

GPC2015



- ● ● Polymer and Polyolefin
Characterization
- Conference



September 1 and 2, 2015
Fairmont Washington, D.C.,
Georgetown

www.GPCconference.com



Congratulations to Dr. Hongkun He!

Dr. He is the winner of the student submission award for his abstract entitled *"Molecular Weight Characterization of Poly(ionic liquid)s by Gel Permeation Chromatography using Salt-Containing Eluents: A Facile "Vaccination" Approach"*.

Dr. He is currently working as a postdoctoral associate under the guidance of Prof. Paula T. Hammond in the Department of Chemical Engineering at the Massachusetts Institute of Technology. He received his Ph.D. from Carnegie Mellon University in 2015 under the supervision of Prof. Krzysztof Matyjaszewski, where he worked on the development of functional porous polymers and poly(ionic liquid)s. He received his B.S. in Chemistry in 2008 and M.S. in Polymer Chemistry and Physics in 2010 from Shanghai Jiao Tong University, China.

Dr. He's abstract can be found on page 12 of this program. His award will be presented to him, followed by his talk, on Tuesday, September 1 at 2:10pm.

Welcome to GPC2015: Polymer and Polyolefin Characterization Conference!

September 1, 2015

Dear Colleagues,

Professor Jimmy W. Mays and I welcome you to Washington D.C for the inaugural GPC2015 conference. This two-day conference focuses on Gel Permeation Chromatography (GPC) as a powerful analytical tool for characterization of macromolecules and its various applications in the understanding of natural and synthetic polymers.

The program offers a balance of invited and contributed lectures, poster sessions, discussions and information exchange, bringing together analytical chemists, polymer chemists, chemical engineers and material scientists from a variety of sectors that are involved in using and developing methods for polymer characterization that utilize GPC.

We encourage the exchange of ideas and thoughts among the attendees and hope that you take advantage of this opportunity to renew and establish new friendships with colleagues in the GPC industry.

On behalf of the scientific committee and the organizer, we welcome you to this exciting event!



Dr. Jimmy W. Mays
Distinguished Professor
The University of Tennessee



Mr. Oscar Yamasaki
President
Tosoh Bioscience LLC

Scientific Committee

Committee Chair

Jimmy W. Mays, Ph.D.

Distinguished Professor, The University of Tennessee, Knoxville, TN

Professor Jimmy Mays received a B.S. degree in Polymer Science from the University of Southern Mississippi in 1979 and a Ph.D. in Polymer Science from the University of Akron in 1984 under the direction of Professor Lewis Fetters. After graduation he worked for several years at Hercules Research Center, prior to joining the faculty at the University of Alabama at Birmingham. In 2002 he moved to his current position as UT/ORNL Distinguished Scientist at the University of Tennessee, Knoxville, and Oak Ridge National Laboratory.

Professor Mays' research is centered on polymer synthesis and polymer characterization and solution properties. He is a Fellow of the American Chemical Society, Fellow of the Royal Society of Chemistry, and Fellow of the American Association for the Advancement of Science.

Committee Members

Erik Berda, Ph.D.

Associate Professor, University of New Hampshire, Durham, NH

Professor Erik Berda is currently an Assistant Professor of Chemistry and Materials Science at the University of New Hampshire. His research group studies the synthesis and characterization of biomimetic polymers, polymer folding, and responsive materials.

Before beginning his independent academic career, Erik was a postdoctoral fellow in Eindhoven, the Netherlands with Prof. Bert Meijer. Prior to Eindhoven he received his Ph.D. from the University of Florida under Prof. Ken Wagener (2008) and a B.S. in Chemistry (2003) from Penn State working with Prof. Harry Allcock.

Paul DesLauriers, Ph.D.

Research Fellow and Manager of the Polymer Science Group, Chevron-Phillips Chemical Company, Bartlesville, OK

Paul J. DesLauriers is currently a Research Fellow and Manager of the Polymer Science Group for Chevron-Phillips Chemical Company at the Bartlesville Research and Technology Center, Bartlesville, OK.

Before joining Phillips/CPC in 1989, he worked as a senior research chemist for Pennzoil Petroleum Company (the Woodlands, TX). Paul received his B.S. degree in Chemistry (1980) from the University of West Florida and a Ph.D. degree (1985) from the University of Southern Mississippi in physical organic chemistry.

Dr. DesLauriers has authored or coauthored over 30 patents and 54 publications/ presentations. His accomplishments include inventing the technology used for the clear mineral gel product line, Versagel™ currently marketed worldwide by Penreco, and developing the SEC-FTIR methodology for measuring short chain branching in polyolefins (now sold by Agilent Technologies). He has also held adjunct professorships at the University of Tulsa, Rogers State University, and Oklahoma Wesleyan University.

Wei Gao, Ph.D.

Research Scientist, Dow Chemical Company, Collegeville, PA

Dr. Wei Gao received her B.S. from Fudan University, and Ph.D. from Beijing University in Polymer Chemistry and Physics in 1997. Wei had her post-doc, and then worked as a group leader at the Chemistry Institute, Chinese Academy of Sciences till 1999.

Before she joined Dow through heritage Rohm and Haas in 2006, Wei was a research assistant professor at "Brooklyn Poly", currently known as Polytechnic Institute of NYU. Wei has 35 peer reviewed articles, over 10 patents, and has given many conference presentations.

Wei has received US EPA Presidential Green Chemistry Challenge Awards twice (2003 contribution to "New Options for Polymerization using Mild and Selective Lipases", 2013 contribution to Dow "Evoque Pre-composite Polymer Technology").

Her current research activities related to macromolecular and particle characterization for industrial problem solving, product development and innovation.

David Gillespie

Senior Research Chemist, Performance Plastics Characterization, Dow Chemical Company, Freeport, TX

David Gillespie has been working in the area of GPC since 1986 and has a background in Chemical Engineering from Drexel University (Philadelphia, PA). Gillespie's initial industrial focus was on carbohydrate analysis (working with the USDA-ARS-ERRC) where he specialized in viscometry, multi-angle light scattering, and chromatographic deconvolution methods in both analytical and semi-preparative-scale GPC systems.

