EVALUATION OF NOVEL INTUMESCENT FLAME RETARDANT SYSTEM ON FLAME RETARDANCY OF POLYPROPYLENE

FRPM 11 13th European Meeting on Fire Retardant Polymers
26-30 JUNE 2011 ALESSANDRIA - ITALY

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Introduction and objective

Polypropylene is one of the polyolefin of great importance in the industrial sector, thanks to its superior intrinsic properties such as high chemical resistance, high melting temperature, low density, and the moderate cost. In order to progress the PP properties, intense researches have been carried out on its flame retardancy which may be achieved by the addition of fire retardant (FR) additives using various ways [1].

Among FR additives, i.e "intumescent" ones became a promising generation in thermoplastic for some of their advantages such as low toxicity, and antidripping properties [2-5].

In this context, the objective of this study was to investigate the potential effect of novel intumecent flame retardant system on flame retardancy and antidripping properties of PP.

Experimental Part

Materials and Sample Preparation

Polypropylene (Mopen HP 500N –trademark, abbrev. PP) supplied by Basell.

An intumescent additive combination comprising poly(2,4-piperazinyl-6-morpholinyl-1,3,5,-triazine) and ammonium polyphosphate (MCA®Intumescent system HF7525 – trademark, abbrev. IS) supplied by MCA Technologies GmbH (Switzerland).

PP/IS composite (PP/20 wt% IS) was prepared via direct melt compounding using a Leistritz ZSE 18 HP co-rotating twin screw extruder (screw diameter (D) = 18 mm, L/D = 40).

Measurements

Scanning electron microscope (SEM, LEO 1450 VP) with an energy dispersive X-ray probe (EDS, INCA Energy Oxford) attachment.

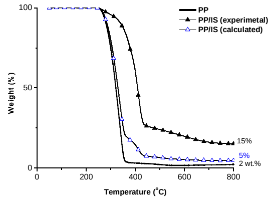
Thermogravimetric (TG) analyses were performed using a Q 500 Thermal Analyzer at a heating rate of 10 ºC · min $^{-1}$ from 50 ºC to 800 ºC under air flow (40 ml/min).

UL-94 vertical burning test was carried out according to ASTM D 3801-00 standard procedure.

Cone calorimeter test was carried out in accordance with ISO-5660-1 standard procedure using a Fire Testing Technology Cone Calorimeter.

Results and discussions

TG analysis



Sample	T_{onset} (°C)	T_{max} (°C)	Residue at 800 ºC (%)
PP	271	335	2
PP/IS (experimental)	306	406	15

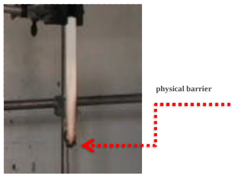
T_{onset} (the temperature at which the sample has loss 5% of its original mass);
T_{max} (the temperature at which the maximum rate of mass loss occurs).

TG experimental curve compared with calculated ones reflects that interactions between the oxidized products of polymer degradation and of IS occurs leading to higher thermal stabilization of the formulation. Furthermore, the amount of the residue at 800 ºC is higher than the one resulted from independent decomposition of PP and IS. These data evidenced that IS promote charring of PP.

UL-94 vertical burning test

PP

PP/IS

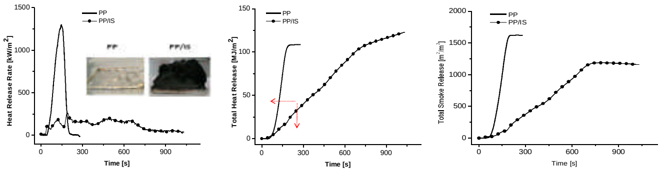


Sample	t_1 (s)	t_2 (s)	Drip/ignition of the cotton	Time to start dripping (s)	UL-94 rating
PP	70	-[a]	Yes	6	No rating
PP/IS	1	3	No	0	V-0

t_1 - the burning time after first ignition;
t_2 - the burning time after second ignition;
[a] - the whole sample burns after first ignition and thereby t_2 can not be recorded.

Neat PP start burning spontaneously after the first 10 s flame application, which continues till the test sample is completely burn up to the sample holding clamp, whereas the PP/IS composite even after the second application of the flame does not burn (*as it can be seen from the digital image above*) due to the formation of intumescent char layer at the surface of the sample which hinder the flame to reach a fresh unburnt surface sample and therefore providing an efficient barrier effect against the propagation of the flame.

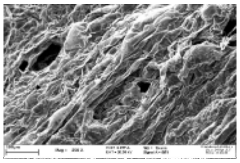
Cone calorimeter

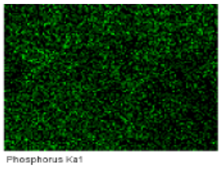


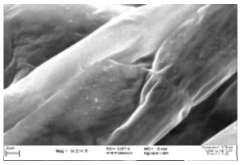
Sample	TTI ± σ (s)	PHRR ± σ (kW·m⁻²) (%, reduction)	THR± σ (MJ· m⁻²)	EHC (MJ· Kg⁻¹)	AMLR (g·s⁻¹)	ASEA± σ (m²·kg⁻¹)	TSR ± σ (m²·m⁻²)
PP	54±6	1305 ±37	108±2	48.9±1.9	0.035±0.001	732± 43	1621±73
PP/IS	39	276 ±1 (-79)	116±10	41.61±2.2	0.008±0.001	425±49	1188±177

By addition of IS into PP it was revealed that the peak of heat release rate (PHRR), the optical density of the smoke (ASEA), and the total smoke release (TSR) were remarkable decreased. A general conclusion which can be drawn from these results is that the intumescent shield developed during the combustion of the PP/IS composite provide an excellent insulation against heat transfer from the heat flux/flame to the unburned fuel, diffusion of external oxygen into the degradation zone and diffusion of the volatile degradation products in the burring zone.

Morphological structure of the intumescent char residue
obtained at the end of the combustion test







SEM observation:
- some voids
- irregular swollen structure
- almost compact surface

In the light of the above mentioned findings, it is possible to conclude that the new intumescent system could be taken into consideration as flame retardant for PP.

FUTURE WORK

Further investigation will focus to determinate the optimal concentration of IS as well to have
a better understanding of the mechanism of actions of IS on the flame retardancy of PP.

References
[1] S. Zhang; A. R. Horrocks; *Prog. Polym. Sci.* 2003, 28, 1517; [2] G. Camino, In: G. Nelson, editor. Fire retardant polymeric materials, ACS, USA, 1995, chapter 10, 461-492; [3] S. Bourbigot; M. Le Bras; R. Delobel *J. Fire Sci.* 1995, *13*, 3; [4] G. Camino; L. Costea; L. Trossarelli; *Polym. Degrad. Stab.* 1984, *6*, 243; [5] S. Bourbigot; M. Le Bras; S. Duquesne; M. Rochery; *Macromol. Mater. Eng.* 2004, *289*, 499.

Acknowledgments: Thanks are addressed to MCA Technologies GmbH (Switzerland) for providing the intumescent flame retardant.