Modern Analysis Methods for Polyolefin Compounds

The Fraunhofer Institute for Structural Durability and System Reliability LBF has recently commissioned a newly developed chromatograph for polyolefin chromatography. It is unique in the way it applies novel spectroscopic detection technology. This opens entirely new perspectives for the characterization of polyolefins, olefin copolymers and their formulations.

Safety-relevant components are increasingly fabricated from plastics. This development is, particularly with regard to polyolefins, driven by a wide variability of their property profiles and the economic attractiveness as compared to traditional materials. The application properties of polyolefins are rooted in their molecular structure. Important molecular parameters are the chemical composition, the average molecular weights and the molecular weight distribution. For copolymers, the chemical heterogeneity comes into focus, which is hardly assessable in routine analysis. In formulation development, additives have become a driving factor to tune the properties. A particular challenge in this field is to ensure consistency with regard to the type and content of additives. The determination of the aforementioned parameters has been conducted at Fraunhofer LBF for some time now. According to the researchers at Fraunhofer LBF, modern chromatographic methods are indispensable tools during the development of polyolefins and for ensuring their quality. They allow to separate the polymer chains contained in a material sample according to their size and chemistry and then to quantify them. This makes these methods a highly valuable instrument for the analysis of material samples in the sense of an incoming goods inspection, for example to detect off-specification batches, as well as for the quantitative determination of usage-induced material changes (*Figure 1*).

Monitoring batch consistency – precisely and quickly

In routine analysis, gel permeation chromatography (GPC) is a very common method for the molecular characterization of polyolefins as well as other polymers. It allows to separate a sample according to molecular weight. Typically, only the concentration of the separated molecules is determined. Information regarding their composition is not gained in this manner, however. The exact determination of the chemical composition does thus present a challenge to the detectors employed, which can only be mastered via a combination of suitable spectroscopic techniques. In particular with regard to poly-

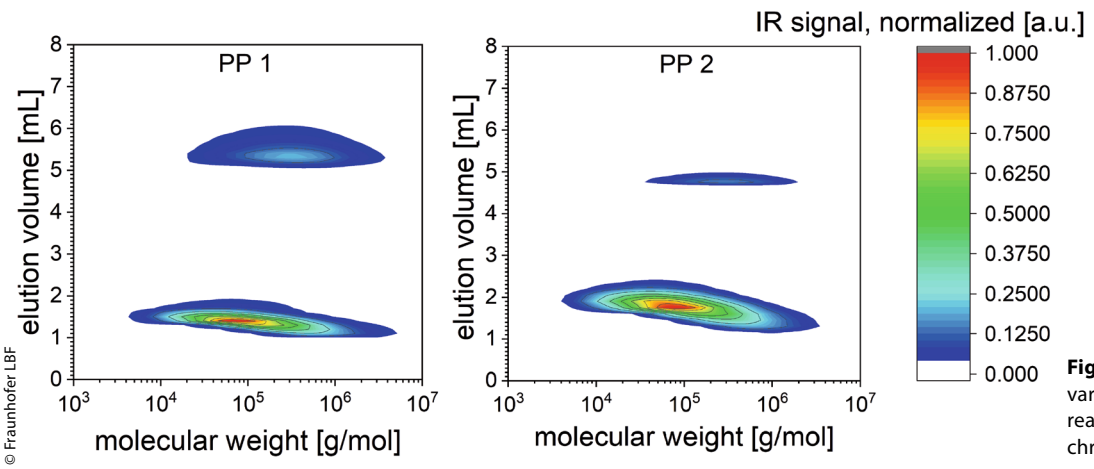

Figure 1 > Compositional variability of propylene-based reactor blends as identified by chromatography

