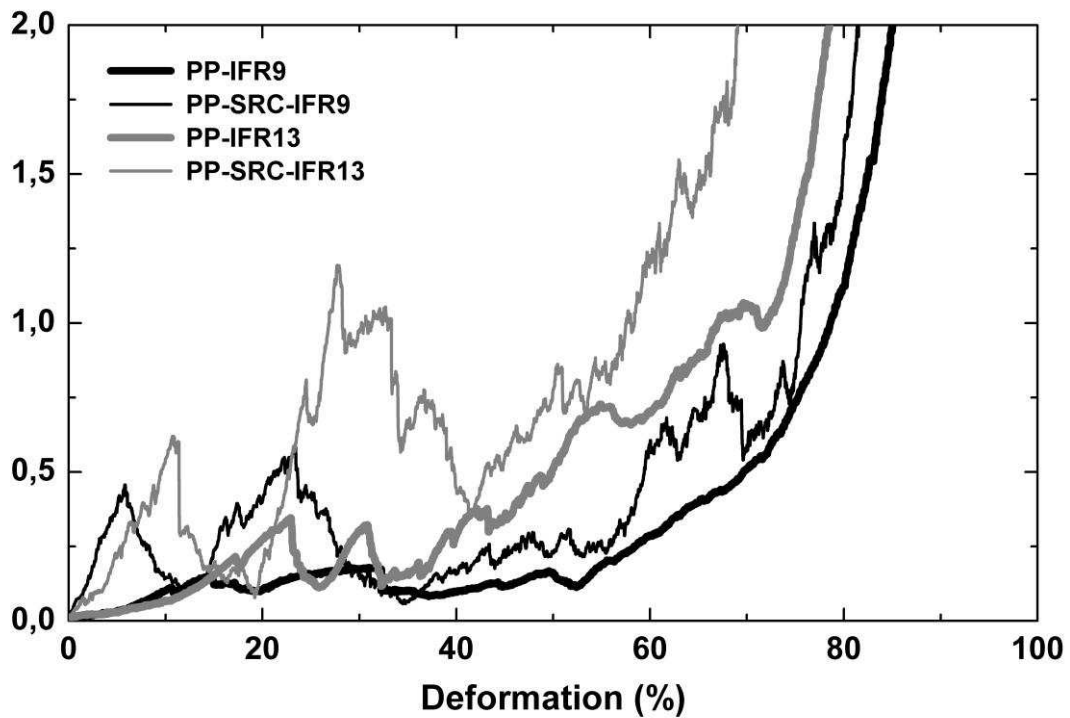


Figure 7 Compression test of the charred residues obtained after combustion under cone heater

Table 2 Calculated foam strength of the charred residues

Sample	Foam strength (Pa/def%)
PP-IFR9	36.7
PP-SRC-IFR9	154.8
PP-IFR13	93.7
PP-SRC-IFR13	211.9

#### 4. Conclusion

Based on comprehensive analyses, performed to study the ignitability, char forming process, burning behaviour and post-combustion remains of flame retarded SRCs, important conclusions could be drawn for better understanding of the phenomenon behind their outstanding flame retardancy.

It was established that the significant shrinkage, exhibited by the highly-stretched PP tapes when exposed to heat, is the key factor behind the self-extinguishing behaviour of flame retarded SRCs with surprisingly low additive content (only 9 wt% IFR). As a consequence of the shrinkage, which is fairly



noticeable both during horizontal and vertical ignition, the elongation of the molten specimens, and thus the formation of virgin polymer surfaces, is hindered. In this case the forming compact charred surfaces can act as barrier of increased efficiency against heat and fuel transport, resulting in immediate fire extinction.

In a cone calorimeter, where the composites are exposed perpendicularly to heat flux, further effects of the embedded PP tapes could be confirmed. It was concluded that the embedded (additive-free) PP fabrics hinder the migration of flame retarding substances towards the top of the polymer bulk, resulting in somewhat higher initial rates of heat emission, but suppressed peak of heat release rates and higher amount of residual chars compared to their unreinforced counterparts of same compositions.

Also the post-combustion charred residues are of different character for the two types of examined samples. In the case of the compounded flame retarded PP samples the observed loose foam structures could be attributed to the intensive gas release and instantaneous foaming process occurring at the initial stage of burning, while in the case of the multilayered SRCs the abrupt gas escape and expansion is also hindered and the char formation process became prolonged in time. Mainly closed cell structure is characteristic for such char. Compression tests showed evidence for the more compact and rigid character of the charred residues obtained after combustion of the flame retarded SRCs.

It can be concluded that the embedding of additive-free, highly-stretched (oriented) fibers into the polymer matrix can basically change the burning behaviour (ignitability, char forming process, migration effects, etc.) of a flame retarded polymer system. It is proposed that the described advantageous phenomena could be utilized in other polymer types/systems, with other flame retardants, or in other self-reinforcing structures (e.g. in injection moulded all-polymer composites). It is prospected that high amount of flame retardant additives could be spared this way and by this means a more cost-effective flame retardancy of polymers could be realized in the near future.

## **5. Acknowledgement**

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