Gillespie joined Viscotek in 1990 and served as its Technical Services Manager, while also headed their Applications Laboratory, and Instrument Repair Services. Gillespie joined Chevron Chemical Corporation in 1997, where he focused on microstructure characterization of polyolefins, mainly involving the simultaneous interpretation of ATREF and GPC techniques to ascertain a more robust picture of these semi-crystalline polymers. Gillespie has been with the Dow Chemical Company since 2000, where he currently is in Performance Plastics Characterization, focused mainly on polyolefins.

Besides his direct focus on interpreting GPC data, he is involved in chromatography simulations, deconvolution, and structure-property correlations. Additionally, Gillespie was involved in developing 2D crossfractionation (ATREF-GPC) technology for polyolefin technology and he works with semi-preparative and preparative scale systems involving molecular weight and crystallinity separations of polyolefins.

He has been awarded several patents on polymer resin compositions and polymer analysis technology. Above all, he appreciates others' time in lively discussions on solution properties vs. melt properties and how to extend the bounds of current polymer characterization technology. David Gillespie looks forward to meeting you and discussing how we together can continue to evolve, incorporate, and optimize GPC technology.

André Striegel, Ph.D.

Research Chemist, Institute of Standards & Technology (NIST), Chemical Sciences Division of the Material Measurement Laboratory, Gaithersburg, MD

André Striegel received his Ph.D. in Analytical Chemistry in 1996 and his B.S. in Chemistry in 1991, both from the University of New Orleans. From 1996 to 1998 he performed postdoctoral research for the U.S. Department of Agriculture, at the National Center for Agricultural Utilization Research. For the next six years he worked for Solutia (now Eastman Chemical), at their Springfield, Massachusetts R&D center, first in the Physical & Analytical Sciences Center and then in Films R&D, achieving the rank of Research Specialist.

From 2004 to 2011 he was Assistant Professor of both Analytical and Materials Chemistry in the Department of Chemistry & Biochemistry at Florida State University (FSU). In September 2011 he joined the National Institute of Standards & Technology (NIST), where he is a Research Chemist in the Chemical Sciences Division of the Material Measurement Laboratory. His research interests are in the area of polymer characterization, in particular applying separation science to determining structure-property relations of complex macromolecules, and in the fundamental aspects of separation and detection methods.

He has received the Eli Lilly Analytical Chemistry Grantee Award, the inaugural ACS-DAC Award for Young Investigators in Separation Science, and an FSU First Year Assistant Professor Award. He also received a Solutia Technical Achievement Award for his research in private industry, and served as inaugural Professor in Residence for Preservation Research and Testing at the U.S. Library of Congress. He is the author of over 60 peer-reviewed publications, lead co-author of the second edition of "Modern Size-Exclusion Liquid Chromatography" and editor of the book "Multiple Detection in Size-Exclusion Chromatography" and was, until recently, associate editor of the Encyclopedia of Analytical Chemistry, and is a member of the editorial board of various polymer science and analytical chemistry journals.

Conference Agenda

Tuesday, September 1, 2015

- 7:00-9:00** **Registration/Breakfast Open**
- 9:00-9:15** **Opening Remarks**
- 9:15-10:15** **Key Note Lecture: *Polymer Synthesis At and Beyond the Limits of Characterization***
Jimmy Mays, University of Tennessee, Knoxville
- 10:15-10:30** **Coffee Break**
- 10:30-12:20** **Session I**
- 10:30-11:10 *Characterization of Aqueous Phase Oligomers Produced in an Acrylic Emulsion Polymerization Process by GPC-RI/MS*
Wei Gao, Dow Chemical Company
- 11:10-11:40 *Improved Performance of Resins Modified by Reactive Nanogel Additives*
Matthew Barros, University of Colorado at Denver
- 11:40-12:20 *How to Use GPC-IR to Identify the Minimum Number of Active Site Types on Ziegler-Natta and Phillips Catalysts Used to Make HPDE and LLDPE*
Joao Soares, University of Alberta
- 12:20-1:30** **Lunch in the Colonnade Room**
- 1:30-3:10** **Session II**
- 1:30-2:10 *Copolymer Characterization by Quadruple- and Quintuple-Detector SEC*
Andre Striegel, National Institute of Standards (NIST)
- 2:10-2:20** **Student Submission Award Winner Announcement and Introduction**
- 2:20-2:50 *Molecular Weight Characterization of Poly(ionic liquid)s by Gel Permeation Chromatography using Salt-Containing Eluents: A Facile "Vaccination" Approach*
Hongkun He, Carnegie Mellon University
- 2:50-3:20 *Enhanced High-Temperature GPC of Polyolefins by Using Filter-Based IR Detectors*
Alberto Ortín, Polymer Char
- 3:20-3:30** **PM Break**
- 3:30-4:40** **Session III**
- 3:30-4:10 *Characterization of Single-chain Nanoparticles by Triple-Detection SEC*
Erik Berda, University of New Hampshire
- 4:10-4:40 *Detailed Characterization of Gelatins using Aqueous Gel Permeation Chromatography with on-line Triple Detection System*
Dawn Z. Herrick, Pfizer Consumer Healthcare
- 4:40-5:30** **Poster Session**
- 6:30-8:00** **Group Dinner**

