



Analysis of Impurities in Composites Using the IMV-4000

Jasco, Inc.

The IMV-4000 multichannel IR microscope incorporates a 16-pixel linear array MCT detector. The combination of a linear array MCT detector, high speed scanning FTIR, high precision auto-stage, and fast parallel data processing circuitry allows up to 9600 data points to be mapped in 1 min. Compared to conventional mapping with a single-point detector, this system provides a reduction of the mapping time of up to two orders in magnitude. In addition, multiple components can be measured simultaneously in complex heterogeneous mixtures where a single component can be difficult to resolve by means of only imaging or by using single point detectors. In this case, the spectra of all components are measured together and because the search program used to qualitatively analyze the data comprises individual spectra for each component, accurate analysis by separating each component is extremely difficult. Of course, with single point detectors, multiple component samples can be mapped quite adequately by reducing the slit size, but generally, extremely long measurement times are required.

By comparison, because broadband mapping using the IMV-4000 utilizes a relatively small aperture to carry out efficient analysis of the spatial distribution of multiple components, measurement of biological materials, multi-layer films, etc. can be done extremely well. The present example examines microscopic IR imaging of the spatial distribution of small impurity particles in a polymer resin. Utilizing the imaging data, the characteristic spectra from each area were analyzed using the spectral search program, and cellulose, polyester, and organo-silicone were detected. Our present mapping analysis program includes not only a 3-D display of the measured region using the peak heights or areas of key bands but by depicting multiple 3-D graphics from multiple key bands, the visualization of multicomponent mixtures is feasible. Using this feature, color-classified depictions using the relevant key bands were generated, confirming the distribution of the three components.



Figure 1: Impurity in resin.

Experimental

Impurities in the resin (Figure 1) were extracted and a KBr plate was made by mixing KBr powder and the impurities. The resulting KBr pellet was measured using the transmission mode, the impurities identified by the obtained spectra.

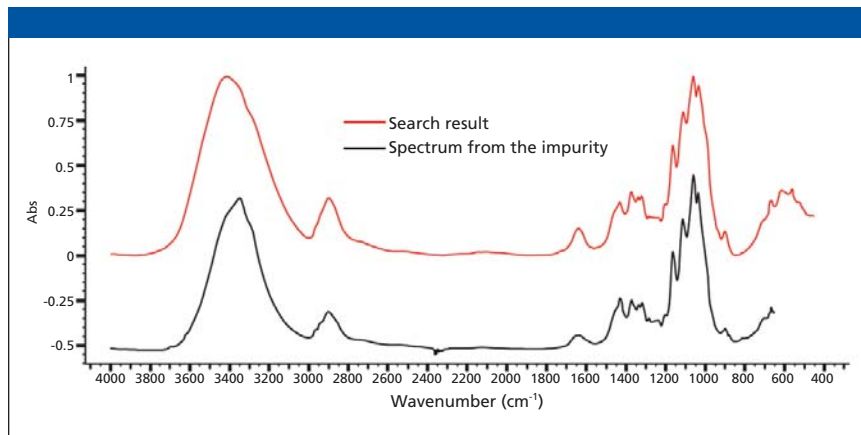


Figure 2: Search result (cellulose).

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Conditions

Detector: Linear array
MCT (16 element)
Resolution: 8 cm^{-1}
Scans: 50
Measurement method:
transmission
Cassegrain: 16 X
Measured area: 600×600
mm (48×48 points)
Measurement time: 15 min

Results and Discussion

The results of the particle mapping were analyzed using a spectral search program and three different spectra identified, confirming that the impurity is a composite mixture of the three contaminants.

Furthermore, from the spectra provided here, a color-keyed distribution image was generated using key peaks from the composite spectra.

