INVESTIGATION ON FLAME RETARDANCY AND ANTIDRIPPING OF NOVEL INTUMESCENT FLAME RETARDANT TOWARDS A TRADITIONAL ONE ON POLYPROPYLENE

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The main goal of this work is to investigate the effectiveness of novel phosphorous-nitrogen intumescent flame retardant (IFR) on the flame retardancy of polypropylene at incorporation level below the standard amount (25-30 wt.%¹⁻³) required for intumescent flame retardants. Furthermore, the flame retardancy performance of this novel intumescent flame retardant was compared with that of traditional IFR, such as Exolit AP760.

Experimental

Polypropylene (Mopen HP500N-trademark) supplied by Basell. An intumescent additive combination comprising poly(2,4-piperazinyl-6-morpholinyl-1,3,5,-triazine, abbrev. MMP Triazine HF) and ammonium polyphosphate (abbrev. APP), (MCA[®]Intumescent system HF7525-trademark, abbrev. IS1) supplied by MCA Technologies GmbH. Exolit AP760-trademark (abbrev. IS2) supplied by Clariant. Pentaerythritol (Charmor PM15-trademark, abbrev. PER) supplied by Perstorp. All the PP/IS composites (PP/10 wt.-% IS1, PP/15 wt.-% IS1 and PP/20 wt.-% IS1), with the exception of the PP/20 wt.-% IS2 composite, were prepared via direct melt compounding using a Leistritz ZSE 18 HP co-rotating twin screw extruder. The PP/20 wt.% IS2 composite was prepared with an internal Rheomix-Brabender OHG 47055 mixer.

The UL-94 vertical burning (UL94-V), the limiting oxygen index (LOI) and cone calorimeter tests were carried out in accordance with ASTM D 3801-00, UNI EN ISO 4589-2 and ISO-5660-1 standard procedures. Thermogravimetric (TG) analyses were performed using a Q 500 Thermal Analyzer at a heating rate of 10 °C \cdot min ⁻¹ from 50 °C to 800 °C under nitrogen flow.

Results and discussion

The UL-94 V Flammability ratings provide an indication of material's ability to extinguish a flame once ignited. The results (Table 1) revealed that the addition of 20 wt.-% of IS1 into neat PP is enough to reach V-0 rating (short burn without dripping). In contrast, IS2 at the same concentration level of IS1 did not pass the UL94 V rating (the test specimen is completely burnt up to the sample holding clamp; Fig. 1 digital pictures taken during the UL-94 V test).

The LOI testing allows to measure the minimum oxygen concentration needed for the self-sustained combustion of polymeric material. A higher increase of the oxygen concentration for ignition was also found with the addition of novel intumescent flame retardant than traditional one to neat PP (Table 1).

The combustion behavior of the PP/IS composites was investigated by means of cone calorimeter under a heat flux of 35 kW/m² and the fire response parameters measured are reported in Table 1. As a general conclusion, novel intumescent flame retardant showed better flame retardancy performance than the traditional one pointing out that the intumescent char formed resists oxidation better; this aspect was confirmed also by the thermogravimetry analysis, in other words we investigated the synergistic effect between APP and PPM Triazine HF (IS1) as well as APP and PER (IS2) during their pyrolysis by comparing the experimental TG curves of the mixtures with the calculated ones. It is worthy to note that (Fig 2), although the char residue at 500 °C (char formation) is almost the same for both types of IS, however at 800 °C (char degradation) for the APP-PPM Triazine HF mixture is substantially increased (48%) in comparison with that of the APP-PER mixture (18%) pointing out the higher oxidation stability of the char. Based on the final char yield it can be assumed that the synergistic effectiveness is better for APP-MMP Triazine HF mixture than APP-PER mixture.

In the light of the aforementioned findings, we concluded that, the novel intumescent flame retardant is more suitable for flame retardancy of polypropylene towards to traditional one at just 20 wt.% loading.

Table 1. Cone calorimeter data of neat PP and the PP/IS composites at kW/m^2 .

Sample	UL-94V	LOI	$TTI \pm \sigma$ (s)	$\frac{\text{PHRR}\pm\sigma}{(\text{kW}\cdot\text{m}^{-2})}$	$FIGRA \pm \sigma$ $(kW \cdot m^{-2} \cdot s^{1})$	$FPI \pm \sigma$ $(s \cdot kW^{-1} \cdot m^2)$	$THR \pm \sigma$ (MJ· m ⁻²)	$ASEA \pm \sigma$ $(m \cdot kg)$	$TSR \pm \sigma$ (m · m)
neat PP	Unclassified	19	62±9	1221±28	3.35±0.13	0.05±0.01	265±16	604±27	3144±135
PP/10IS1	Unclassified	26	44±1	313±12	1.21 ± 0.05	0.16±0.02	235±5	638±29	3305±139
PP/15IS1	Unclassified	29	45±2	148±18	0.46 ± 0.07	0.31±0.03	191±9	399±96	1985±567
PP/20IS1	V-0	31	43±4	115±8	0.24 ± 0.06	0.38±0.05	153±42	313±8	1309±346
PP/20IS2	Unclassified	24	35	136 ±15	2.26±0.16	0.27±0.03	167±16	540±116	2332±392

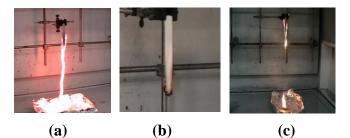


Fig.1.UL-94V of (a) neat PP, (b) PP/20IS1 and (c) PP/20IS2

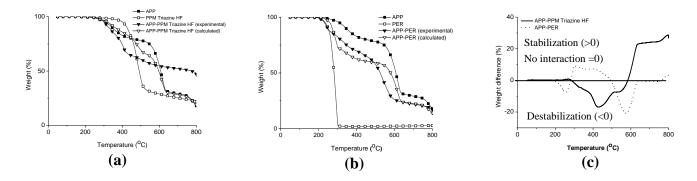


Fig.2. (a) Experimental TG curves under nitrogen atmosphere of APP, MMP Triazine HF, APP-MMP Triazine HF mixture and calculated curve obtained for this mixture, (b) Experimental TG curves under nitrogen atmosphere of APP, PER, APP-PER mixture and calculated curve obtained for this mixture and (c) curves of the weight difference between APP-MMP Triazine HF experimental and calculated and APP-PER experimental and calculated.

References

- 1. Troitzsch T. Plastic Flammability Handbook, Principles, Regulations, Testing, and Approval, Carl Hanser Verlang, Munich, 3rd Edition, 2004.
- 2. Vasile C. Handbook of Polyolefins, Revised and Expanded, New York: Marcel Dekker Inc, 2nd Edition, 2000.
- 3. Raymond Hindersinn R. In Fire and Polymers: Historical Aspects of Polymer Fire Retardance, ACS Symposium Series 425, 1990; Chapter 7, p.87-89.