Wednesday, September 2, 2015

- 7:00-9:00** **Breakfast Open**
- 9:00-10:00** **Session IV**
- 9:00-9:30 *Small Polymer Characterization by GPC/MS for Big Applications*
William J. Simonsick, Jr., The Lubrizol Corporation
- 9:30-10:00 *Size Exclusion Chromatography (SEC) Measurement of Polyamides*
Fumiya Nakata, Tosoh Corporation
- 10:00-10:20** **Coffee Break**
- 10:20-12:00** **Session V**
- 10:20-10:50 *Bridging the Gap between Theory and Method Development in Adsorption Chromatography*
Kathryn Beers, National Institute of Standards (NIST)
- 10:50-11:20 *Not All Long Chain Branching In Polyethylene Is Created Equal*
Youlu Yu, Chevron Phillips Chemical Company, LP
- 11:20-12:00 *GPC Separation and Detection as Observed through Chromatographic Simulation*
David Gillespie, Dow Chemical Company
- 12:00-1:15** **Lunch in the Colonnade Room**
- 1:15-2:15** **Session VI**
- 1:15-1:45 *The Elucidation of Macromolecular Architecture Using Gel Permeation Chromatography and Mass Spectrometry*
Scott Grayson, Tulane University
- 1:45-2:15 *The Effect of Nanoconfinement on Free Radical Polymerization*
Haoyu Zhao, Texas Tech University
- 2:15-2:30** **PM Break**
- 2:30-3:30** **Session VII**
- 2:30-3:00 *Microstructure Separation and Analysis of Polyisoprenes by Online LCCC-NMR and SEC-NMR*
Pritish Sinha, National Institute of Standards (NIST)
- 3:00-3:30 *Evaluation of Long Chain Branching of Polyphenylene Sulphide Using Gel Permeation Chromatography with Triple Detection*
Graham Cleaver, Agilent Technologies LDA UK Ltd
- 3:30-3:40** **Closing Remarks**

Oral Presentation Abstracts

Polymer Synthesis At and Beyond the Limits of Characterization

Jimmy Mays, Ph.D.

University of Tennessee, Department of Chemistry, Knoxville, TN

Size exclusion chromatography (SEC) has, over the past half-century, become the method of choice for characterizing molecular weights and molecular weight distributions of polymers and copolymers. The popularity of SEC in part reflects its simplicity and easy implementation; using only three solvents and commercially available columns, hardware, and software, almost any soluble polymer can be characterized. Advances in calibration methods and implementation of molecular weight/molecular size sensitive detectors have further entrenched SEC as the premier technique for polymer molecular weight determinations.

However, a number of recent studies on model polymers using new and powerful techniques, such as matrix-assisted laser desorption/ionization time-of-flight mass spectrometry MALDI-TOF-MS and, especially, temperature gradient interaction chromatography (TGIC), reveal serious limitations of SEC as a method for characterizing polymers ranging from simple linear polymers made by anionic polymerization to complex polymer and copolymer architectures. In this presentation, I will review some of these limitations which are largely a consequence of three factors: the large band broadening effects in SEC, its inherently poor resolution, and the size-based separation mechanism.

Characterization of Aqueous Phase Oligomers Produced in an Acrylic Emulsion Polymerization Process by GPC-RI/MS

Wei Gao, Ph.D., Tianlan Zhang, Ralph Even, Debra Kline
Dow Chemical Company, Collegetown, PA

Emulsion polymerization is a widely used technique for the synthesis of dispersions of polymer particles, which have been used in a wide variety of applications and formulation types, ranging from architectural and industrial coatings to delivery systems for agricultural chemicals. The use of polymer dispersions is increasing worldwide, due to a variety of properties, an inexpensive production process, the simplicity of their handling, and the environmental advantage of working with water as dispersion medium instead of organic solvents.

Various models have been proposed in order to understand different physical and chemical events taking place during emulsion copolymerization¹. Although many aspects of the process are now partially understood², there are still many remaining issues³. The less understood aspects of the emulsion (co)polymerization process are the events taking place in the aqueous phase. The study of oligomers present in the aqueous phase is important to understand the entry and exit of radicals, as well as their roles in particle stabilization, nucleation and particle morphology in emulsion polymerization.

In this talk, the aqueous phase oligomers produced in a model process (gradually fed, seeded butyl acrylate/acrylic acid emulsion with redox activated persulfate initiator) will be discussed. The aqueous phase oligomers formed during polymerization were isolated and characterized by GPC-RI/MS to determine the concentration, the molecular structure, and the formation mechanism of the aqueous phase oligomers as well as soluble radicals during the emulsion polymerization. The z-mer radicals captured by 4-hydroxy-TEMPO were also identified in the analysis and their compositions were in good agreement with the suggested composition of sulfate anion free radical adducts based on hydrophobic free energy of adsorption⁴.

References

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Improved Performance of Resins Modified by Reactive Nanogel Additives

Matthew Barros
University of Colorado at Denver, Denver, CO

The objective of this study was to controllably prepare and introduce compositionally equivalent, reactive nanogels of different size and molecular weight into resin to demonstrate how nanogel design can be used to favorably balance the desired reduction in polymerization stress with the impact of prepolymer on viscosity.

Nanogels were synthesized by thermal polymerization of isobornyl methacrylate and urethane dimethacrylate (UDMA; 70:30 mol) with a chain transfer agent in methylethyl ketone at varied solvent concentrations. Following nanogel formation, methacrylate functional groups were added to chain ends. The nanogels were characterized by triple-detector GPC and then loaded at 30 wt% in a UDMA/triethylene glycol dimethacrylate (TEGDMA) resin (60:40 mass). Resin viscosity was determined (viscometer) and visible-light photopolymerizations were conducted with real-time conversion measured by near-IR spectroscopy. Photopolymer flexural strength and modulus were obtained in three-point bending with dynamic stress measurements monitored by tensometer.

Nanogel molecular weight was inversely proportional to the solvent concentration used during nanogel synthesis with a range of 47 - 11 kDa for the 1:5 and 1:10 monomer-solvent ratios, respectively.

Monomer:solvent	Conversion, %	Viscosity, mPa*s	Flexural Strength, MPa	Modulus, GPa	Stress, MPa
Control resin	68.3(0.3)	2265(144)	77.5(6.3)	1.72(0.09)	1.66(0.08)
1:5	66.5(3.5)	7515(34)	60.2(29.7)	1.63(0.12)	0.71(0.03)
1:6	65.3(0.8)	6860(114)	88.4(9.6)	1.83(0.22)	0.65(0.05)
1:7	69.7(0.2)	5805(86)	105.8(8.7)	2.63(0.13)	0.61(0.02)
1:8	68.4(0.3)	5655(114)	76.8(8.3)	2.16(0.27)	0.68(0.06)
1:9	65.7(1.1)	4645(92)	84.8(10.9)	2.44(0.31)	0.69(0.00)
1:10	69.9(0.7)	4350(117)	98.1(7.4)	2.57(0.13)	0.67(0.02)

The addition of 30 wt% nanogel, which is above its percolation threshold, had no significant effect on conversion and in many cases resulted in increased mechanical properties. Even though the interfacial surface area increases dramatically as nanogel size and molecular weight is decreased, much lower resin viscosities were achieved with the smallest nanogels and over the range examined here, substantial polymerization stress reduction could be obtained coupled with enhanced polymer modulus.