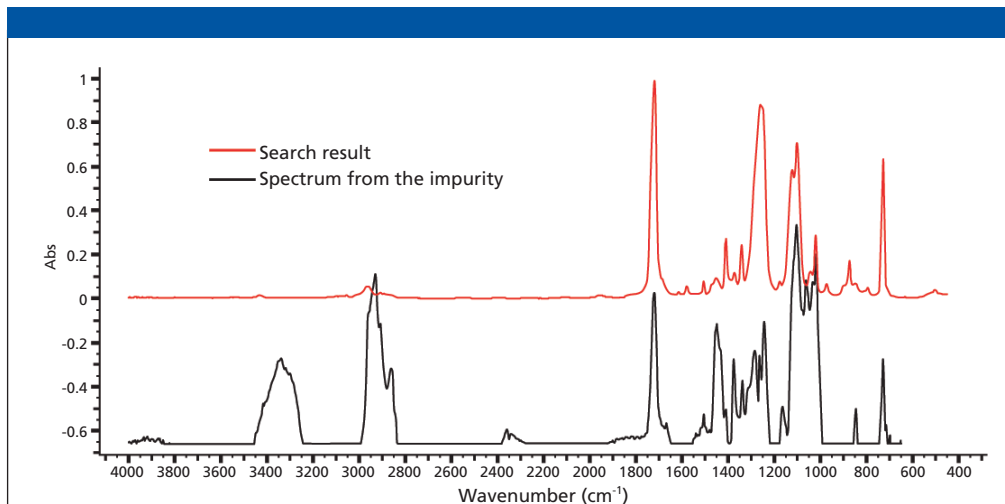


Figure 3: Search result (polyester).

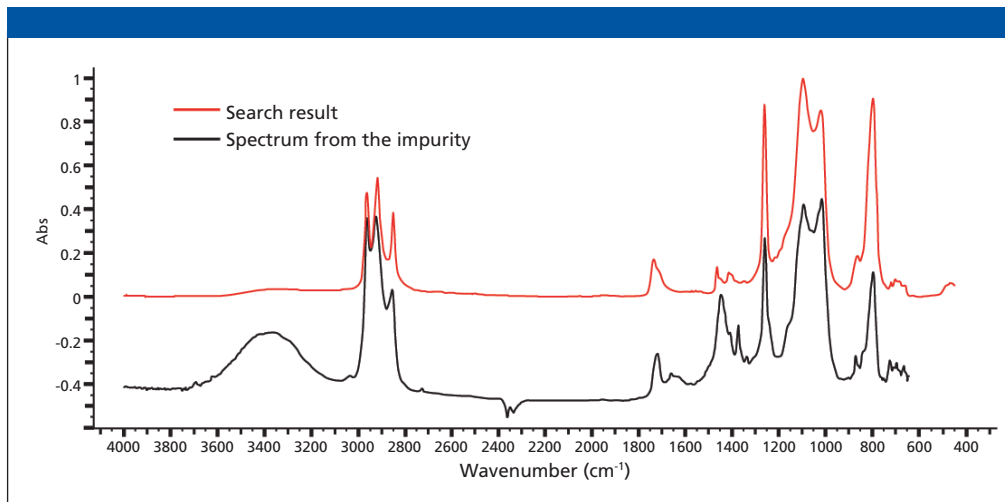


Figure 4: Search result (organo-silicone).

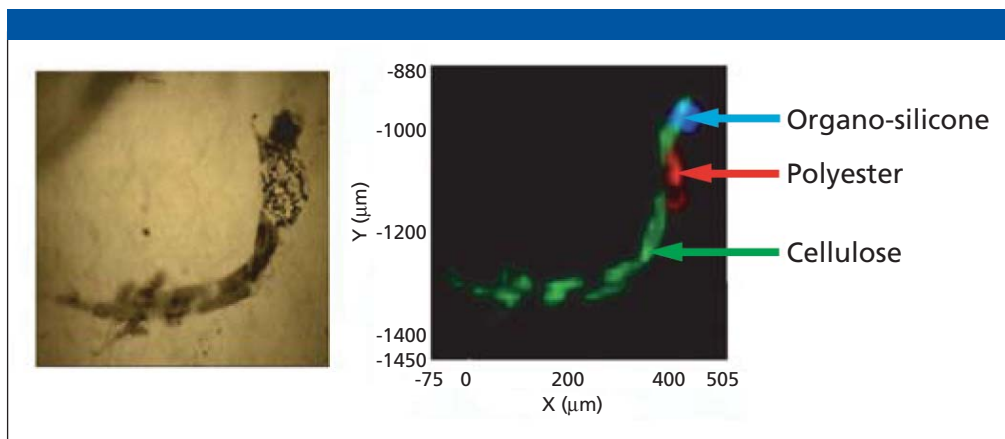


Figure 5: Impurity distribution and impurity image: green (cellulose at 3344 cm^{-1}), red (polyester at 1261 cm^{-1}), blue (organo-silicone at 1800 cm^{-1}).