APPLICATION NOTE

FTIR Spectroscopy

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Use of FTIR to Characterize and Monitor the Curing of Sealants

Introduction

Sealants have a wide range of commercial and industrial applications often requiring highly specialized formulations for the different environments they may be used in, such as extreme temperature environments.

FTIR (Fourier-Transform Infrared) spectroscopy utilizing the Attenuated Total Reflectance (ATR) sampling technique offers a rapid and simple technique for the chemical characterization of the sealant and the ability to monitor the curing process. Adhesives and sealants have historically been slightly difficult to analyze using ATR since they are difficult to remove from the ATR crystal after they have cured. A novel approach using disposable ATR crystals removes this limitation.

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Experimental

A PerkinElmer Spectrum Two+™ FTIR (shown in Figure 1) equipped with a UATR (Universal Attenuated Total Reflectance) accessory along with a Specac Arrow™ top plate shown as Figure 2 was used to characterize three sealants each with a different chemical base. The Arrow top plate utilizes low cost, disposable silicon ATR crystals giving similar spectral performance to diamond crystals. The three sealants analyzed were a silicone based, an acrylic based, and a polyurethane based.

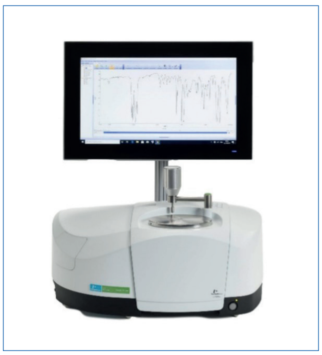

Figure 1: PerkinElmer Spectrum Two+ with UATR Accessory.

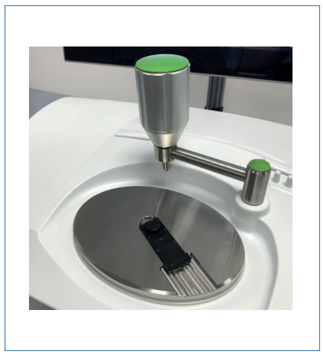

Figure 2: Specac 'Arrow' Top-Plate and crystal insert for PerkinElmer Spectrum Two UATR.

ATR spectra were rapidly scanned for each of the sealants using the scan conditions shown in Table 1, the resultant spectra shown in Figure 3. After each sample had been measured the disposable crystal could be removed and either disposed of or retained for further measurements.

Table 1: Scan conditions for ATR measurements of sealants.

	Value
Spectral Range	4000-600 cm^{-1}
Resolution	4 cm^{-1}
Number of Scans	4

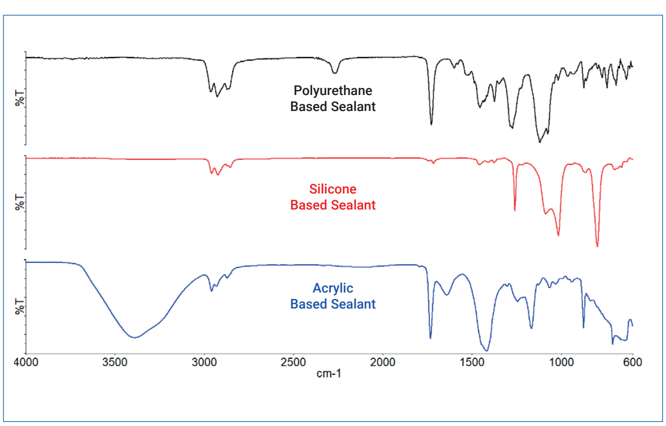

Figure 3: ATR Spectra of the 3 different sealants.

It can be seen that there are significant differences between the spectra of the sealants, with the largest difference being the peak between 3200-3700 cm^{-1} in the spectrum of the acrylic based sealant corresponding to an O-H peak that is absent in the spectra of the other two sealants. Another significant difference is the presence of peaks at approximately 1730 cm^{-1} for the acrylic and polyurethane based sealants, corresponding to a carbonyl peak absent in the silicone spectrum.

The Silicone based sealant has a typical pattern of peaks between 1350-700 cm^{-1}.