Support NIH/NIDCR R01DE022348

How to Use GPC-IR to Identify the Minimum Number of Active Site Types on Ziegler-Natta and Phillips Catalysts Used to Make HDPE and LLDPE

João B. P. Soares

Department of Chemical and Materials Engineering, University of Alberta Edmonton, AB, Canada

Polyolefins are made with comonomers that contain only carbon and hydrogen atoms, but despite their apparent simplicity, polyolefins are present everywhere in domestic appliances, automotive and aeronautical parts, and biomedical devices. The key to their versatility lies in the variety of ways that their simple monomers can be combined to form different polyolefin microstructures. GPC-IR is a powerful technique to quantify these microstructures.

High-density (HDPE) and linear low-density (LLDPE) polyethylenes are made using Ziegler-Natta, Phillips, and metallocene catalysts. The first two catalysts have more than one active site type, while metallocenes are single-site catalysts. This difference appears clearly in their respective GPC-IR profiles (Figure 1).^[1]

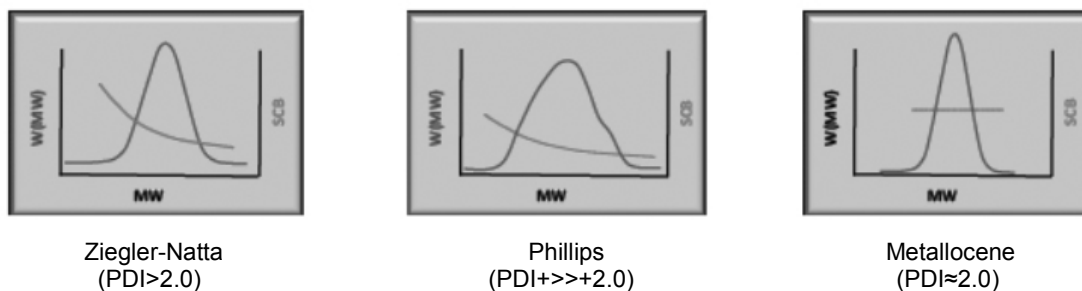


Figure 1. Typical GPC/IR plots of commercial polyethylenes made with different catalyst types.

The breadth of the molecular weight distribution (MWD) and frequency of short chain branching (SCB) across the MWD strongly affect the properties of HDPE and LLDPE.^[2] The typical broad MWD and the characteristic decrease in SCB frequency with increasing molecular weight of polyethylenes made with Ziegler-Natta and Phillips catalysts is caused by the presence of more than one active site type on these catalysts. Each active site type makes polymer populations with different molecular weight and SCB averages. If we want to determine the minimum number of active site types on these catalysts, we can deconvolute these MWD-SCB profiles measured by GPC-IR (Figure 2).^[3,4]

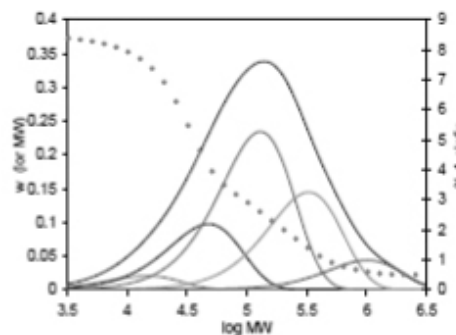


Figure 2. Representation of a broad MWD-SCB profile as a superposition of several narrow MWD-SCB profiles for each site type on the catalyst.

In this presentation, we will review techniques for MWD-SCB deconvolution and show how they can be used to understand the nature of active sites on Ziegler-Natta and Phillips catalysts.

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Copolymer Characterization by Quadruple- and Quintuple-Detector SEC

André M. Striegel, Ph.D.,^a Imad A. Haidar-Ahmad,^b Steven M. Rowland^c

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^bNovartis Pharmaceuticals, San Carlos, CA

^cFuture Fuels Institute, Florida State University, Tallahassee, FL

The properties imparted, oftentimes synergistically, by the different components of copolymers and blends account for the widespread use of these in a variety of industrial products. Most often, however, processing and end-use of these materials (especially copolymers) is optimized empirically, due to a lack of understanding of the physicochemical phase-space occupied by the macromolecules. Here, this shortcoming is addressed via size-exclusion chromatography (SEC) coupled to a variety of detection methods, including multi-angle static light scattering (MALS), quasi-elastic light scattering (QELS), differential viscometry (VISC), ultraviolet absorption spectroscopy (UV), and differential refractometry (DRI). The quadruple-detector method (which doesn't include QELS detection) and the quintuple-detector method (which does) have been applied here, respectively, to organic-soluble and water-soluble copolymers in which either only one or both of the functionalities present absorb in the UV region of the electromagnetic spectrum.

Molecular Weight Characterization of Poly(ionic liquid)s by Gel Permeation Chromatography using Salt-Containing Eluents: A Facile “Vaccination” Approach

Hongkun He, Ph.D.,^{1,†} Hunaid Nulwala,^{1,2,*} and Krzysztof Matyjaszewski^{1,*}

¹Center for Macromolecular Engineering, Department of Chemistry, Carnegie Mellon University, Pittsburgh, PA

²National Energy Technology Laboratory, United States Department of Energy, Pittsburgh, PA

[†] Presenting author:

Objectives:

Polymerized ionic liquids or poly(ionic liquid)s (PILs), polymers with ionic liquid moieties as the repeating unit, have attracted much attention in recent years due to their combination of properties from both ionic moieties and polymers. As a special type of polyelectrolyte, PILs are an important class of technologically relevant materials and have a large variety of applications. However, gel permeation chromatography (GPC) characterization of PILs is challenging, which poses an obstacle to the research and applications of PILs. This is because traditional GPC is not suitable for the characterization of PILs due to the interaction between the ionic groups and column fillers, which caused the adsorption of PILs in GPC columns.

