SAS-Chicago

Understanding Particle Sizing and Zeta Potential

by Eric Olson,
Particle Technology Labs

One of the key physical parameters of various polymers is the particle size. Quite often, the particle size may be used to predict rheological properties, dissolution rates, the ease of flow, and other manufacturing responses. Polymers vary considerably in their particle size, but many can be found in the range of approximately 0.01 to 1 mm. This submicron range is that which is too low to be measured by laser diffraction, but is easily analyzed by dynamic light scattering (DLS).

Another key parameter of various polymers is zeta potential. Zeta potential is often measured in conjunction with DLS since both measurement techniques share common sources, detectors, and other instrumentation. Zeta potential is usually used as a measurement of colloidal stability, but can also be used to predict shelf life and other parameters in certain applications.

Practical Applications of GPC/SEC

by Tom Dent and Michael Kamerlink
Varian

The presentation will focus on practical considerations for successful GPC/SEC analysis. A brief introduction and theory of GPC/SEC will be followed by considerations for column and condition selection. Examples of common problems will be presented, along with troubleshooting advice to overcome these obstacles. These techniques can easily be applied in the laboratory for successful GPC/SEC analysis.

This presentation is suitable for the beginner and the more experienced analyst. New GPC/SEC users will find the material educational and experienced chemists will benefit from the troubleshooting section. A brief introduction to new products and multi detector analysis will provide new tools for solving complex material problems.

The differences between Laser Light Scattering technology and ELSD technology will be discussed to provide an overview of the two technologies. Both are use as detectors for GPC/SEC and determining particle size distribution for molecular and particle characterization in solution.

Industrial Polymer/Material Characterization using MALDI-ToF MS and GPC

by Mark Arnould, Ph.D.
Xerox

Industrial polymers/materials, or polymers in general, were traditionally defined as any substance produced by condensation or addition reactions that yielded a product of very high molecular weight that performed in a desired way. These products were often produced by empirical methods in developing recipes, reactions, processes and control tests. The new substances often found industrial application long before their chemistry or physics were understood. Now, we think of industrial polymers/materials as chemical compounds possessing a chain with repeating units that is used in a produced product; regardless of molecular weight, i.e. specialty chemicals. The more typical compounds are still usually produced by condensation or addition (free radical) reactions yielding large molecular weight compounds, which are complicated mixtures possessing broad polydispersities. In some cases, these compounds are still used in industrial applications before being completely characterized.

Mass spectrometry has been used in the study of synthetic polymer systems for 50 years mainly focusing on polymer additive systems and polymers that had either been chemically or thermally degraded. The development of alternative ionization approaches using lasers (MALDI) and advances in analyzer technology spurred by better electronics (ToF) have increased the use of mass spectrometry in the analysis of polymeric systems. MALDI-ToF MS is ideally suited for the analysis of molecular weight distributions. It is a complimentary technique to more classical approaches for probing polymer microstructure by yielding detailed information on end groups, chemical variations within polymer distributions and the sequencing of complete oligomeric systems. Coupling MALDI-ToF MS to chromatographic techniques further extends the capability by introducing a second dimension of dispersion simplifying the resultant mass spectra allowing for more complete analysis. By more completely characterizing the polymers/materials used in industrial products, a greater understanding of product longevity, production processes, interactions, batch-to-batch variation and “how or why” these products work may be gained.

Modern Solution NMR Methods for Characterizing Synthetic Polymers

by Peter Rinaldi, Ph.D.
University of Akron

This presentation will illustrate the use of solution NMR to characterize the structures of synthetic polymers. NMR is unique among spectroscopic methods because the signal intensities in properly collected spectra can be directly related to the quantities of structures from which they arise; and a multitude of NMR methods are available for proving molecular structure.

This presentation will start with an introduction of some basic NMR concepts. We will discuss the use of NMR methods to resolve and assign resonances, and to measure the quantities of polymer structures. The NMR methods to be discussed will include simple

1D-, multi-pulse 1D-, 2D- and 3D-NMR sequences. Their applications to the determination of monomer sequence distribution, molecular weight, chain-end structure, branching structures and polymerization mechanisms will be discussed.