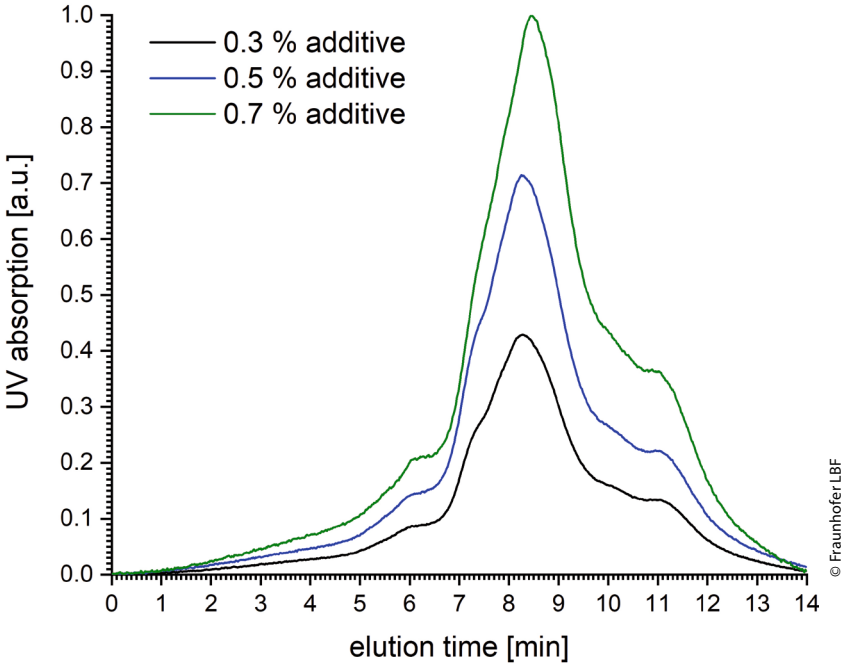

Figure 2 > Detection of varying additive contents in polyethylene by GPC-UV

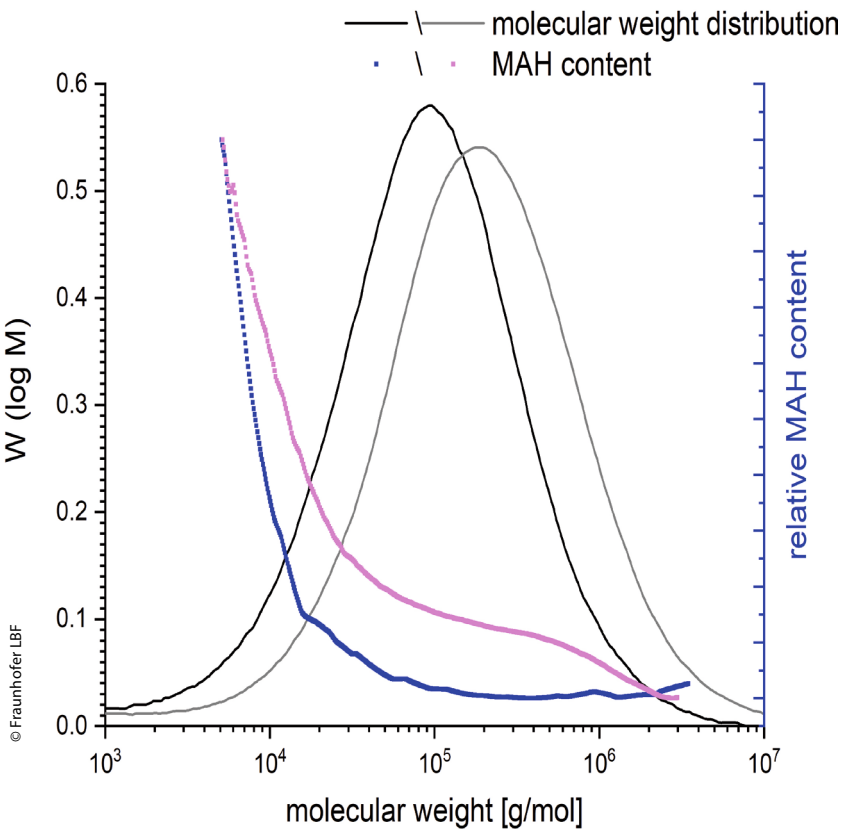

Figure 3 > Comparison of MAH content as a function of molecular weight for two polyolefin samples modified by means of reactive extrusion – determined from GPC-IR data

olefins, a technological gap existed here, as the semi-crystalline nature of these polymers makes elevated temperatures necessary for their dissolution, requiring suitably heat resistant instrumentation for their analysis. The new chromatograph from Polymer Char, which has recently been commissioned by Fraunhofer LBF is capable of operating up to 200 °C. The instrument is equipped with two newly developed detectors, an infrared (IR) and a UV detector, covering the whole spectrum of vibrational spectroscopy. According to the LBF, this offers entirely new perspectives for the analysis of polyolefins, olefin copolymers and their compounds. For example, propylene-based reactor blends could be analyzed with hitherto unknown precision, revealing batch variations.

Comprehensive consideration of formulations

Nowadays, the application properties of formulations are tailored flexibly and in a cost-efficient manner using additives. Existing methods for their characterization, which necessitate an enormous amount of time and labor, are, however, not suited for close-knit quality control. A general problem is the fact that additives that are hard to extract cannot be analyzed at all in this manner. Here, the newly developed IR and UV detection capability offers innovative options for a fast and accurate analysis of additives. Formulations may be separated chromatographically and the eluting fractions characterized spectroscopically in a single step (*Figure 2*). This expands the spectrum of additive analysis dramatically.

Reactive extrusion

Reactive extrusion is a highly flexible and economical option to produce polyolefin compounds with tailor-made property profiles. A well-known example is the modification of polyethylene or polypropylene with maleic anhydride (MAH) to increase the polarity. The final properties of the modified polyolefins are, in particular, determined by the amount and the distribution of MAH groups along and across the molecular weight axis. Enhancing the GPC elugram by compositional information, the new chromatograph gives crucial information regarding the quality of the MAH-modified material in just one step (*Figure 3*).

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