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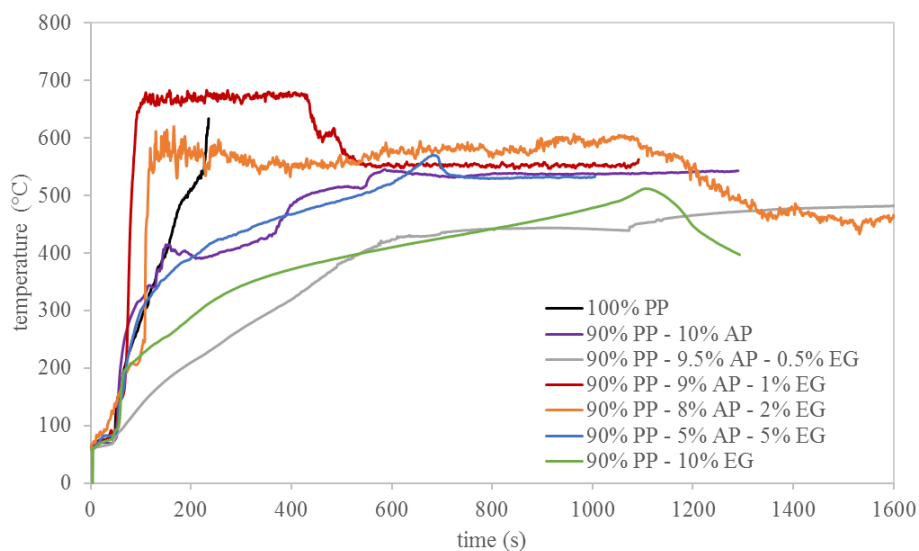
# INTUMESCENT POLYPROPYLENE: INTERACTIONS BETWEEN PHYSICAL AND CHEMICAL EXPANSION

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To limit heat release from polymeric materials subjected to a flame, intumescent additives are incorporated into the polymeric matrix. These additives act as flame-retardants by forming a coating which protects the underlying material from the action of the heat flux or the flame. This study focuses on the behavior of two flame-retardants and their interactions in polypropylene (PP): expandable graphite (EG) and modified ammonium polyphosphate (AP). These two materials were chosen because they represent two types of intumescence: physical expansion for EG and chemical expansion for AP.

PP was blended with 10%wt flame-retardants in a twin-screw extruder. Different ratios AP:EG were evaluated: 10:0, 9.5:1.5, 9:1, 8:2, 5:5 and 0:10 (wt/wt). Samples measuring 100 mm x 100 mm x 3 mm were exposed in horizontal orientation to an external heat flux of 35 kW.m<sup>-2</sup> at 35 mm from the electrical resistance in a mass loss cone calorimeter. Heat release rate (HRR) was measured as a function of time following the procedure defined in ASTM E 906. In addition, a thermocouple was embedded in the polymer at the backside to measure the temperature changes as a function of time during the cone calorimetry experiment. The purpose is to quantify the efficiency of the intumescent barrier. Results were compared to those obtained with neat PP.

Figure 1 shows the temperature as a function of time at the backside of the intumescent materials. Compared to neat PP, the temperature rise is slower with intumescent PPs containing 10% AP, 10% EG and 5% AP-5% EG. By contrast, with PP containing AP and a small amount of EG (9% AP-1% EG and 8% AP-2% EG), an important temperature rise followed by a stabilization is observed.



*Figure 1: Temperature as a function of time at the backside of intumescent PPs during cone calorimetry experiment (external heat flux = 35 kW.m<sup>-2</sup>)*

This unexpected behavior of the two latter systems is also visible on HRR curves (Figure 2). Peaks of HRR are about 70% lower for intumescent PPs with ratios AP:EG 10:0, 5:5 and 0:10 than for neat PP. With 9:1 and 8:2 systems, peaks of HRR are only decreased by about 30%.

