Multidimensional high temperature liquid chromatography.

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The discovery that polyolefins can be adsorbed from dilute solution on graphite and then selectively be desorbed by using gradients of either solvent or temperature opened the door to separate polyolefins and olefin copolymers according to composition or microstructure by liquid chromatography [1,2]. Subsequently corresponding multidimensional chromatographic techniques were developed to further enhanced the molecular information which can be retrieved from a fractionation based approach [3,4].

Liquid chromatography at critical conditions (LCCC) is a key chromatographic technique which enables, for example, to separate homopolymers according to different end groups. In LCCC the elution of the chains occurs independent of their molar mass for a given monomer unit (Fig. 1a) [5].

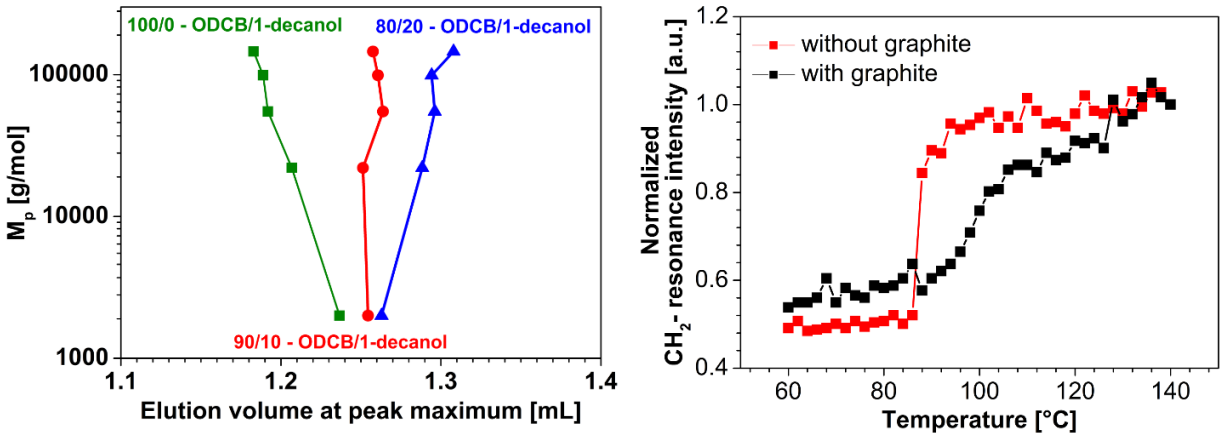

Fig. 1: a) correlation between elution volume at peak maximum and the average molar mass (Mp) of PE in ortho dichlorobenzene (ODCB)/1-decanol and b) concentration of PE in solution in the system graphite/ODCB as monitored by 1H-NMR.

The dynamic development of chromatographic techniques for polyolefins also created the need to understand the mechanism underlying the interaction between polyolefins and graphitic surfaces in solution. In particular Nuclear Magnetic Resonance using carefully optimized experimental parameters has been proven to be a powerful technique to monitor the adsorption of polyolefin chains of the surface of graphite (Fig. 1b).

The recent progress in HT HPLC of polyolefins will be reviewed, giving particular emphasis on the development and applications of LCCC for polyolefins. Evidence about the mechanism of interaction will be described.

References:

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