Vibrational Spectroscopic Tools for Polymer Analysis

by Fred LaPlant, Ph.D.
3M Corp.

Polymer analysis is one of the few areas where classical spectroscopic techniques such as IR continue to be of primary importance. Because such a wide range of properties can be rapidly determined both qualitatively and quantitatively in condensed phase with relatively inexpensive instrumentation and minimal sample preparation, spectroscopy continues to provide solutions that would be difficult or impossible with HPLC or mass spectrometry. Recent advances in instrumentation and sampling have also enabled new applications in on-line control, imaging, and field measurements beyond traditional bench techniques.

This talk will provide a broad overview of the vibrational tools currently available including near-, mid- and far-IR, as well as Raman, with comparisons of the relative strengths and weaknesses of each. The practical considerations such as sample preparation methods and presentation to the instrument associated with these measurements will also be reviewed. Because FT-IR continues to be the most commonly found instrument in industry, focus will be given to using mid-IR techniques to analyze properties such as polymer composition, additives and fillers, crystallinity, and cure monitoring. Special cases such as adhesives, fibers, thin film coatings, and trace analysis will be reviewed. Finally, the relative merits of spectroscopic imaging tools will be reviewed and their place in the analytical lab discussed.

Applications of Rheological Testing for Material Characterization and Performance Predictions

by Greg Kamykowski, Ph.D.
TA Instruments.

Rheology is the study of the flow and deformation of matter. Testing by rheological means is a well-established and sensitive way of characterizing materials and making processing and performance predictions. It is truly a multi-disciplinary field, as rheological testing is employed in diverse areas such as polymer engineering, bioengineering, food science, structured fluids, and nano-composites, just to name a few.

In this talk, the presenter will go over fundamentals of rheological testing, including the definitions of terms used frequently in this field, will show the typical test configurations and test methods employed and the reasons for certain choices. Finally, we will discuss practical examples of how rheological testing has been used as a characterization tool and as a predictor of product performance. Some innovations in accessories will also be mentioned.

Eric W. Olson

Eric received a B.S. in Chemistry from Illinois State He then attended graduate school at Illinois State University from 1997 to 2001, earning his M.S. in Physical Chemistry with an emphasis on Computational Chemistry and Quantum Mechanics.

Eric began his professional career with Cabot Corporation as an Analytical Chemist. There he was promoted to Particle Characterization Scientist, after which he supported the manufacturing and quality units through statistical analysis of data, generation and interpretation of DOE's, and variability reduction efforts. In 2001, Eric accepted the position with Eli Lilly and Company in their Pharmaceutical Product Development division. There transitioned into the Global Quality unit as a Senior Analytical Chemist. There he was in charge of method development, transfer, validation, and verification of chromatographic and spectroscopic methods, which were used in an anti-counterfeiting effort in conjunction with law enforcement agencies, U.S. Customs, and the FDA forensic chemistry center. Eric joined Particle Technology Labs in 2009 as a Senior Chemist.

Eric is a member of the American Chemical Society, ACS Division of Colloid and Surface Chemistry, the Society of Applied Spectroscopy, Council for Near-Infrared Spectroscopy, the Coblenz Society, and the Federation of Analytical Chemistry and Spectroscopy Studies. He has collaborated on peer-reviewed journal articles, has been named on a patent, and has given a two week course on DOE and Statistical Analysis in the U.S., Germany, and the U.K.

Tom Dent

Tom has 14 years of experience in HPLC of small molecule and bio/synthetic polymers. He spent 10 years as an analytical chemist in industry before joining the Varian Polymer Characterization group via the acquisition of Polymer Laboratories. His responsibility for Polymer Characterization products in the Midwest includes technical sales, training and application support. His education includes a Bachelors of Business Administration at Ohio University and Chemistry at the University of North Carolina Wilmington.

Michael Kamerlink

Michael joined Waters in 1986 as a Technical Sales and Support Representative. In 2002 he joined Precision Detectors as North American Sales Manager until Polymer Labs/Varian, Inc acquired Precision Detectors in April 2008. Shortly after that all Laser Light Scattering technology was folded in to the Varian product line of instrumentation for molecular and particle characterization in solution. This includes GPC/SEC and particle size distribution. His position is Product Support Specialist for Laser Light Scattering Technology.

