PHOSPHINIC ACID FUNCTIONALIZED POLYETHYLENE IMINE (HPEI) – A NOVEL FLAME RETARDANT AGENT FOR GLASS REINFORCED POLYBUTYLENE TEREPHTALATE

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Introduction

Glass fiber-reinforced Poly(Butylene Terephthalate) (GF-PBT) is a thermoplastic widely used for electrical and electronic devices. Since it is easily flammable, it has to be flame retarded to comply with fire standards[1]. The objective of this study is to synthesize a novel flame retardant agent for GF-PBT: a phosphinic acid functionalized hyperbranched-polyethylene imine (HPEI). This approach was motivated by the P/N synergism widely reported in the field of flame retardancy[2] and by the high nitrogen content of HPEI. Fire performances of the material will be evaluated using standardized fire tests (UL94, Limiting Oxygen Index, cone calorimeter).

Materials and Methods

The phosphinic acid functionalized hyperbranched-polyethylene imine (fHPEI – Figure 1) was obtained from a reaction between hyperbranched polyethylene imine (Lupasol WF from BASF) and oxophospholane oxide (Exolit PE110 from Clariant). The reaction was carried out in CH_3Cl at 60°C for 6h. The reaction products were then precipitated in diethyl ether and used after drying.

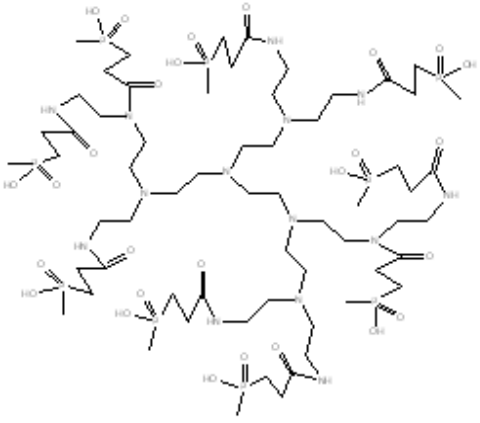

Figure 1. Phosphinic acid functionalized polyethylene imine (f-HPEI)

The flame retardant additive (fHPEI) was incorporated to PBT/GF using a micro-compounder from DSM Xplore (3min, 250°C, 80 rpm). UL-94 classification was obtained on sheets (130x12.7x 0.8cm^3) according to the conditions of the standard test (ASTM D 3801). Limiting Oxygen Index (LOI) was carried out according to ISO4589 on barrels (10x10x0.3cm^3). Mass Loss Calorimeter was carried out on samples (10x10x0.3cm^3) following the procedure defined in ASTM E 906 at an external heat flux of 35 kW/m² (mild fire scenario).

Results and Discussion

Table 1 presents the flame retardant properties of PBT/GF including 20 and 30wt-% fHPEI. It is observed that the addition of fHPEI in PBT/GF leads to an increase in the flame retardant properties. The LOI increases from 19vol.-% for PBT/GF to 27vol.-% when 30wt.-% additives are incorporated in the PBT/GF matrix. This latters material achieves V0 classification at UL94 test whereas at 20wt.-% only V2 classification is obtained. Those results also show that at 20wt% of fHPEI in PBT/GF lead to a 61% decrease of the peak of Heat Release Rate (pHRR) as compared to PBT/GF whereas the time to ignition decreases. The total heat release (THR) is also sharply reduced when fHPEI is used as flame retardant in PBT/GF.

Table 1. Flame retardant properties of PBT/GF and PBT/GF/fHPEI

	LOI vol.-%	UL94 rating	PHRR (kW/m²)	TTI (s)	THR (MJ/m²)
PBT+25%GF	19	NC	400	62	52.8
PBT+25%GF+20%f-HPEI	23	V2	154	39	34.6
PBT+25%GF+30%f-HPEI	27	V0	-	-	-

Conclusion

A novel flame retardant additive (phosphinic acid functionalized hyperbranched-polyethylene imine) was successfully synthesized. Its efficiency as flame retardant additive in reinforced PBT was demonstrated using various standardized fire tests.

References

1. Levchick S.V., Weil E.D., Polym. Int. 54:11-35, 2005.
2. Leu T.S., Wang C.S. J. App. Polym. Sc. 92(1):410-417 (2004)