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Application News

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Spectrophotometric Analysis

# High-Speed Monitoring of Curing Reaction in UV-Irradiated Resin by Rapid Scan

Some polymeric materials have a molecular structure that is altered due to treatments such as mixing, heating, light irradiation, etc. Some of these changes take place within seconds, while others progress slowly over several hours. A Fourier transform infrared spectrophotometer (FTIR) is an instrument that is extremely effective for tracking chemical reactions and changes because measurements can be conducted very easily and quickly. Here, using the Shimadzu IRTracer-100 FTIR spectrophotometer Rapid Scan feature, we introduce the results of measurement of the curing reaction process that occurs in UV-curable resin.

## Curing Reaction of UV-Irradiated Resin and Rapid Scan

A UV-curable resin such as acrylate can be hardened (cured) relatively quickly through the process of radical polymerization, which is accomplished by irradiating the material with ultraviolet light. Although the curing process proceeds relatively slowly if a typical indoor fluorescent light source is used, a powerful commercial UV lamp provides much stronger ultraviolet irradiation permitting nearly complete curing in a matter of seconds. To keep up with these very fast reactions, it is necessary to use an FTIR equipped with high-speed scanning capability. The rapid scan feature of the IRTracer-100 FTIR permits acquisition of 20 infrared spectra per second, enabling precise observation of the curing reaction process even when it is completed within a few seconds. Fig. 1 shows a photographic image of the Shimadzu IR Tracer-100 FTIR spectrometer with the Rapid Scan feature.

Fig. 1 Shimadzu IRTracer-100 FTIR Spectrophotometer

## Measurement

Using the specular reflectance method, we conducted measurement of the infrared spectra generated during the curing process of commercially available acrylate-based UV-curable resin which was coated on a metal plate in a thin layer. Although measurement can also be conducted by the transmittance method, in which a thin layer of sample is coated on a potassium bromide (KBr) window plate for transmission of infrared light, this requires use of a new window plate after each measurement.

After conducting background measurement using the metal plate as the reference, a layer of sample that was thin enough to avoid peak saturation was coated on the metal plate. Measurement was started in the Rapid Scan Measurement mode at an acquisition rate of 20 spectra per second, and after 5 seconds, ultraviolet irradiation was begun. Fig. 2 shows a photograph of UV-irradiation for sample.

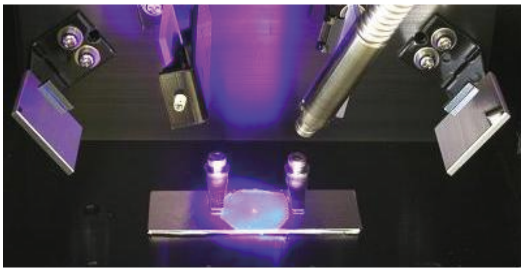

Fig. 2 UV-Irradiation for Sample

The FTIR measurement conditions and the UV irradiation conditions are shown in Table 1. In addition, a 1-second excerpt of the infrared spectrum measurement results is shown as a 3D graph in Fig. 3.

Table 1 FTIR Measurement Conditions and UV Irradiation Conditions

| | |
|---|---|
| Instrument | : IRTracer-100 |
| Resolution | : 16 $cm^{-1}$ |
| Accumulation | : 1 |
| Apodization | : Happ-Genzel |
| Detector | : MCT |
| UV Lamp | : HAMAMATSU L9588 |
| | Power < 45 mW |

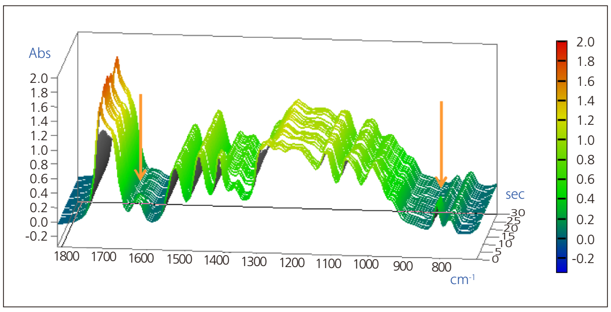


Fig. 3 3D Spectra of Rapid Scan Measurement Generated in Curing Reaction in UV-Irradiated Resin

Also, the expanded spectra of the peaks near 1635 cm$^{-1}$ and 810 cm$^{-1}$ acquired over approximately 2 seconds directly following UV irradiation are shown in Figs. 4 and 5. It is believed that the peaks at 1635 cm$^{-1}$ and 810 cm$^{-1}$ are associated with the C=C stretching vibration of the vinyl group and the CH out-of-plane bending vibration of the vinyl group, respectively. It is evident that an abrupt decrease in the vinyl group occurred following UV irradiation. Fig. 6 shows radical polymerization reaction of acrylate resin.

