Advanced characterization of decomposing flame-retarded polymers

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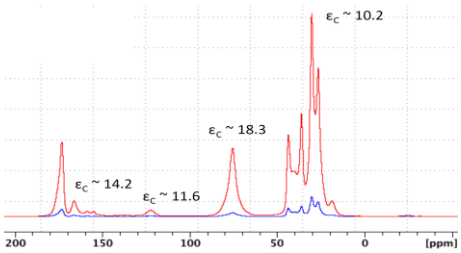
Flame-retardants (FRs) are incorporated into polymers to reduce the fire hazards. FRs can modify the decomposition pathway of polymers to yield char and/or to change the evolving gaseous products [1]. The aim of this paper is to show how to characterize decomposing FR polymers using advanced characterizations namely, solid state NMR (2D NMR and dynamic nuclear polarization-enhanced (DNP) NMR) and conceptualization (fractal approach).

Solid state NMR

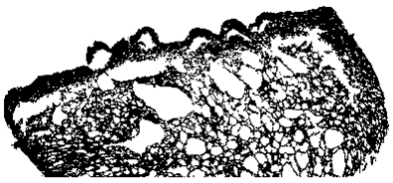
Dynamic nuclear polarization (DNP) is a technique that significantly enhances the sensitivity of NMR spectroscopy by transferring using microwave irradiation the very large polarization of electrons to nuclei. Under the right conditions, DNP enhancements can reach several orders of magnitude but it has never been applied to decomposed polymers [2]. A typical example is polyamide-6 (PA-6) containing cyanurate melamine (MCA) as FR heat treated at 400°C where the comparison of CP-MAS ^{13}C NMR with and without DNP is shown on Figure 1 – (a).

Fractal dimension of intumescent char [3]

An intumescent coating submitted to hydrocarbon fire test was analyzed after testing by X-ray micro-computed tomography (µCT) (Figure 1 – (b)). Quantitative analyses of mass density autocorrelations were performed using fractal analysis (box-counting technique). The calculation of the fractal dimension gives 1.8 on 100-800 µm.



(a)



(b)

Figure 1: (a) 1D $^{1}H \rightarrow ^{13}C$ CP-MAS NMR spectrum of PA-6/MCA heat treated at 400°C with and without DNP (ε gives the enhancement of signal intensity) and (b) binarized slice images of the inner structures of the char (1 pixel = 80 microns) given by low resolution X-ray µCT

References

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