

Butadienes: Thermal FFF vs. GPC/SEC

It has always been known that large elastomer molecules can undergo shear degradation when separated using traditional Gel Permeation/Size Exclusion Chromatography (GPC/SEC) columns. To demonstrate this phenomenon, two samples of polybutadiene with a known 30% difference in relative viscosity were prepared and analyzed using *both* SEC and Thermal FFF.

ThFFF is an open channel separation method that separates molecules on the basis of their thermal diffusion coefficient. As there is no stationary packing phase involved in the separation, shear effects are minimized, if not completely eliminated. Because of this, Thermal FFF combined with a DAWN is the ideal analytical technique for characterizing very high molar mass molecules.

Figure 1 illustrates the separation obtained using SEC-MALS. Despite their large difference in viscosity, both samples appeared to be virtually identical, with a peak molar mass around 500,000 g/mol. The distributions were also observed to have a long low molar mass tail.

By comparison, Figure 2 shows the distributions obtained by ThFFF-MALS for the same samples. These were highly non-gaussian, making the determination of "peak" molar mass impossible. However, their median MWs varied from 1,000,000 g/mol., in the case of the low viscosity material, to around 2,000,000 g/mol. for the high. This shift in MWD would easily account for the observed viscosity.

It should be noted that the SEC results gave *lower* MWD than with those analysed by ThFFF, demonstrating the degradative effect of using packed column chromatography when separating shear-sensitive polymers.

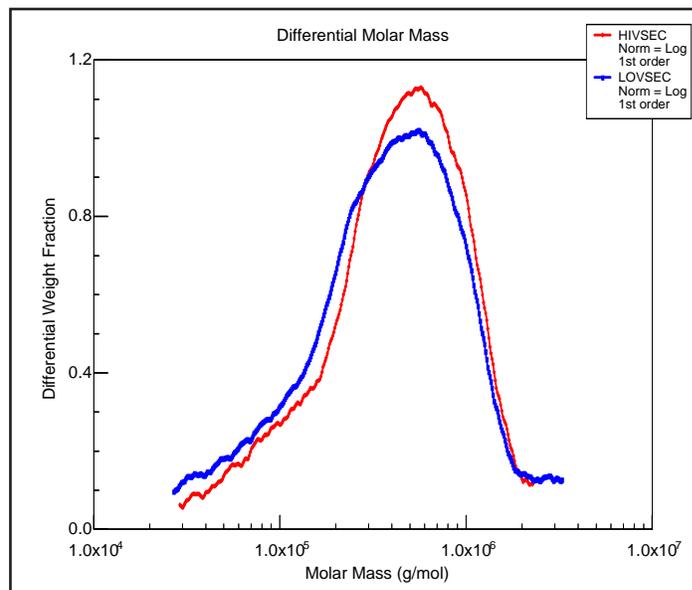


Figure 1. Differential mass distribution shown for the GPC/SEC separation, showing a maximum molar mass of about 2 million.

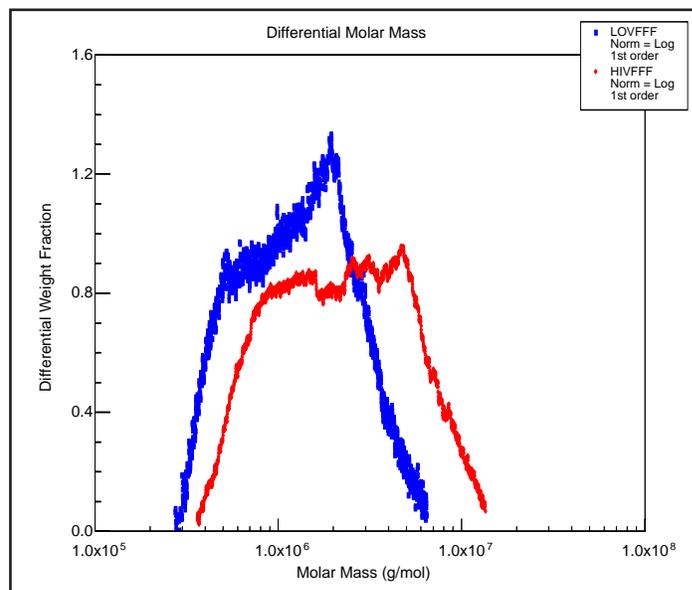
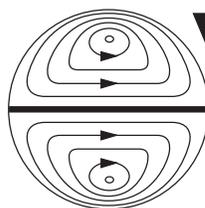


Figure 2. Thermal FFF separation creates a distribution significantly higher than by GPC/SEC, indicating the shear forces in conventional chromatography were absent with ThFFF.

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