One of the most notable peaks in the polyurethane spectrum is the one at 2270 cm^{-1} representative of a -N=C=O bond, Spectra of these 3 materials could be stored and used as representative spectra of the materials for QC testing. Alternatively, if the materials were unknown then they could be identified using spectral library searching against commercial spectral libraries or a company's own library of IR spectra.

Time-based Measurements of Sealants

When a sealant is removed from its container it will come into contact with moisture in the atmosphere and it will start to cure. The chemical changes can be monitored by observing changes in the ATR spectra of the material as the cure proceeds with data collection performed using the Spectrum Timebase software.

Figure 4 shows the initial ATR spectrum of the acrylic based sealant and the ATR Spectrum after 3.5 hours of curing. There are significant changes between the spectra, with the most significant being the decrease of the broad peak at about 3400 cm^{-1} indicative of the loss of water.

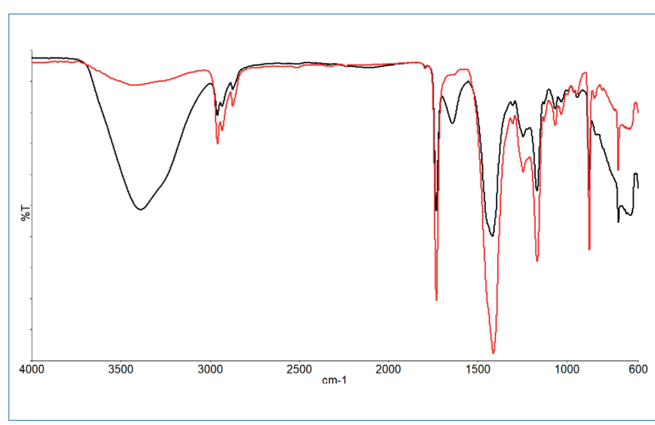

Figure 4: Spectra of the acrylic; black- before curing, red – after curing.

Figure 5 shows the intensity profile of the peak at 3400 cm^{-1} with the intensity decreasing over time. After 6000 seconds the vast majority of the changes had taken place, but even after 12000 seconds there is still evidence that the material has not fully cured.

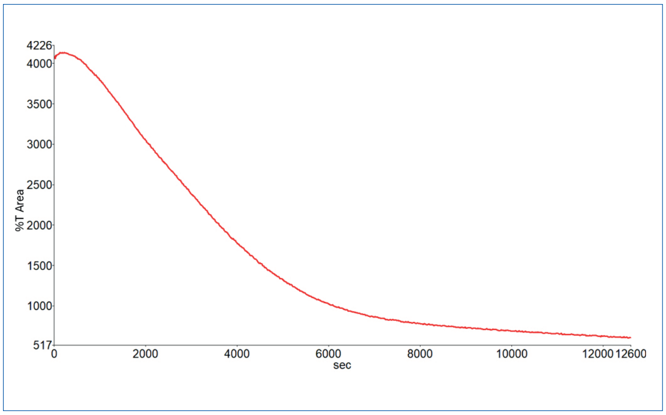

Figure 5: Acrylic sealant - Area profile at 3400 cm^{-1}.

For the polyurethane-based sealant the most notable change is the disappearance of the peak at 2270 cm^{-1}, attributed to the -N=C=O bond, as shown in Figure 6.

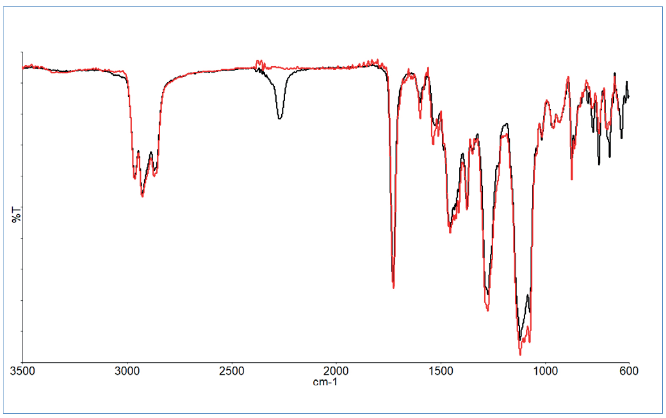

Figure 6: Spectra of the polyurethane sealant; black- before curing, red – after curing.