Mark A. Arnould, Ph.D.

Mark received a B.S. in Chemistry from the University of Akron with two years of undergraduate research in free-radical polymerization in the lab of Dr. H. James Harwood of the polymer science department. He remained at the University of Akron and received a Ph.D. specializing in the area of matrix assisted laser/desorption ionization time-of-flight (MALDI-ToF) and electrospray ionization quadrupole ion trap (ESI-QIT) mass spectrometry of synthetic polymers prepared by several lab groups in the polymer science department and completed the first dissertation at the University of Akron in the area of polymer mass spectrometry titled: Mass Spectrometric Studies of Synthetic Polymers.

At the National Research Council, Mark served as a post-doctoral fellow at the National Institute of Standards and Technology (NIST) in the Material Science and Engineering Lab, Polymers Division. The work focused on studying the MALDI process and the covalent cationization and analysis of synthetic, crystalline hydrocarbon polymers using a novel mass spectrometer with a heated source allowing MALDI to be done from room temperature to 200°C.

In 2005, he joined the Xerox Corp. as a Polymer/Materials analytical chemist, where ehe continues to develop applications of MALDI-TOF mass spectrometric techniques coupled with GPC in solving novel synthetic polymer/materials problems including structural determination, sequencing, minor component analysis and quantitation.

Mark has 20 peer reviewed publications in the area of polymer MALDI and his work has been presented in over 20 national and international conferences.

Peter L. Rinaldi, Ph.D.

Peter L. Rinaldi obtained his Ph.D. in 1978 from the University of Illinois at Urbana-Champaign. After an NIH postdoctoral fellowship spent studying with G. C. Levy at Florida State University and with P. Laszlo at the Universite de Liege, Liege Belgium (1978-80), he joined the faculty at Case Western Reserve University as an Assistant Professor. He worked for Varian Instruments as an NMR applications chemist, and later as Manager of NMR Field Applications Laboratories (1983-87). In 1987, he joined the faculty at The University of Akron where he is now Professor of Chemistry, Director of the University of Akron Magnetic Resonance Center, and a Fellow of The University of Akron Institute of Polymer Science and Polymer Engineering. His current areas of research interest focus on development of new NMR methods for studying synthetic macromolecules and applications of new NMR techniques to the characterization of macromolecules. He is involved in all aspects of applying NMR to the study of research problems in the chemical sciences. He is author of over 150 publications, and has taught numerous short courses on basic and advanced NMR methods at national meetings.

Fred LaPlant, Ph.D.

Fred LaPlant received his B.S. in Chemistry from San Diego State University, and his Ph.D. in Analytical Chemistry from Purdue. Fred's research focused on developing novel applications of Raman spectroscopy and microscopy, including fiber-based sensors, detection of subsurface materials, and high-pressure high-shear modeling of fluids.

He spent five years in product development at Perceptron in Ann Arbor, Michigan, a developer of process control and monitoring devices for the auto industry. His principle contribution was the development of a non-contact, laser-based ultrasound system to measure wet paint film thickness. He then moved into Analytical Research and Development at the Pfizer Ann Arbor site, where he applied his process monitoring experience to PAT, as well as promoting the use of spectroscopic tools in pharmaceutical development. He is currently the spectroscopy group leader for 3M Corporate Analytical in Saint Paul, Minnesota, where he is involved in a wide variety of projects, including introduction of new spectroscopic technology, nanomaterial characterization, and development of on-line monitoring methods.

Fred has been active in various capacities in the Society for Applied Spectroscopy at both the local and national level, and is the current president of the SAS.

Greg Kamykowski Ph.D.

Dr. Gregory Kamykowski is a Senior Applications Scientist for TA Instruments and is based in Schaumburg IL. Dr. Kamykowski has a BS in Chemistry from Loyola University Chicago and a PhD in Physical Chemistry from the University of Wisconsin – Madison. Dr. Kamykowski has held a few different industrial positions, all of which have had some relation to rheology. He is a member of SPE, ASTM, and the Society of Rheology.