

Characterisation of Adhesive Materials

Adhesive formulations are often very complex and the chemical and physical characterisation of these materials requires a multi-disciplinary approach.

Intertek MSG is able to characterise these complicated systems and has much to offer companies that are seeking to prove and improve their products and/or understand competitors' formulations. It works with clients in the pharmaceutical, medical and agrochemical sectors as well as for those in the personal care and foods arenas, in addition to its traditional heartland of the chemicals, polymers and plastics industries. Its high-level capability includes instrumentation and many years of expertise that is appropriate to characterisation and problem solving in the adhesives field.

Activities within MSG that are relevant to adhesive characterisation are shown in the following diagram.

Some more specific examples of chemical characterisation of adhesive formulations are described below.

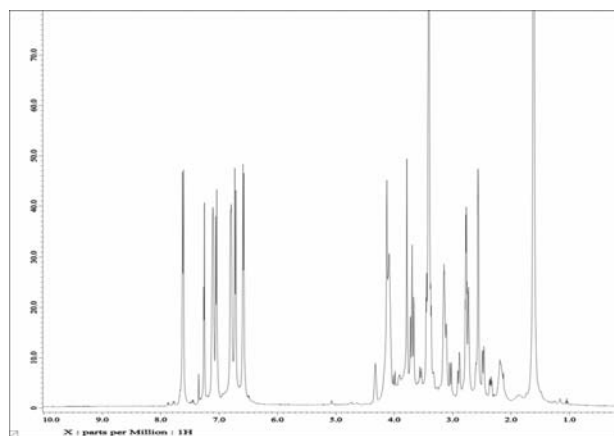
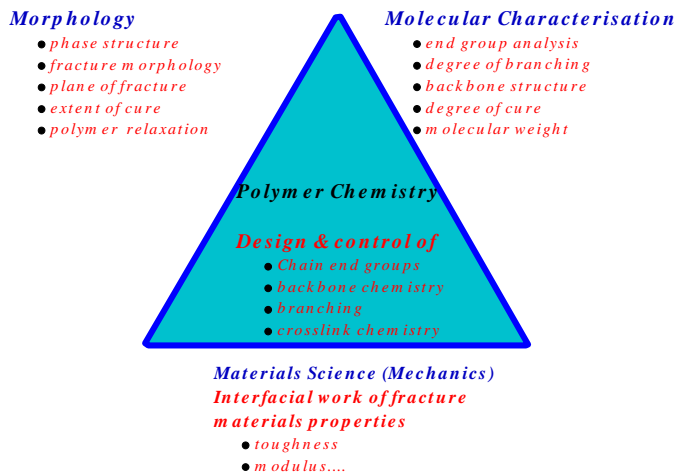
- Contamination
- Technical Service issues
- Technical marketing
- Competitors' products evaluation

Methods of chemical characterisation cover a range of adhesive formulation types and physical states:

- Non-cured/partially cured systems
- Cured systems
- Adhesive layers and composites

Some examples of deformation of adhesives are described below.

A combination of various spectroscopic and mass spectrometric techniques is routinely used by MSG to deformulate adhesives. NMR spectroscopy is typically used to quantify the detailed chemical composition. The spectrum below is from a complex mixture and, although useful, is difficult to interpret due to overlapping signals from different components.