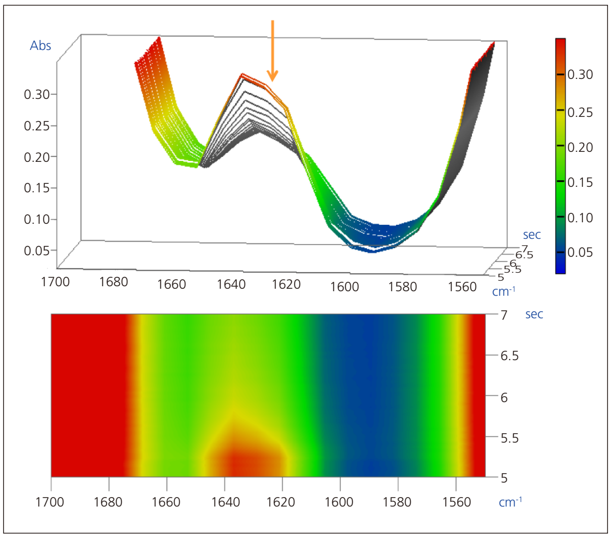


**Fig. 4 Expanded Spectra of Peak at 1635 cm$^{-1}$, Upper:3D, Lower:2D**

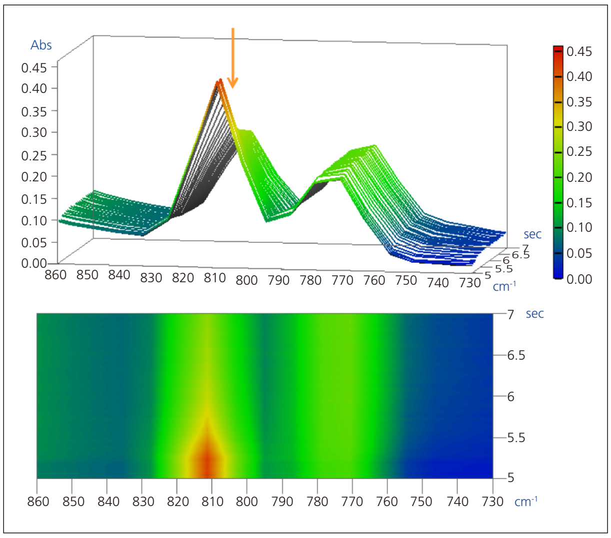


**Fig. 5 Expanded Spectra of Peak at 810 cm$^{-1}$, Upper:3D, Lower:2D**

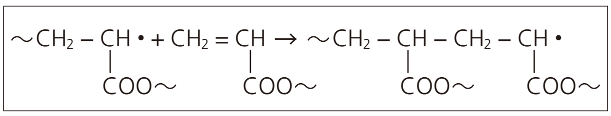

**Fig. 6 Radical Polymerization Reaction of Acrylate Resin**

## Peak Time-Course Graph and Reaction Rate Calculation

Fig. 7 shows the peak area time course graphs of the peaks at 1635 cm$^{-1}$ and 810 cm$^{-1}$. During the few seconds directly following UV irradiation, a dramatic reduction in the sizes of these peaks is clearly noticeable. Thus, specifying the wavenumbers of peaks of interest beforehand in this type of time-course graph permits observation of the real-time changes that occur in multiple peaks.

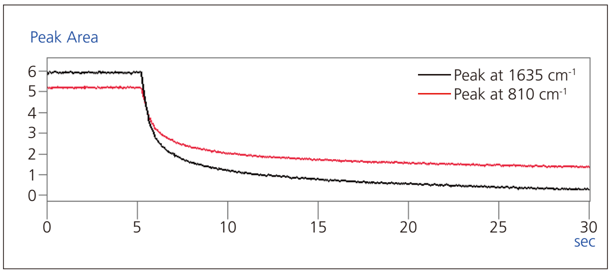


**Fig. 7 Peak Area Time-Course Graph of Peaks at 1635 cm$^{-1}$ and 810 cm$^{-1}$**

Using the peak area value of the peak at 1635 cm$^{-1}$, Fig. 8 shows the reaction rate time-course graph assuming a reaction rate of 0 % prior to UV irradiation, and a 100 % reaction rate at the point the peak disappears, or in this case, at the completion of the curing reaction. The graph clearly indicates that the reaction was more than 50 % completed in less than one second, and reached 80 % completion in about 5 seconds, after which the reaction progressed slowly. This type of reaction rate calculation is one example of the type of the analytical functions included in the Rapid Scan measurement software.

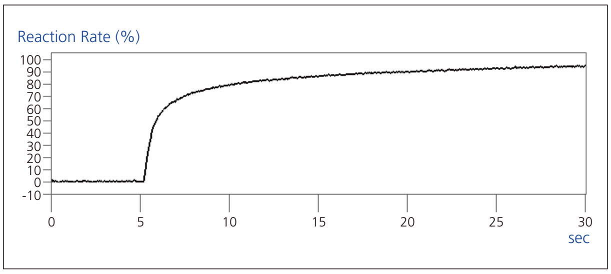


**Fig. 8 Time-Course Graph of Reaction Rate**

## Conclusion

Here we introduced the results of investigation of the reaction process in UV-curable resin in which the process was tracked in real time. The FTIR-Rapid Scan effectively tracks chemical reactions and changes that progress very quickly, permitting close observation of the reaction process.

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