

THE DEGREE OF POLYMER CURING, AS MEASURED BY ATR SPECTROSCOPY

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INTRODUCTION

The degree of cure is an important QC parameter in many applications. The extent of polymerization determines the physical properties of many materials. Typical examples are inks, varnishes, coatings for electronic components, etc. Many of these materials are cured with UV or electron beam radiation.

In this short example we show how the data can be correlated with process parameters which in turn determine the basic properties of the final product. In this example, the critical parameter is adhesive performance, known

to be dependent on the equivalent coating speed.

EXPERIMENTAL

In all cases, it is possible to use the Split Pea accessory to obtain excellent spectra in short time. The data we present here are from a series of UV-cured adhesives. These adhesives all use acrylate chemistry.

The samples were irradiated in a pilot plant with UV light, the samples travel through the UV irradiation chamber at different speeds.

The SplitPea ATR accessory, shown in Figure 1, was used to measure spectra quickly and reliably; the



Figure 1. The SplitPea™.

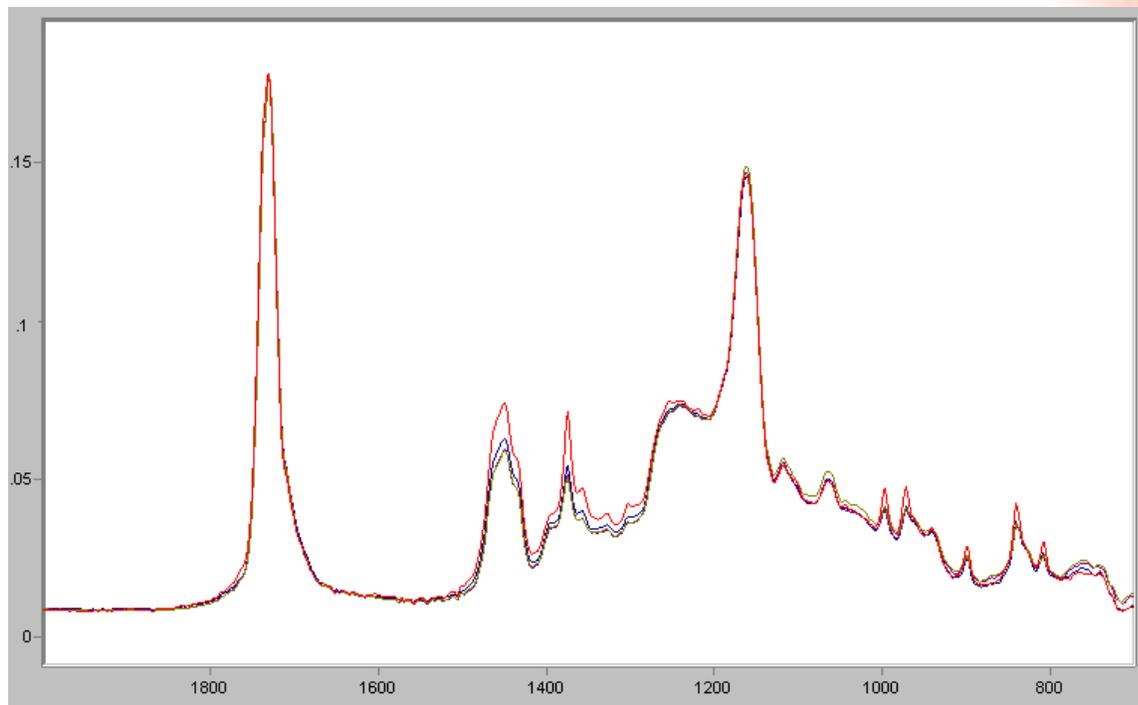


Figure 21: ATR spectra of 4 samples of UV-cured adhesive.

The Degree of Polymer Curing, as Measured by ATR

Spectroscopy

collection time was 30 seconds output.
at 4 cm⁻¹ resolution.

RESULTS AND DISCUSSION

Spectra of four samples are shown in Figure 2; these samples were irradiated under different conditions. The differences are clearly evident in the measured spectra. There are some clear trends in band intensities with irradiation time. As a first approximation, the band intensity at ~808 cm⁻¹ to provide a quantification of the changes observed in the spectra.

The change in the relative intensity of the signal at ~808 cm⁻¹ can be correlated to the known lamp power as seen in Figure 3 and also to the known coating speed as shown in Figure 4.

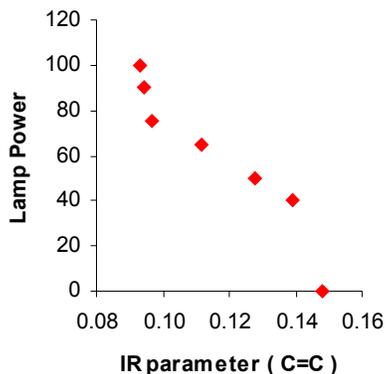


Figure 3: Variation of the relative intensity at 808 cm⁻¹ with lamp power. The lamp power is expressed as a percentage of the total possible

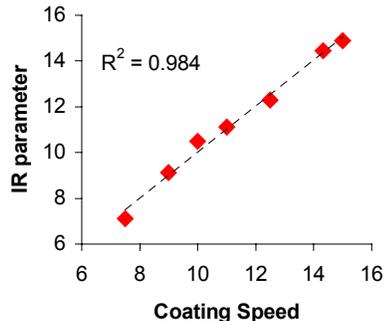


Figure 4: Variation of the relative intensity of the 808 cm⁻¹ signal with coating speed in m/min.

CONCLUSION

In some cases, it might not be possible to generate the data for curves of this type. For example, it might not be possible to produce samples at different coating speeds, radiation intensity, etc. However, it is still possible to implement a QC test based on ATR spectra measured with the Split Pea accessory. A group of samples that have passed all the performance tests can be used as the standard. Spectra of production samples are compared to those of the standard group to determine quickly if the production samples meet specification criteria. There are several chemometric and search-style mathematical tools available for comparing spectra reliably



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