Development of an in-line filter for analysis of complex polyolefin compounds with CRYSTEX technique and comparison with off-line filtration

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Industrial polymer samples may contain different kinds of fillers and pigments to impart some desired mechanical properties. All those may cause difficulties during the analysis of the polyolefin compounds, either by interfering the determinations of some properties or, even worse, by plugging the columns or damaging the instruments. To remove those interfering species, it is necessary to filter the solution, which becomes more difficult in high-temperature applications (polyolefins). In the case of CRYSTEX it is specially challenging due to the high concentration of sample analysed (normally 10-20mg/ml for PP).

Off-line filtration of the sample solutions has been traditionally the only option, which was tedious and difficult due to the elevated temperature required to prevent precipitation of the polymer. Moreover, when a large volume of solution needs to be prepared (up to 200 mL are required to dissolve 2 – 4 grams of PP and EP materials for CRYSTEX QC analysis) the off-line filtration becomes highly impractical.

In this project an in-line filter was developed which is placed in the CRYSTEX QC sampling line inside of the instrument oven, kept isothermal at elevated temperature. The filtration process is therefore integrated within the automated analytical workflow, and performed with no user intervention. One advantage is that only a minimal portion of the prepared solution is filtered, that is required to be loaded into the crystallization column for the analysis.

The optimization of the analysis method when using this filter and results of durability testing are presented. Advantages and limitations of this in-line filtration method are discussed as well as a comparison to external off-line filtration using an EFS device, which was developed for filtration of carbon black or other small size particle fillers prior to GPC analysis.

References

[1] Soluble fraction analysis in polypropylene for QC (CRYSTEX® QC) LCGC - LCGC EU and NA. The Applications Notebook, December 2013.

[2] Rapid characterization of high-impact ethylene–propylene copolymer composition by crystallization extraction separation: comparability to standard separation methods, Int. J. Polym. Anal. Charact., Vol 25, 2020 – 8, L. Jeremic, A. Albrecht , M. Sandholzer, M. Gahleitner

[3] ISO-16152:2022 Plastics – Determination of xylene-soluble matter in polypropylene

[4] Filtration of polymer solutions and carbon black removal prior to a GPC analysis. T. González, B. Monrabal, E. López, A. Roig, ICPC 2018, Houston