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Analyzing Chemical Composition of Rigid Polyurethane (PU) Foams using FTIR Microscopy and DRIFTS-IR

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John Kinyanjui, Ph.D., Jeff Head, Mark Talbott, Ph.D.;
Shimadzu Scientific Instruments, Columbia, MD, USA

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Introduction

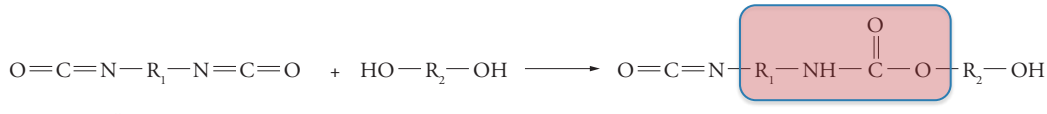
- The chemical composition of rigid polyurethane foams was studied using FTIR microscopy and diffuse reflectance Fourier transform spectroscopy (DRIFTS) FTIR.
- Temperature gradients during the processing of rigid polyurethane (PU) foams leads to chemical gradients that might be observed across the radial and axial dimensions of the foam.
- Unreacted reactants and products from side reactions can remain trapped in the cavities of the foam hampering mechanical strength.
- A highly **complex and heterogeneous** polymeric composite results.
- The chemical composition of the cellular structure of the foam was studied with distinct spectra of the gas filled cell nucleus and cell wall.
- FTIR spectroscopy offers an ideal tool for studying variations in chemical composition at both bulk and microscopic levels.

Experimental Conditions - Reactants

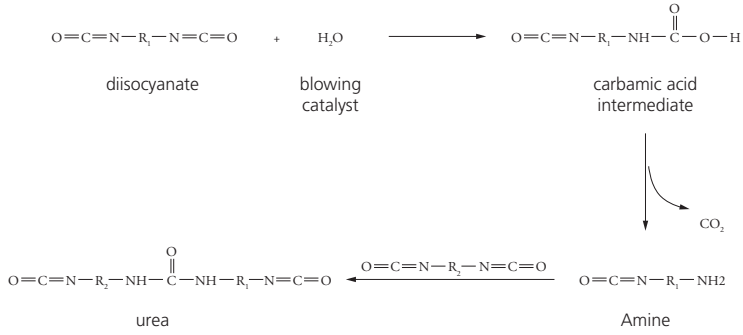
- Commercially available rigid polyurethane pour foam starting materials (designated as Part A and PART B by manufacturer) were used.
- Although the exact formulation was not provided
  - PART A normally contains the diisocyanate.
  - Part B normally contains the polyol, water (blowing agent), a catalyst and a surfactant premixed at optimized ratios.
- Equal amounts of PART A and PART B were mixed and stirred until the mixture appeared uniform.
- The foam was then allowed to rise and "cure" over a period of five (5) days.
- Foam was sectioned and sample measurement carried out on the fifth day.
- Based on FTIR results unique **uretoneimine** crosslinking functionality might be present in the foam giving clues on exact formulation of starting materials. [1,2]

Rigid PU Foam Formation Reaction

General Reaction [1,2]



Foam Blowing Reaction [1,2]



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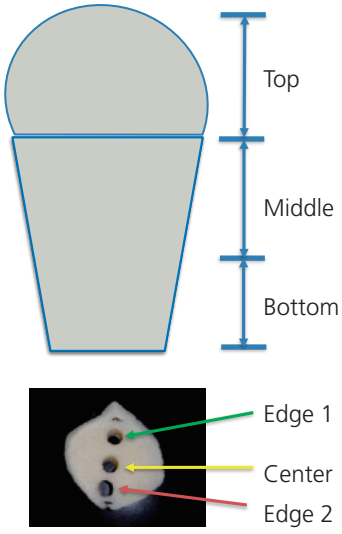
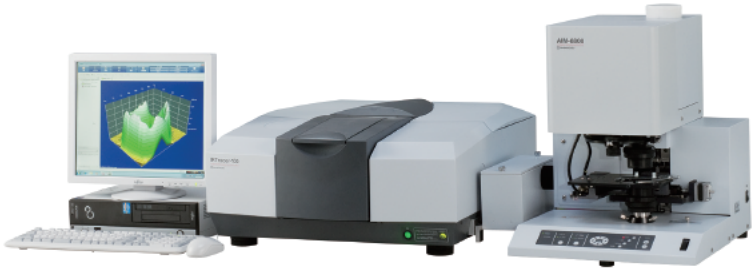
Experimental Conditions - Foam Mold Designation

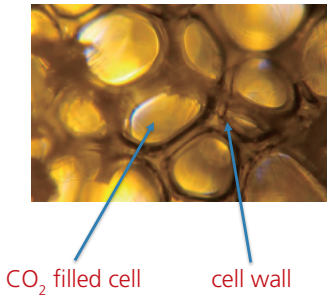


- Three sections in the mold were cut and designated as “top”, “middle” and “bottom” for sampling.
- Samples were bored out from the radial center of each area.
- For the middle section, three samples were bored out and designated as “center”, “Edge 1” and “Edge 2”.

