

## Polystyrene Nanocomposites after Melt Intercalation

Polymer intercalation into clays increases tensile and yield strengths, reduces flammability and lowers permeability. Melt intercalation exposes polymers to thermal and shear histories that can induce degradation. An extruded macrocomposite comprising polystyrene and montmorillonite was statically annealed in an O<sub>2</sub>-rich environment at 250°C to induce localized chain scission. The smaller chains are more thermodynamically favored for insertion into the clay interlayer and can, therefore, transform the macrocomposite into an intercalated nanocomposite.

Molecular weight distributions were characterized with a DAWN detector and Optilab refractometer, along with a Waters 2950 HPLC system. Size-exclusion chromatography was typically performed at 25°C and 0.5 mL/min in tetrahydrofuran with Styragel® columns and all runs were analyzed with ASTRA®.

Annealing shifts the first maximum in the MALS and differential refractive index (DRI) signals (Fig. 1) to higher elution volumes, with the shift in the DRI signal being more pronounced than the MALS signal since DRI is based on the number of molecules and is therefore more sensitive to chain scission.

If linear PS chains constitute the scission products, then the chains eluting after the first maximum would be of reduced molecular weight. However, the MALS signal grows faster than the DRI signal, which is attributed to the development of branched PS chains with a reduced hydrodynamic size that elute later. Universal calibration *incorrectly* reports molecular weights for the second maximum.

To determine the extent of non-linear chains generated, the root-mean-square (RMS) radius is plotted in Fig. 2 as a function of molar mass. The RMS radius (after 3.5 h) increases with molar mass, and the corresponding scaling exponent is 0.5, which is consistent with Gaussian coils composed of linear chains. After annealing for 28 h or more, the RMS radius in the vicinity of 10<sup>5</sup> Da becomes highly degenerate, indicating formation of high-molecular-weight, branched chains.

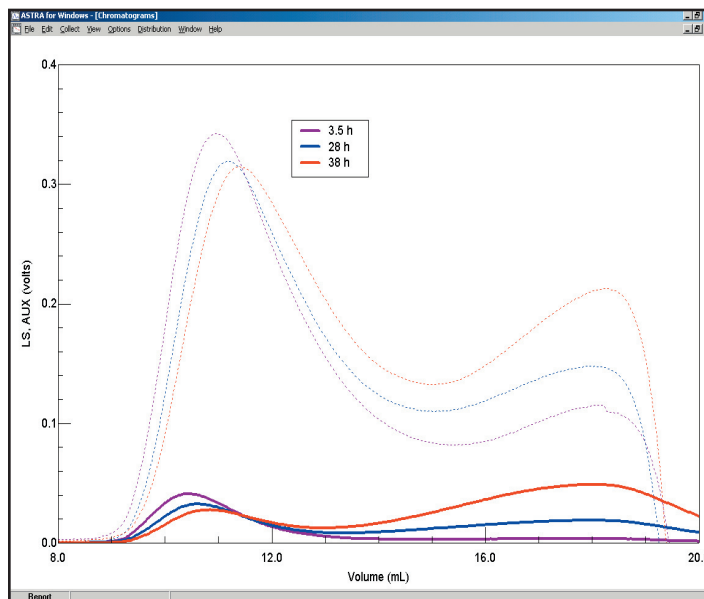


Figure 1. Complementary MALS (solid lines) and DRI (dashed lines) signals after annealing at different times (see legend) in an O<sub>2</sub>-rich environment at 250°C.

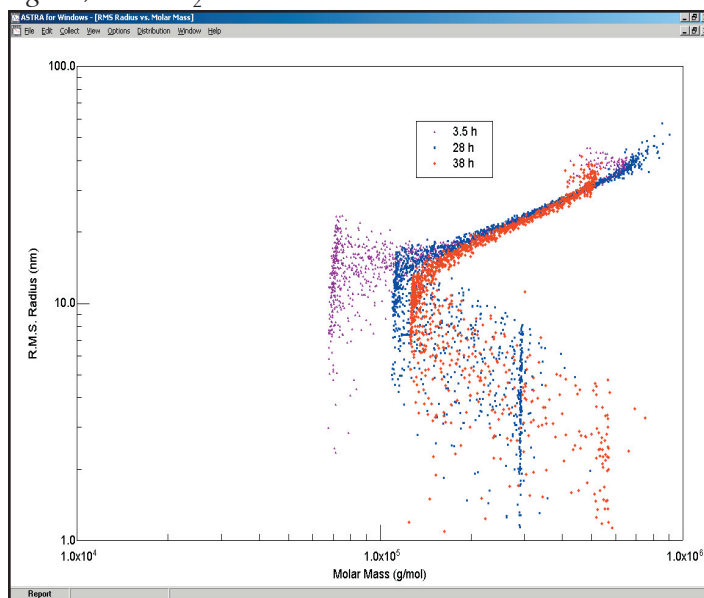


Figure 2. Abnormal elution behavior indicative of PS branching or other nonlinear molecular architectures after prolonged annealing times (in h)—3.5 (purple), 28 (blue) and 38 (red)—in an O<sub>2</sub>-rich environment at 250°C.

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