Investigating the Drying Process of Pressure-Sensitive Adhesives

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The drying of a pressure-sensitive polymer adhesive was monitored by attenuated total internal reflection (ATR) IR spectroscopy. Correlational analysis revealed changes in the composition of the film surface with time.

On a fundamental level, adhesion is the interaction between the surface of one material and that of another.1 The formulation of new adhesives, the optimization of current compounds, and the troubleshooting of manufacturing processes are greatly assisted by a characterization of the surface structure of the materials. This is particularly critical for pressure-sensitive adhesives2 where no solvent or heat is applied during the bonding process.

Experimental Conditions

A 10-reflection PIKE HATR accessory (Figure 1) was used to analyze adhesive reactions. A butyl-acrylate-based adhesive containing acrylic acid was placed on a Ge ATR crystal. Spectra were collected at 4 cm⁻¹ resolution with 200 co-additions, every hour for 12 hours. The temperature was controlled to 21±0.5 °C, and the humidity set at 45±1%. Data acquisition and analyses were performed using custom lab-written software.

Results

Post-acquisition, spectra were analyzed using a two-dimensional correlation routine.3 Figure 2 shows results of the synchronous correlation maps, where the intensity of the diagonal peaks indicate the degree to which bands changed during the drying process.

The bands corresponding to water (686, 1645 and 3300 cm⁻¹) are all decreasing in intensity, and this is related to a subsequent overall increase in the strength of the polymer modes (1160, 1250, 1450, 1730, 2870, 2935 cm⁻¹). This is indicative of an increase in the polymer density within the penetration depth due to the loss of water. Cross peaks in Figure 2 indicate a synchronous change in intensity between bands; if the bands are both growing or shrinking during the drying process, the peaks are positive (red). If they evolve in opposite directions, they are negative (blue). For example, the negative cross peak at 850 cm⁻¹ indicates that the acrylic acid band is growing while the 1650 cm⁻¹ monomer signal decays. This is evidence that the acrylic acid monomer is diffusing to the surface as the sample is drying, possibly concomitant with polymerization of some of the acrylic acid.

Figure 1. PIKE HATR Accessory

Figure 2. Synchronous correlation spectra of butyl acrylate pressure sensitive adhesive. Red cross peaks are positive; blue peaks are negative.
Conclusions

Studying the changes of adhesive composition during the drying process is made easy with the use of the HATR accessory. When time-resolved spectra are combined with correlation analysis, multiple components may be readily identified and trends in structural evolution observed.

References