Evaluation of Solvent Effects in Solution Crystallization Fractionation of Polyolefins.

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Temperature rising elution fractionation (TREF), crystallization analysis fractionation (CRYSTAF) and crystallization elution fractionation (CEF) are the most commonly used fractionation analysis methods for the chemical composition separation of polyolefins. Several factors such as cooling rate, polymer chain length (molar mass), and chemical composition of the individual chains, are known to influence the co-crystallization of various components during their precipitation from solution. These have been extensively investigated by several authors.[1] So far no systematic reports on the effect of various solvents on the crystallization and dissolution of polyolefins in TREF and CRYSTAF have been presented in the literature up to now. This knowledge, however, is essential; in particular for the comparison of results obtained by separation methods which use different solvents, e.g. preparative fractionations such as prep TREF. Frequently, prep TREF is performed in xylene, while the separated fractions are analysed by analytical TREF, CRYSTAF, CEF using solvents like 1,2,4-trichlorobenzene (TCB) and ortho-dichlorobenzene (o-DCB).

Unfortunately, these techniques are limited with regard to the variety of solvents that are used for fractionation and analysis. In the present study, the effect of various solvents on the solution crystallization and dissolution of polyolefins were investigated by using a unique analytical tool - solution crystallization analysis by laser light scattering (SCALLS).[2] For a blend of polyethylene and isotactic polypropylene standards, it was found that co-crystallization effects are minimal in solvents such as TCB and o-DCB, whereas co-dissolution effects are minimal in solvent xylene. In the following step, effects of two different solvents, xylene and TCB, and their influence on solution crystallization of chemically different components of an heterophasic ethylene-propylene copolymer were systematically investigated by using a simple preparative fractionation approach named as preparative solution crystallization fractionation (prep SCF).[3] A detailed analysis of the separated components was obtained by combining prep SCF with a variety of analytical techniques including CRYSTAF, FTIR, DSC and high temperature solvent gradient interaction chromatography.

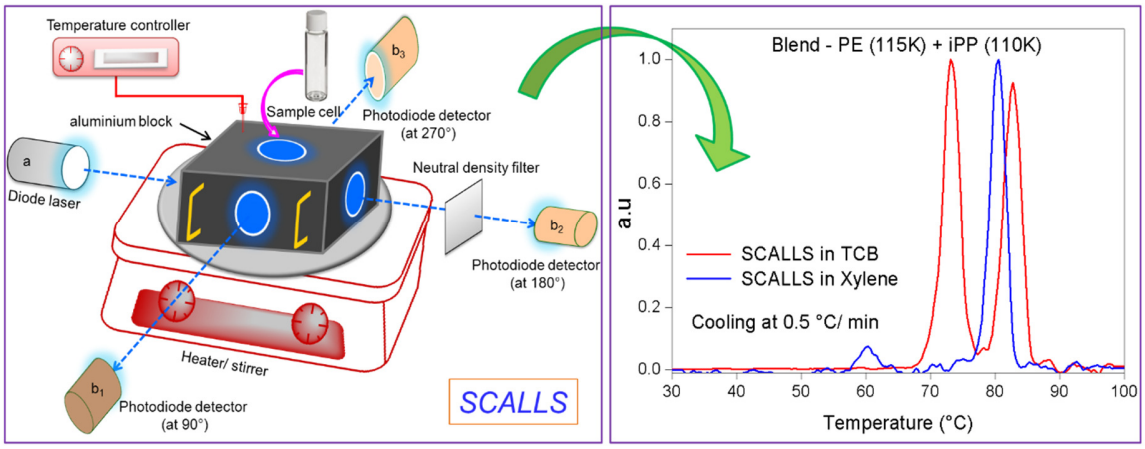

Figure illustrates SCALLS instrument set up (left) and solution crystallization behavior obtained for a blend of PE and iPP in solvents, xylene and TCB (right).

References:

1. S Cheruthazhekatt, N Mayo, B Monrabal, H Pasch. Macr. Chemistry and Physics 214 (19), 2013, 2165-2171.
2. S Cheruthazhekatt, DD Robertson, M Brand, A van Reenen, H Pasch. Analytical Chemistry 85 (15), 2013, 7019-7023.
3. S Cheruthazhekatt, H Pasch. Analytical and Bioanalytical Chemistry, 2014, DOI: 10.1007/s00216-014-7714-y.