Methods:

We developed a novel method to solve this problem by using salt-containing eluents. For the GPC characterization of PILs containing bulky organic anions (such as bis(trifluoromethylsulfonyl)imide (Tf₂N⁻), BF₄⁻, and PF₆⁻), salts containing the same type of anions (such as LiTf₂N, NaBF₄, and KPF₆, respectively) were added into the eluents (such as tetrahydrofuran (THF) and N,N-dimethylformamide (DMF)) to prepare salt-containing eluents. In addition, a GPC calibration curve was generated by using PILs with known molecular weights and narrow molecular weight distributions as the standards, which is essential for obtaining accurate GPC results.

Results:

By using the salt-containing eluents, the PILs showed normal polymer peaks in the GPC traces. The accuracy of this approach was verified by nuclear magnetic resonance (NMR) end group analysis. This method has been successfully used for the GPC characterization of different types of PILs containing imidazolium, phosphonium, ammonium, or rhodocenium cations with Tf₂N⁻, BF₄⁻, or PF₆⁻ anions, and PIL block or random copolymers. This approach is analogous to the “vaccination” process. By injecting with salts as the “vaccines” into conventional GPC eluents, GPC columns can be “vaccinated” and become enduring for the PILs containing the same type of anions.

Conclusion:

In summary, a simple and universal GPC technique was established to characterize PILs using salt-containing eluents, opening an avenue for the molecular characterization of PILs.

Acknowledgements:

NSF support (CHE-1039870 and DMR-0969301) and DoE support (ER-45998) are acknowledged. This technical effort was also performed in support of U.S. Department of Energy’s National Energy Technology Laboratory’s ongoing research on CO₂ capture under contract DE-FE0004000.

Enhanced High-Temperature GPC of Polyolefins by using Filter-Based IR Detectors

A. Ortín, B. Monrabal, P. del Hierro, E. López
Polymer Char, Spain

Filter-based IR detectors are becoming the standard in the industry for Polyolefin HT-GPC applications. Reasons for this are the high quality of the concentration chromatograms (1), and the additional information on chemical composition they provide (2-4). A higher quality concentration signal results in improved precision of molar mass distribution (MMD) and moments when using conventional calibration methods, but also when applying universal calibration or triple detector methods, for absolute MMD and also long chain branching (LCB) analysis.(5)

The availability of chemical composition information along the MMD provides a new dimension to the HT-GPC analysis, helping in catalyst research, competitive analysis and also in solving technical service problems. The filter-based IR detector provides this information in a very simple way, without adding any analytical complexities nor requiring external utilities such as Liquid Nitrogen.

In this talk, the filter-based IR detector operation principles and calibration procedures are described, and different applications are presented to illustrate the above-mentioned advantages.

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Characterization of Single-Chain Nanoparticles by Triple-Detection SEC

Erik B. Berda, Ph.D., Alka Prasher, Christopher K. Lyon and Justin P. Cole
University of New Hampshire, Durham, NH

Well-defined soft nanostructures with dimensions in the 5-20 nm size regime remain a challenging target by conventional synthetic methods. In recent years our group has been examining the intramolecular cross-linking of linear polymers in dilute solution as a viable route to such nanomaterials. These so called Single-chain Nanoparticles (SCNP) present challenges from both synthetic and analytical standpoints. Our talk will present some recent synthetic breakthroughs in this area while highlighting the application of triple detection SEC as a primary characterization tool.

Detailed Characterization of Gelatins using Aqueous Gel Permeation Chromatography with On-Line Triple Detection System

Dawn Z. Herrick; Benjamin Crawshaw; X. Michael Liu
Pfizer Consumer Healthcare, Richmond, VA

Gelatins are mixtures of water soluble proteins obtained by denaturation and partial hydrolysis of collagen, the main fibrous protein in animal bones, cartilage and skins. The use of gelatins plays an important role in pharmaceutical and consumer healthcare industries in widespread applications such as hard and soft gelatin capsules, gummies, coating, and numerous drug delivery media. In solution, gelatins show a broad molecular weight distribution based on size exclusion chromatography (SEC); however, information available on gelatin characterization using gel permeation chromatography with triple detection (GPC-TD) is limited. The intent of this research is to determine absolute molecular weight, molecular weight distributions, molecular sizes, intrinsic viscosity, and other physical properties of gelatins by GPC-TD to relate and predict process-ability and product performance of soft gelatin capsules. Raw material gelatins from various sources and proprietary blends were examined by GPC-TD, and significant differences in average molecular weight and molecular weight distributions were acquired from the light scattering detectors, differential refractometer and differential viscometer. GPC-TD may be used to perform reverse engineering, competitive product analysis, drug-gelatin interactions, and crosslinking studies of gelatin-based products.

Small Polymer Characterization by GPC/MS for Big Applications

William J. Simonsick, Jr., Ph.D.
The Lubrizol Corporation, Cleveland, OH

We have interfaced gel permeation chromatography (GPC) to mass spectrometry (MS) through an electrospray ionization source. GPC/MS affords an on-line absolute molecular weight detector for molecules that have been size-separated. Only 0.5% of the GPC effluent is required for MS. A sheath flow addition of sodium iodide to the electrospray source facilitates soft ionization by sodium adduction. GPC/MS coupling reduces the number of components entering the MS ion source, and yields accurate molecular weight information for different polymer architectures. High resolution with accurate mass measurements performed on Fourier-transform MS based systems allow the direct determination of charge state, monomer repeat unit and end groups to approximately 10,000 g/mol. Tandem MS studies performed on polymer molecular ions provide information about the specific sequence and functionality location. Highly functional low-molecular-weight materials are used in today's high-solid coating formulations and as precursors to complex polymeric architectures such as block, graft or comb polymers. Applications of GPC/MS to polyesters, acrylics and polyethers will be presented.