The intensity profile of the -N=C=O bond at 2270 cm^{-1} is shown in Figure 7, showing no intensity changes beyond approximately 8000 seconds.

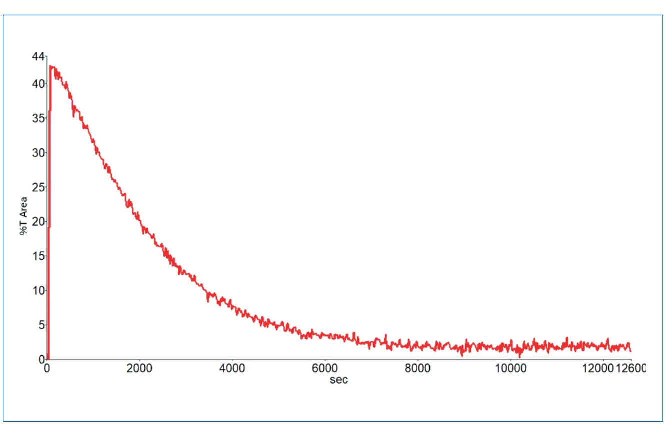

Figure 7: Polyurethane sealant –Area profile at 2270 cm^{-1}.

The silicone sealant shows slight differences between the sample before and after curing as shown in Figure 8. One difference is the carbonyl peak at 1715 cm^{-1}, that is present at the start but almost completely disappeared by the end of the curing period.

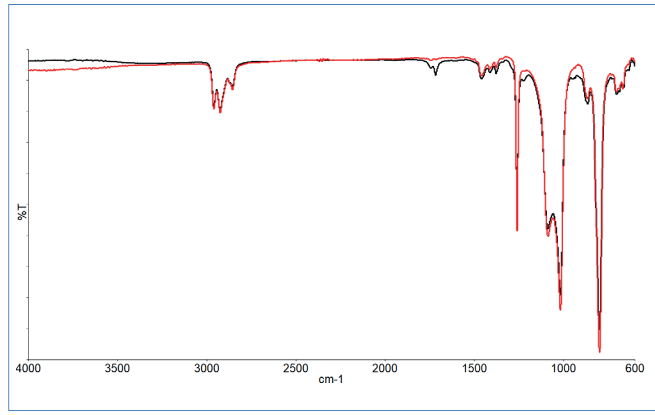

Figure 8: Spectra of the silicone sealant; black- before curing, red – after curing.

Figure 9 shows the intensity profile for this carbonyl peak. It is clear that there are still some small changes occurring even after 12000 seconds.

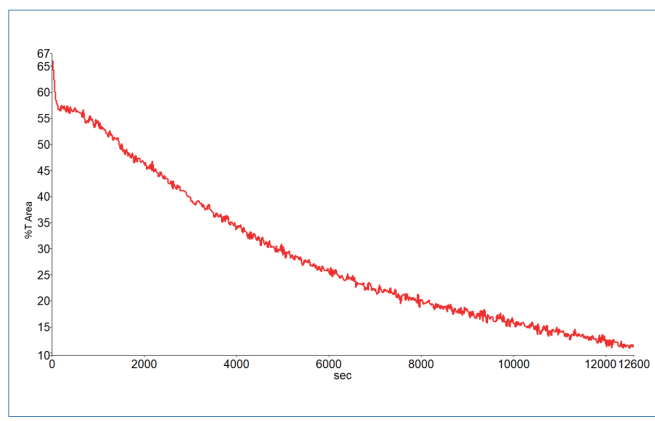

Figure 9: Silicone sealant – Area profile at 1715 cm^{-1}.

Conclusion

The Spectrum Two+ used with the Specac Arrow disposable ATR crystals has been demonstrated to be effective for the characterization of sealant materials. It offers a fast and simple method for the routine measurement of the IR spectra of sealants allowing quality control checks of products and identification of unknown materials. In addition, IR spectroscopy has been shown to be a valuable technique for monitoring the chemical changes that occur during the curing process. Further experiments could be performed to see how the curing rate changes for varying formulations and under different environmental conditions such as various temperatures and humidity levels.

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