Real-time analysis of conversion degree for light curable varnishes

Dr. S. Shilov, Bruker Optics

Technology

Introduction

- Photo-curable polymers are widely used in automotive, consumer electronics, printing, and coating industry because of their multifunctional properties.
- Four steps are characteristic for the basic kinetics of radical polymerization.

1. The reaction is initiated by energy induced radical formation (e.g. via UV-irradiation
2. The radicals react with monomers and form monomer-radicals.
3. After the reaction has been initiated, the propagation step takes place: monomer-radicals react with monomers and the polymer chain grows.
4. The last step is termination, where different reactions can occur (e.g. combination of two active chain ends).

The most important characteristics of curable polymers are the speed of cure and the degree of conversion in the final product. FTIR spectroscopy is an excellent analytical tool to measure these parameters.

Photo-curable polymers

- The properties of light cured polymers depend not only on their composition but also on their reaction kinetics.
- The most important characteristics of curable polymers are the:

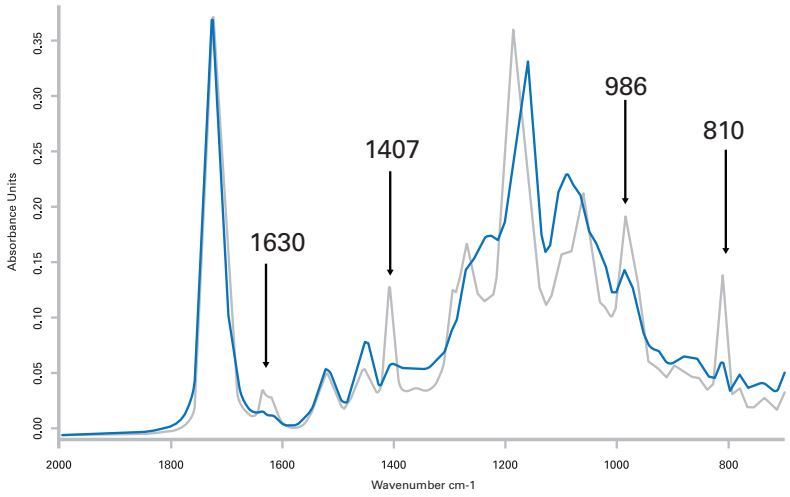
speed of cure

degree of conversion in the final product

Methods

A temperature-controlled diamond ATR accessory mounted on Bruker Vertex 80 FTIR was used in this study

- The degree of conversion and the speed of cure can be measured within a few seconds using fast scanning spectrometer from Bruker.
- Attenuated Total Reflectance (ATR) is the easiest and most common technique for collection



IR Spectrum

This chart indicates that the reactant functional groups are almost completely absent after the polymerization reaction.

Conversion Kinetics

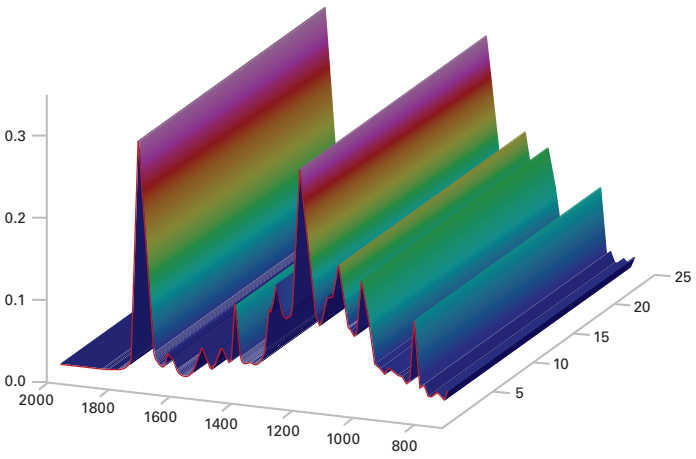
- Start photoreaction by exposing sample to the light

Acquire spectra at 8 cm^{-1} spectral resolution at the highest rate: 87 spectra/sec (Bruker Vertex 80/80V FTIR), 50 spectra/sec (Bruker INVENIO FTIR).

Measurement

- Temporal progression of absorption band intensities is displayed in the 3D view.
- IR band intensity is proportional to the concentration of the functional group assigned to this band.
- Conversion kinetics is calculated from the band intensities vs time under lights exposure.
- A simple two-point calibration model can be constructed based on the intensity of these bands in the spectra of uncured and completely cured acrylate.
- This simple model allows calculating the conversion degree for the unknown acrylate sample.

Experimental setup for measuring the polymer curing rate. A diamond ATR accessory is mounted in the sample compartment of FTIR spectrometer. The sample is irradiated by a UV light via fiber optics.



3D display of the time-resolved spectra of an acrylic polymer during cure under UV light.

Conclusion

FTIR Spectroscopy is an ideal analytical tool that allows the determination of curing times of curable varnishes to vary the degree-of-curing in the final product.

www.bruker.com/optics

Bruker Scientific LLC
Billerica, MA · USA
Phone +1 (978) 439-9899
info.bopt.us@bruker.com

Bruker Optics GmbH & Co. KG
Ettlingen · Germany
Phone +49 (7243) 504-2000
info.bopt.de@bruker.com

Bruker Shanghai Ltd.
Shanghai · China
Phone +86 21 51720-800
info.bopt.cn@bruker.com