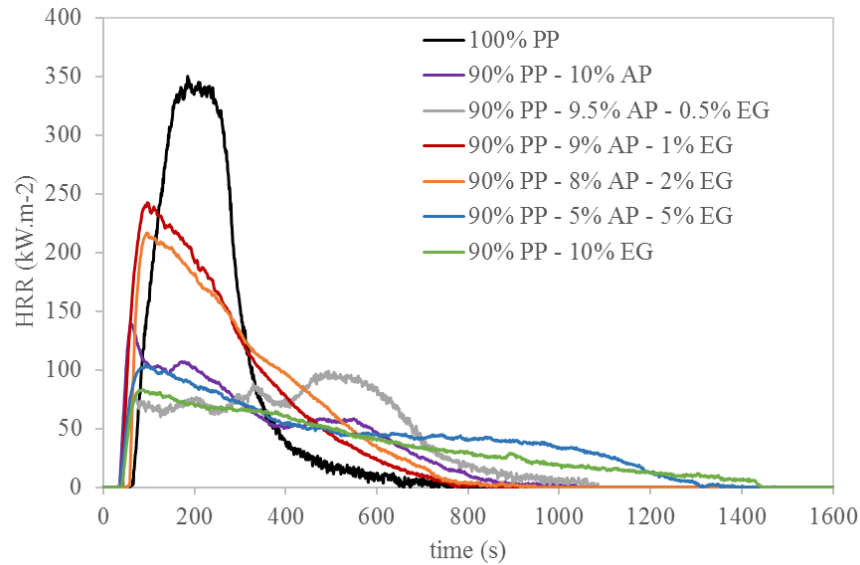


Figure 2: Heat release rate (HRR) as a function of time of intumescent PPs during cone calorimetry experiment (external heat flux = 35 kW/m<sup>2</sup>)

The nature of intumescent phenomena could explain these observations. With AP, an intumescent char is formed by a series of chemical reactions during the burning. The char exhibits high expansion and a smooth surface. With EG, expansion is due to the decomposition of the intercalation compounds inherent in the EG structure (sulfate in this case) into gaseous products. These products blast off the graphite flakes and an entangled network of graphite worms is formed at the surface of the material. In this last case, expansion begins before the ignition. Figure 3 presents pictures of char residues obtained after the cone calorimetry experiments for the different samples. When AP and EG are both present in the polymer (5% AP – 5% EG), visual observations suggest that the network of graphite worms is trapped in the intumescent char formed from AP. However, when EG is in small amount (1% and 2%), there are not enough worms to form an entangled network. Worms are trapped in vertical position. They may then disturb the action of AP and enhance heat conductivity through the material. This could explain the trends observed in figures 1 and 2.

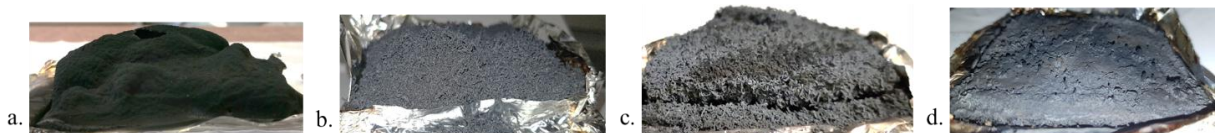


Figure 3: Char residues after cone calorimetry experiment for PP with fire-retardants in different proportions (AP:EG): a. 10:0; b. 0:10; c. 5:5; d. 9:1

The sample with 9.5% AP and 0.5% EG does not present the particular behavior of systems with small amount of graphite. Performance of this system is close to that of PP with 10% AP. It can be assumed that, from a certain threshold, there is too little graphite to disturb the action of AP and modify heat conductivity.

Results may suggest that an anisotropy is induced by worms of graphite and allows EG to dissipate heat during PP burning. Nevertheless, when EG is added in small amount in a chemical intumescent system, worms disturb the system, increase the transverse heat conductivity and decrease the fire performance.