Size Exclusion Chromatography (SEC) Measurement of Polyamides

Fumiya Nakata
Tosoh Corporation, Japan

Objectives:

Polyamides are superior polymers in high durability and toughness, which have repeated amide linkages. The aliphatic polyamides are often called Nylons (originally, the trade name by DuPont). They are one of the polymers having difficulties in the SEC measurement because of their low solubility. Approximately 20 years ago, the SEC measurements of polyamides were performed by using m-cresol or phenol as an eluent at high temperature. However there existed problems of an unstable baseline with refractive index detector and poor reproducibility. Recently, eluent of HFIP (hexafluoroisopropanol) have enabled the SEC measurement at normal temperature, but this solvent is very expensive. In this study, 2 kinds of cost effective and easier SEC measurement methods for several polyamides were studied.

Methods:

- 1) We applied N-trifluoroacetylation with trifluoroacetic acid (N-TFA reaction) to dissolve polyamides in common SEC solvents such as THF. It is known that N-TFA- polyamides can easily get inverse reaction and return to original polyamides in the existence of water. Thus the stability of N-TFA-polyamides in THF solvent was studied every hour after reaction.
- 2) Other cheap fluorine solvents such as TFE (trifluoroethanol) were used as eluent for SEC measurement. Some of polyamides, which were not dissolved in TFE were dissolved in HFIP and measured with TFE as an eluent. Measurements were carried out with TSKgel SuperH columns (polystyrene divinylbenzene resin packings) and TSKgel SuperAW columns (polymethacrylate resin packings). The elution behavior of polyamides with each of columns was investigated.

Results and conclusions:

- 1) Appropriate chromatograms of N-TFA-polyamides were obtained for SEC measurement with THF. The stability of N-TFA-polyamides in THF was confirmed to be for up to 15 hours. The molecular size of N-TFA-polyamide was different from original polyamides. The degree of the difference depended on the number of amide functional group in polymer. Therefore, this method could be applied only for comparison evaluation of polyamides with the same molecular structure.
- 2) Different results were obtained with the two types of columns. Eight kinds of polyamides which we tried in this study were analyzed well by TSKgel SuperAW column, while some polyamides did not eluted by TSKgel SuperH column because of difference of hydrophobicity of resin. It was suggested that TSKgel SuperAW column was suitable for SEC measurement of polyamides with TFE as an eluent.

Bridging the Gap between Theory and Method Development in Adsorption Chromatography

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The experimental link between thermodynamic theory, computational models and predictive tools for adsorption chromatography still have limited applicability. We are seeking to determine experimental methods that test current theory, detect weak transitions that govern separation methods, and accelerate model development that is truly predictive for varied and complex chemistries. We will present examples using polymer brush dynamics to study chain conformation near surfaces using neutron and x-ray reflectivity. The talk will also reference some related work at NIST aimed toward advancing theory and computational tools.

Not All Long Chain Branching in Polyethylene is Created Equal

Youlu Yu

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Long-chain branching (LCB) is a very important polymer architectural attribute for polyethylene resins and has been extensively studied since the creation of the first polyethylene resin made using peroxide catalyst under high temperature and high pressure (i.e. LDPE). The presence of LCB not only affects polymer processability but also polymer mechanical properties. Depending on applications, the right amount, type, and distribution of LCB are necessary in order to produce products that meet the desired processability and end-use properties. In this talk, we will present LCB characterization of LDPE, and polyethylene resins made using the Phillips catalyst, Ziegler-Natta catalyst and metallocene catalyst under low pressure and low temperature using size-exclusion chromatography (SEC, also known as gel-permeation chromatography, GPC) hyphenated with multi-angle light scattering (MALS) (i.e. SEC-MALS) in conjunction with other spectroscopic, rheological and fractionation techniques. Some of these results will be compared to those obtained via asymmetrical flow field-flow fractionation (AF4) coupled with MALS (i.e. AF4-MALS). Polymer architectural characteristics of these four types of PE resins will also be discussed in this presentation.

GPC Separation and Detection as Observed through Chromatographic Simulation

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Separation of structural blends in GPC is challenged through the co-elution of different molecular weight chains of independent molecular structures. Moreover, the general slope of the GPC separation is dependent upon the molecular structure, itself, with more linear molecules separating with a higher resolution than their branched counterparts. Size exclusion artifacts such as band-broadening, column exclusion, and late elution of ultra-high MW components can complicate structural interpretation. Furthermore, various single-detector and multi-detector solutions can yield different perspectives on the breadth of the molecular weight distribution, and modeling allows us to reconcile data between methods. By modeling local polydispersity along the column separations window we can gain insight into probable artifacts and consider them properly when interpreting molecular information. Furthermore, modeling allows us to address multi-variate topics such as the relationship between band-broadening, effective column resolution, and targeted accuracy of the distribution information required. Finally, we can enhance our understanding of precision and accuracy by simulating white noise, drift, spiking, and oscillations to explore we can use modeling to assess best practices for baseline setting, integration limits, and detector extrapolation.

The Elucidation of Macromolecular Architecture using Gel Permeation Chromatography and Mass Spectrometry

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Objectives: The development of new techniques for making non-linear polymers, e.g. branched or cyclic polymers, comes paired with a characterization challenge: How do we confirm the architecture of a product, and how can we identify the structure of trace architectural impurities. Two complimentary techniques will be explored for two different case studies: first, the use of Gel Permeation Chromatograph (GPC) coupled with Matrix-Assisted Laser Desorption Ionization Time of Flight Mass Spectrometry (MALDI-TOF MS) to examine impurities generated during polymer cyclization; and second, the use of Ion Mobility Spectrometry-Mass Spectrometry (IMS-MS) to elucidate the structure and purity of well-defined PEG star polymers.