Instrumentation



The AIM-8800 connected to IRTracer-100



Light Microscope Image of PU Foam

Sample Preparation

- Sample bored out from the center of the middle section of the mold was tested. A cylinder microtome was used to produce thin specimens for microscopy.

The PIKE EasiDiff DRIFTS Accessory

Sample Preparation

- Pristine samples were bored out and sliced to fit tightly into the 10 mm diameter sample cup provided with the accessory.

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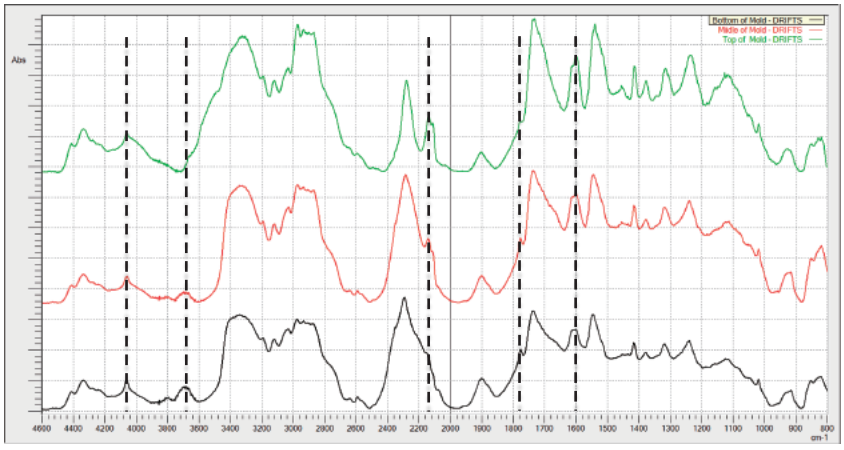
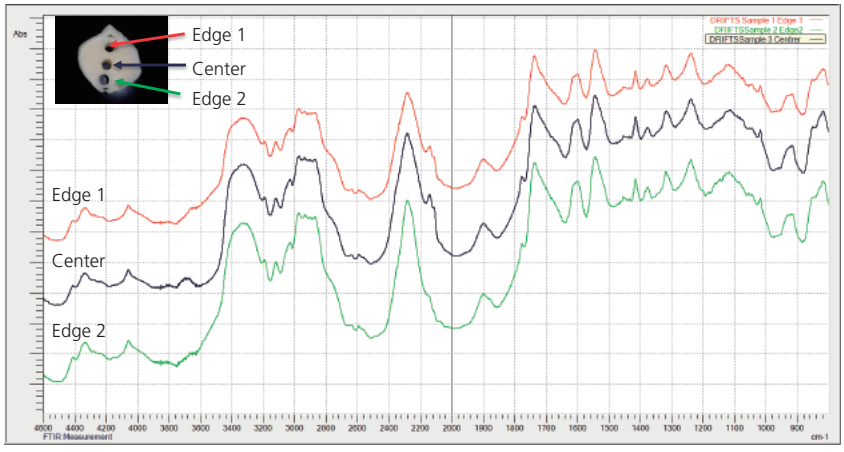
DRIFTS-IR: Top to Bottom of Mold



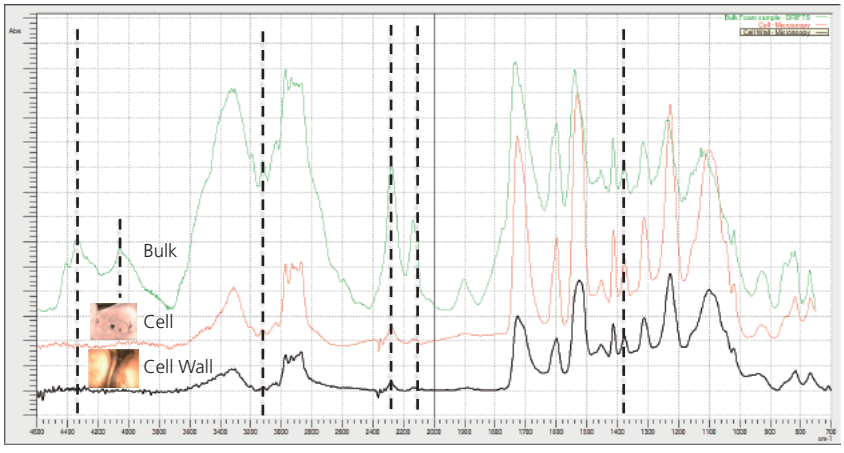
Composition of the mold changes as you move from top to bottom of the mold.

