Coupling of liquid chromatography and fourier-transform infrared spectroscopy for the characterization of polymers

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Epilogue

Coupling of LC and FTIR spectroscopy has proved to be a valuable qualitative tool for the characterization of polymers. As demonstrated in this thesis, it has a high potential to reveal differences in functionality, for instance when aged polymers are studied, or to detect differences in the chemical composition of (co)polymers. In this area FTIR can be advantageous in comparison with MS-techniques.

The quantitative aspects of a commercially available solvent-elimination interface have been extensively investigated and it seems rather unlikely that quantitative results can be obtained for a large number of polymers differing in physical properties and across a broad concentration range. For quantitative, SEC or isocratic LC the use of a flow cell must be considered. However, combining LC and FTIR spectroscopy via a solvent-elimination interface still provides an excellent tool that can be used for (i) screening of functional groups present (or absent) in a sample without interferences arising from other sample constituents or eluent and (ii) elucidation of molecular structure. The position of LC–FTIR with respect to its applicability to polymer characterization is between LC–MS and LC–NMR. The spectra acquired from LC–FTIR are much easier to interpret than LC–MS spectra. NMR is still an expensive and time-consuming technique, especially when coupled to LC. This is due to the use of deuterated solvents (for $^1$H-NMR) and to increased scanning times for elements with a low natural abundance (e.g. $^{13}$C-NMR).

The popularity of two-dimensional chromatography (LC×SEC) is growing and this technique can bring homogeneous fractions to the detector with respect to chemical composition and molar mass. A combination of LC×SEC and FTIR spectroscopy is therefore a valuable tool, which has a high potential to reveal chemical differences between polymers. However, for a wide acceptance it is necessary to reduce the total analysis time. This can be achieved by a decreased second-dimension run-time while maintaining an adequate chromatographic resolution. Further research is therefore necessary in the field of high-speed SEC. Furthermore, interpretation of large LC×SEC–FTIR data-sets is tedious and time-consuming. Therefore, it is expected that (new) multivariate-data-analysis techniques such as DifSub, will be increasingly exploited in the near future for the interpretation of such large data sets. It is quite possible that at some point LC×SEC will be used on a routinely basis, e.g. in aging studies, for quality control, or for competitor-product analysis. Since most of the polymers produced are non-UV-active, there is a major role for FTIR as a universal and selective detector.
Finally, an increased acceptance of LC–FTIR is foreseen when optimization of the nebulization parameters (e.g. nebulizer temperature and gas flow rate) will be fully automated in the case of solvent-elimination interfaces. In addition, the possibility of quantitative detection is another prerequisite for a widespread use of LC–FTIR. However, the lack of intelligent data-processing software that automatically retrieves the most-important features along a chromatographic run still causes a need for a spectroscopy expert to extract the required (functional-group) information. Therefore, LC–FTIR remains a ‘state-of-the-art’ technique, despite the availability of commercial interfaces.