Methods: *GPC-MS Characterization:* GPC data were acquired on a Waters model 1515 isocratic pump (Milford, MA) with THF as the mobile phase with a 1 mL/min flow rate with columns (PLgel 5 μ m MIXED-D, PLgel 5 μ m 500 Å, and PLgel 5 μ m 50 Å) at a constant 30°C and a Model 2487 differential refractometer detector. Mass spectral data were collected using a Bruker-Daltonics MALDI-TOF Autoflex III mass spectrometer in linear mode with positive ion detection. Typical sample preparation were mixed in a 200/5/2 ratio (dithranol/polymer/sodium trifluoroacetate), All MS data were calibrated against SpheriCal dendritic calibrants from Polymer Factory (Stockholm, Sweden).

IMS-MS Characterization: IMS-MS spectra were obtained using a Waters SYNAPT G2. The polymer analyte and cesium acetate were dissolved in 50/50 water/acetonitrile at a 1/10 polymer/salt ratio. Mass spectral data range analyzed was 500-2500 *m/z*. 250 μ L of analyte sample were infused at a rate of 25 μ L min⁻¹ with a capillary voltage of 2.8 kV and a source temperature of 80 °C. Ion mobility spectrometry was carried out with nitrogen drift gas at a pressure of 1.86 mbar (1.40 Torr), an IMS wave velocity of 650 m s⁻¹ and wave height of 40 V.

Results and Discussion: GPC-MS techniques were utilized to elucidate the identity of higher molecular weight impurities observed during the click cyclization of linear polystyrene precursors. By isolating fractions by GPC and carrying out detailed structural characterization, the topology of the cyclic dimers could be confirmed. In a separate study, well-defined PEG stars were prepared such that the length of each arm as well as the number of arms on a star could be controlled. IMS-MS demonstrated the ability not only to differentiate branching according to the number of arms present, but also distinguish between stars with equal-length arms from those with differing arm lengths.

The Effect of Nanoconfinement on Free Radical Polymerization

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The properties of polymers synthesized by nanoconfined free radical polymerization are studied using differential scanning calorimetry (DSC) and gel permeation chromatography (GPC). Two monomers, methyl methacrylate (MMA) and ethyl methacrylate (EMA), are investigated. Nanoconfinement is realized by imbibing the monomer into the pores of borosilicate controlled pore glass (CPG) for pore sizes ranging from 8 to 110 nm. Both hydrophobic and hydrophilic pore surfaces are investigated. The results show that the number-average and weight-average molecular weights increase for PMMA synthesized under CPG confinement, whereas the polydispersity index (PDI) at full conversion decreases relative to the bulk value. Changes from the bulk are larger for the polymers synthesized in hydrophilic pores. At high reaction temperatures, the ceiling temperature is shifted to lower temperatures in nanopores, and this is attributed to a decrease in the change in entropy on propagation (ΔS_p), resulting in a more negative value for the nanoconfined reaction. Modeling of the data suggests that ΔS_p , which is a constant for the bulk equilibrium polymerization, is a function of temperature under nanoconfinement presumably due to reduced chain lengths at high temperatures. The scaling of the confinement entropy on chain length will be tested in future work.

Acknowledgement

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Microstructure Separation and Analysis of Polyisoprenes by Online LCCC-NMR and SEC-NMR

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It is shown that online coupling of liquid chromatography at critical conditions (LCCC) and ^1H NMR spectroscopy can be used to separate and analyze polyisoprenes with respect to their isomeric microstructures. It was possible to separate blends of copolymers consisting predominantly of 3,4- and 1,4-isoprene units by using critical conditions of 1,4-PI. Critical conditions of 1,4-PI were established such that polymers containing predominantly 3,4-PI elute in the size exclusion mode. Furthermore, on-flow NMR detection allows for the complete eluate analysis and the detection of all three isomeric species, such as 1,2-, 1,4-, and 3,4-isoprene, in the eluting fractions. These three individual moieties were correctly quantified in the individual blend components. It was also found that the 3,4-PI samples were random terpolymers of 1,2-, 1,4-, and 3,4-isoprene, whereas the 1,4-PI samples were copolymers of 1,4- and 3,4-isoprene. In addition, SEC-NMR allowed for a fast and precise molar mass calibration and the calculation of the relevant molar mass parameters M_w and M_n .

Evaluation of Long Chain Branching of Polyphenylene Sulphide using Gel Permeation Chromatography with Triple Detection

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Due to its exceptional heat, chemical and flame resistance Polyphenylene Sulphide (PPS) is being used in a wide variety of applications where traditionally metals were used. Such applications include cooking appliances, sterilisable laboratory equipment, hairdryer grills, and automotive components where thermal stability is a pre-requisite.

The properties of the commercial grades depend on both the molecular weight and the structural properties of the resin. The latter can be divided into partial cross-linking (branched) and linear types. Branched PPS was introduced commercially in the 1970s, and the resulting resin is dark in color, hard and brittle. Process refinements in the 1980s led to a linear polymer, which offers significant cost and performance benefits over branched PPS, and overcomes the weak points of branched PPS (1).

Due to its excellent chemical resistance and high melting temperature, the characterisation of these materials by high temperature gel permeation chromatography provides a challenge. PPS will dissolve in 1-chloronaphthalene at temperatures at and above 210°C and in a previous study (2) we presented the possibility to analyze linear PPS at 210°C using a commercially available gel permeation chromatography (GPC) instrument with triple detection (differential refractive index, viscometer and light scattering). We obtained practical solutions to overcome the major difficulty to calibrate the instrument detectors due to the very low value of dn/dc of polystyrene (PS) in 1-chloronaphthalene and succeeded in measuring the Mark-Houwink parameters for linear PPS reporting similar values to those initially determined by Stacy (3) in 1986.

In this study we discuss the measurement of “branched” PPS and report long chain branching distribution as a function of molecular weight.