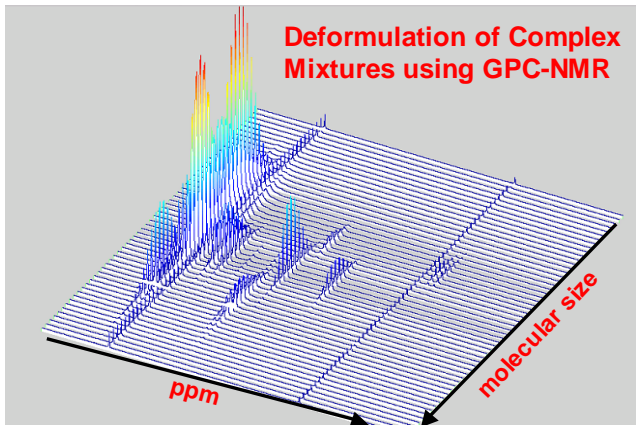
¹H NMR Spectrum from Adhesive

Deformulation

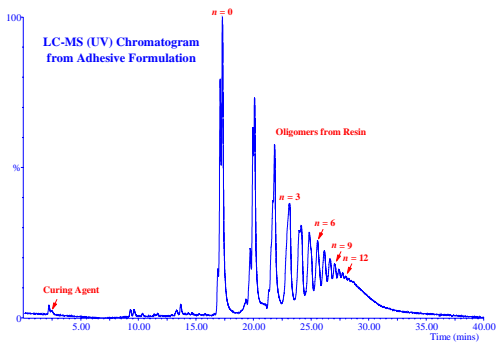
Deformulation of adhesives can be a valuable aid in developing new products or modifying existing formulations, as well as understanding competitors' materials. It can help in a number of areas:

- Development of new products
- Production/formulation problems

By coupling on-line GPC with NMR detection one can separate complex formulations and obtain spectra from the individual components, greatly simplifying the analysis **and** giving the composition as a function of molecular size.

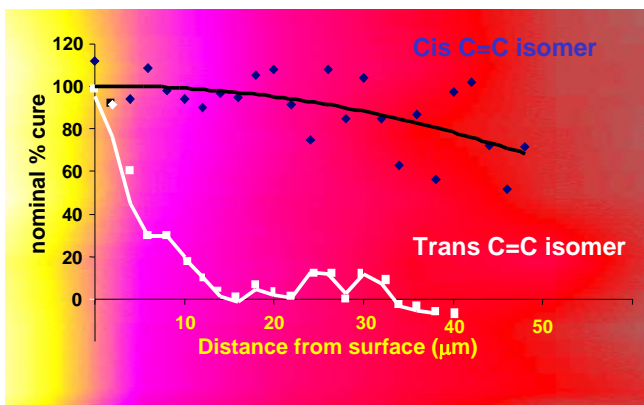


Complementary and further information may be obtained by employing LC-MS, as shown in the example below. Confirmation of the structure of the resin is obtained along with identification of the curing agent in the formulation.



Cure

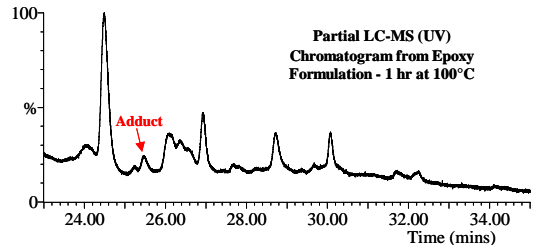
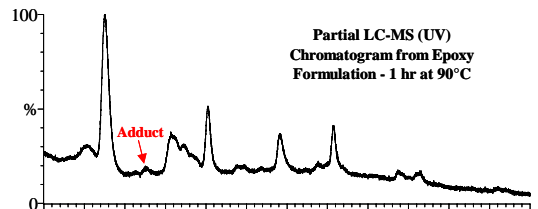
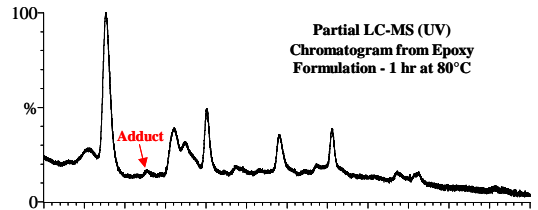
The cure of adhesives may be followed by means of spectroscopic and mass spectrometric techniques.



The above example shows how vibrational spectroscopy can be used to depth profile the extent of cross-linking in a UV cured adhesive. Different reactive isomers cure at different rates, and the extent of cure is depth dependent because of the attenuated UV penetration into the resin.

Mass spectrometry (either MALDI-TOF or LC-MS) may be employed to yield information about the build-up of oligomers during cure. This can include bulk oligomers and the formation of low levels of adducts during mixing. An example of the latter is shown below (where LC-MS has been used to investigate the effect of temperature on adduct formation).

Increase in Abundance of Minor Adducts with Rising Temperature



Thermal Analysis techniques such as Differential Scanning Calorimetry (DSC) or Thermogravimetric Analysis (TGA) are useful in studying the changes in physical properties that can occur during the cure process.

If there is any interest in the above capabilities, please address your enquiries to Drs Allan Stewart or Isla Mathieson by the following means.

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