Characterization of Foams and Impurities with Gel Permeation and Liquid Chromatography / Mass Spectrometry

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Sandia National Laboratories, Albuquerque, Materials Characterization Dept. 1819, Unlimited, Unclassified Release.

Overview

Purpose:

- Detect impurities in precursors and synthetic products
- Provide information to improve synthetic chemistry methods and product quality control
- Characterize impurities and aging products in detail, to enable better understanding of aging mechanisms

Methods:

- Liquid chromatography (LC) mass spectrometry (MS) using high mass accuracy for molecular formula
- DART-MS^N direct analysis of foam materials and precursors to determine or verify molecular structure
- Gel-permeation chromatography (GPC) with UV detection

Results:

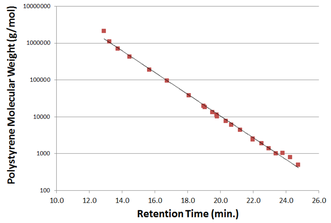
- MS and GPC provide complementary information regarding impurities and unwanted polymerization products
- Results provide insight into thermal aging of the final foam product

Introduction

Measurement of impurities and process by-products of foams and foam precursors can be achieved through GPC and MS methods. This information is critical in the study of foam aging and quality control factors. Trace level species and unknowns cannot be identified by GPC. Alternatively, mass distribution can be difficult with mass spectrometry due to varying ionization efficiencies and sample prep of very large foam polymers. Yet MS can routinely detect and identify chemical species with excellent sensitivity. In this way GPC and MS can provide complimentary information regarding a final foam composition. We attempt to make correlations in the data from these tools on important issues related to the production and aging of foams used in the stockpile.

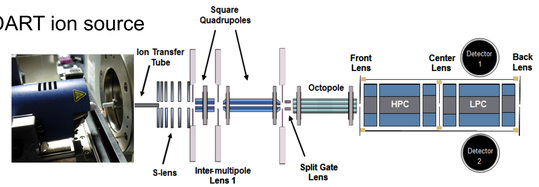
Methods

GPC provides general molecular weight (m.w.) information – retention time correlates well with m.w.



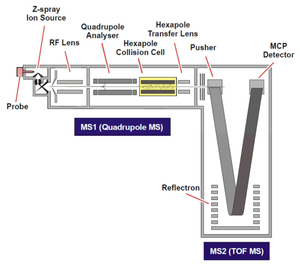
Ambient ionization (DART) allows analysis of polymer precursors and products rapidly. Ion trap allows for molecular identification.

DART ion source



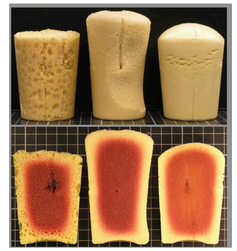
MS^N with Ion Trap Mass Spectrometer.

UPLC with QTOF-MS provides chromatographic separation, but requires method development

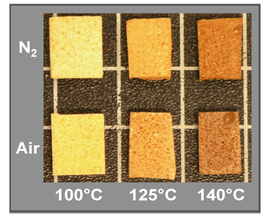


Results

Foams show discoloration associated with increased internal temperature during curing.

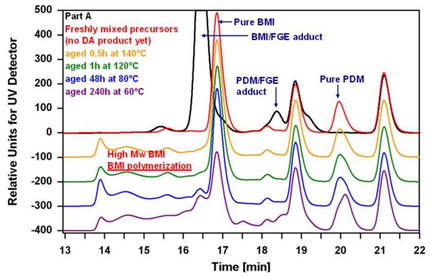


Uniform changes in color observed with aging at elevated temperatures.

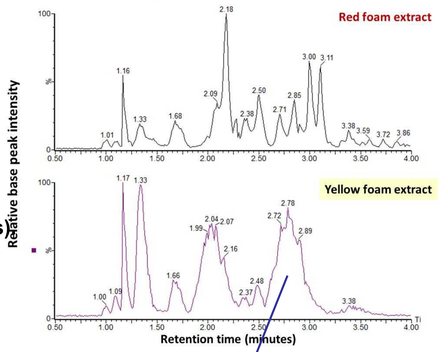


These color changes thus suggest temperature dependent chemical changes during curing.

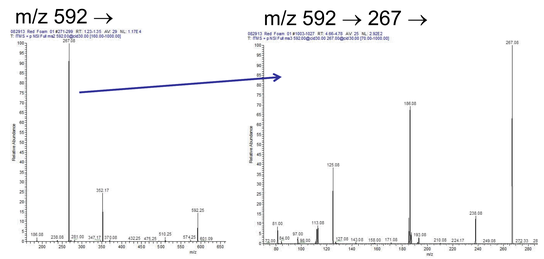
GPC analysis of mixtures of foam precursors shows undesired polymerization products are generated even at temperatures as low as 60°C



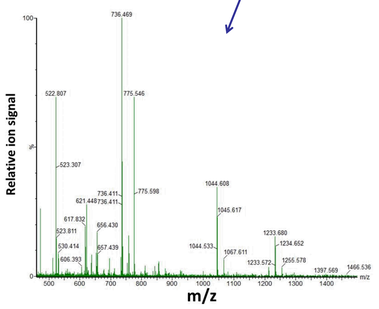
LC/MS analysis of red/yellow foam extracts show a variety of peaks of m.w. higher than precursors and expected product(s).



Among the differences observed – a m/z 592 with 8x higher intensity in red versus yellow foam. DART/MS with MS^n is underway to determine the chemical identity. This m/z is not a precursor or known reactant.



Yellow foam has higher quantities of some species larger than precursors.



Conclusions

Foam precursors and products can be observed using mass spectrometry. Additional data analysis is required to fully understand the temperature-related byproducts in this system. GPC and MS techniques can provide complimentary information regarding impurities, products, and by-products formed during foam-generating reactions or due to aging. GPC can provide information on higher m.w. species whereas MS can provide information and identification of more trace level components. DART-MS can be useful because it does not require sample prep, and allows (in our lab) for quick MS^n experiments. UPLC/MS provides a wealth of data but may require significant method development.

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