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Poster Presentation Abstracts

Poster: Characterization of Synthetic Polymers using Ultra-high Pressure Two-dimensional Liquid Chromatography

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Traditionally, online comprehensive two-dimensional liquid chromatography (2D-LC) has been used in the analyses of complex mixtures for its gain in total peak capacity and the ability to reveal “hidden” peaks. In spite of the improved resolution possible with 2D-LC, the longer analysis time has been hindering wide adoption of this technique in fast-paced industry settings. Analysis of synthetic polymers often adopts size exclusion chromatography (SEC) and interactive high performance liquid chromatography (HPLC) as the two dimensions to reveal molecular weight and chemical compositional distributions. Opportunities exist to improve the throughput in both dimensions. In this work, sub-3 micron particle column technology for polymer SEC (Waters ACQUITY™ Advanced Polymer Chromatography, APC) is applied. Using the separations of copolymer polyol samples as an example, a 2-fold improvement in speed is immediately achieved by using APC columns instead of a regular SEC column. In addition, to optimize the gradient.

Poster: Characterization of a Cationic Copolymer Product by Comprehensive Two-Dimensional Liquid Chromatography

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Introduction:

Many polymer products have more than one distribution, such as molecular weight (MW), chemical composition, and architecture. A simple Size Exclusion Chromatography (SEC) analysis, which separates polymers based on hydrodynamic volume and determines relative MW, is often insufficient to fully characterize such complex polymers. In recent years, Two-Dimensional Liquid Chromatography (2D LC), a combination of High Performance Liquid Chromatography (HPLC) and SEC, has been developed primarily by academia to determine the molecular weight and composition distributions at the same time.

In industry, we also face the challenges of analyzing complex polymer products, e.g., a mixture of polymers or a copolymer product with homopolymer impurities. To address this critical need, 2D LC method development for simultaneous molecular weight and compositional analysis of complex polymers is necessary.

Objectives:

To develop a 2D LC method for characterizing a complex cationic copolymer product.

Methods and Results:

In this presentation, we describe a method for characterizing a complex cationic copolymer product by advanced two-dimensional liquid chromatography. Due to the similarity in molecular weight of the copolymer and the homopolymer impurity in the product, they cannot be separated and characterized by SEC analysis alone. A 2D LC method was developed to resolve this issue. The first dimension of the 2D LC system is an HPLC system and the second dimension is an SEC system. The two independent systems are hyphenated by a ten-port two-position switching valve. Copolymers and homopolymers in the product were first separated using the HPLC technique. The fractions from the HPLC column were continuously delivered to an SEC column in the second dimension of the 2D LC system for MW determination. A detector was placed between the first dimension HPLC column and the second dimension SEC columns to independently monitor the first dimension separation through a splitter. Therefore, a relative quantification of the copolymer and the homopolymer impurity can be estimated by comparing their peak areas.

Conclusion:

Using an advanced 2D LC technique, molecular weights, composition and relative quantification of the copolymer and the homopolymer impurity in a cationic copolymer product was simultaneously achieved in one injection.

Poster: Characterization of Single-Chain Nanoparticles and Star Polymers using Gel Permeation Chromatography combined with Viscometric Studies

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The exploration of different polymer architectures, such as star polymers or single-chain nanoparticles, is rapidly increasing due to the ability to synthetically tailor specific properties and the potential to utilize these types of polymers in many areas including catalysis, imaging, nanoreactors, and nanomedicine. While both of these polymer systems show promising properties and applications their characterization can be a challenge. Gel permeation chromatography (GPC) is a vital tool in polymer characterization and when using multiple in-line detectors such as multi-angle light scattering (MALS) and viscometry much qualitative and quantitative data can be gained. We have been able to demonstrate the successful synthesis, and isolation of multiple arm star polymers and single-chain nanoparticles. A detailed characterization was achieved through the use of GPC to aid in the analysis of unique macromolecular architectures. A contrast of viscometric and light scattering data of unique polymer architectures and linear polymer analogs through the use of molecular conformation plots supported the transformation from rod like arms or linear polymer chains to a sphere like highly branched stars or nanoparticles.

Poster: Infrared Detection in HT-GPC of Polyolefins

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The analysis of the Molar Mass Distribution of Polyethylene and Polypropylene resins by GPC /SEC has always been considered a demanding task. Using multiple detection methods (with online viscometer and light scattering detectors) maximize the information on the polymer microstructure. A high quality concentration signal is of paramount importance for the reliable application of those methods, in order to obtain accurate and precise information. Recent developments in online IR detection have meant a breakthrough in concentration detection for polyolefin applications greatly improving the quality of the data by those multiple detection methods. IR detectors with a high sensitivity and stability are now a convenient alternative to other concentration detection techniques, while also providing superior performance.

In this poster, data collected from IR detector is presented, summarizing the three main advantages of the technique for polyolefin analysis: outstanding baseline stability, absence of spurious injection peaks, and simultaneous measurement of chemical composition.

Poster: Analysis of Ultra-High Molecular Weight Polyethylene (UHMWPE) by GPC-IR and Viscometry

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The unique mechanical and chemical resistance properties of UHMWPE are making this technical polymer grow in its range of applications and industrial importance. From an analytical point of view, it presents many challenges given the extremely long chains that result in very high viscosity of solutions. In this poster, analytical tools that are helpful to succeed in the analysis of UHMWPE are presented.

For HT-GPC analysis, the polymer needs to be dissolved at a very low concentration level so that the injected solution does not block the system frits or columns and a smooth and stable flow is maintained. Besides the limitations posed by the separation range of GPC columns, precise MMD can only be obtained by means of an extremely sensitive and stable detector. IR detection has proved to be successful in this kind of analysis, that is, when the injected mass has to be reduced by more than one order of magnitude.

Measuring the intrinsic viscosity of UHMWPE is not only a useful tool for controlling those products, but also for development and other areas, given the difficulties and limitations of other common techniques. The IVA instrument incorporates a robust two-capillary viscometer, together with a high-temperature autosampler, so that a fully-automated analysis of UHMWPE can be carried out conveniently and with high precision, overcoming the problems of the manual or semiautomated methods. By using the built-in IR detector, the actual injected mass of the sample going through the viscometer capillaries is quantified; thus, improving the accuracy and precision of the IV results.

Common to all analytical techniques is the requirement of handling the samples with care to prevent thermal or oxidative degradation during the dissolution and analysis steps. Guidelines on this and on good practices are explained in this work.

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