DRIFTS IR: Radial Profile of Middle Section of Mold



Composition across the radial axis of the middle section does not change appreciably.

FTIR Microscopy: Cell and Cell Wall Spectra



Composition of cell and cell wall different from the bulk foam due to trapped reactants and side reaction products (such as urea) in foam micro-pores.

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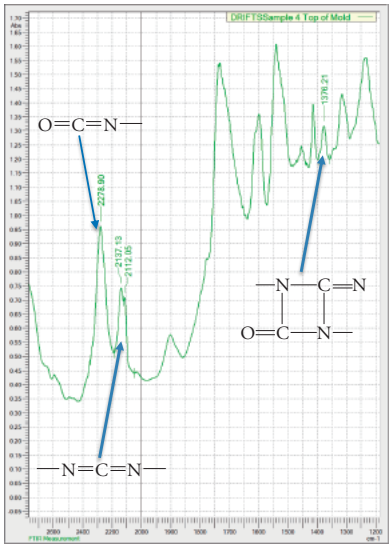
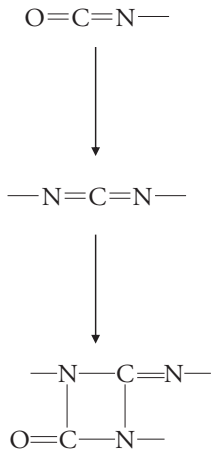
Cross-Linking in PU Foam[1,2]



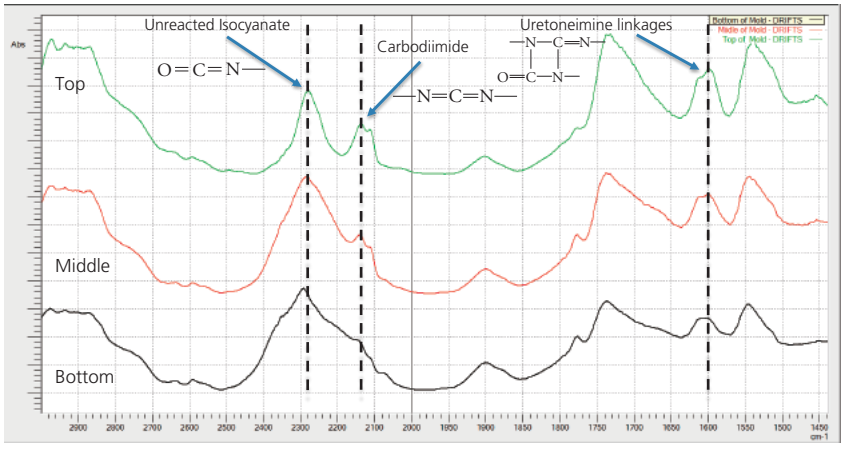
Unreacted Isocyanate reacts by itself to form *carbodiimide*

Carbodiimide reacts with isocyanate to form *uretoneimine* crosslinks

Uretoneimine linkages lead to increased rigidity



DRIFTS-IR: Top to Bottom of Mold



Composition of the mold changes as you move from top to bottom of the mold.

Conclusions

- FTIR microscopy was successfully used to show distinct chemical composition at the cellular level as compared to the bulk composition.
- With DRIFTS-IR the spectra of pristine foam samples can be easily collected with minimal sample preparation to provide the bulk chemical properties of the foam.
- Foam aging and curing including the level of cross-linking can also be monitored using DRIFTS-IR.
- The mechanical properties of the PU foam is highly dependent on the chemical properties of the foam.[1,2] FTIR data can be used as a tool to assist in tuning the mechanical properties of the foam.
- Previous work has shown that the level of cross-linking in PU foam can be directly correlated to the Young's modulus of the foam.[1,2]

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References

1. Mohan, R.B.; O'Toole B.J.; Malpica, J.; Hatchett D.W.; Kodippili G.; and Kinyanjui J.M. **Effects of Processing Temperature on ReCrete Polyurethane Foam.** Journal of Cellular Plastics 2008; 44: 327-345.
2. Hatchett, D.W.; Kodippili, G; Kinyanjui, J.M.; Benincasa, F.; Sapochak, L. **FTIR Analysis of Thermally Processed PU Foam.** Polymer Degradation and Stability; 2005; 87(3), 555-561.

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