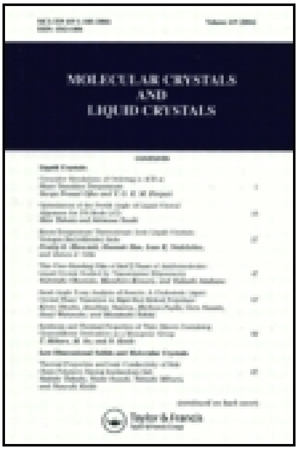

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IN MEMORIAM: ROGER S. PORTER 1928–1998

Eric R. George

The objective of this compilation is to assemble in one coherent body, the history of polymer science as experienced by Professor Roger S. Porter. Professor Porter's life work included scientific innovation, global collaborative research, establishing polymer science as an academic discipline, and actively teaching students. He was a great friend and mentor to many. Professor Porter can best be considered a polymer physical chemist. He was most happy when he was talking, writing, and living science. He was adept in recognizing when scientific discussion had explained ideas clearly then capturing that in writing for publication. He published over 400 journal articles and more than 20 papers in Molecular Crystals and Liquid Crystals. His works are filled with the elucidation of crystalline/amorphous morphology in polymers, characterization of the liquid crystalline state, and the subsequent control of material chemistry, morphology and molecular weight to yield unique balances of rheology and physical properties. Below is a discussion of his scientific career.

I. INTRODUCTION

As opposed to a single chronological order of his complete works, we separated the works into 16 separate categories chronologically. The objective is to promote a continuation of Roger Porter's life works.

From his early beginnings with George Cady in Fluorine chemistry at the University of Washington, his industrial experience at Chevron, to his final days at the University of Massachusetts and the University of Southern Mississippi, Professor Porter's works covered a wide range of chemistry, polymer physics, and polymer engineering. A few highlights include

–Reactions of Trifluoromethyl Hypofluorite
–A thermal method to measure "degree of liquid crystallinity"
–The first observation of a negative normal stress difference in polymer liquid crystals
–Viscosity inflections at critical concentrations in lyotropic systems
–A novel method for processing liquid crystal polymers
–Ultimate mechanical properties in polymers via solid-state extrusion
–Improved methods of molecular weight determination via GPC
–Fundamentals of polymer stress reactions and mechanochemistry
–Transesterification in polyesters and polyester blends
–Reversible plasticization of polyamides
–Functionalization of aromatic polyketones and blends with polyimides

We provide here a representative cross section of his life works and a bibliography of his published works. We would like to note that this compilation of papers came directly from his personal files. Professor Richard Farris at the University of Massachusetts donated them. Therefore, there are some papers that are not accessible via traditional literature search techniques, and unfortunately we probably omitted a few documents.

II. BIOGRAPHY AND RECOGNITION AWARDS

Professor Roger S. Porter was born in 1928 in Windom, Minnesota. He received a B.S., Chemistry, University of California, Los Angeles, 1950. Ph.D., Chemistry, University of Washington, Seattle, 1956 under the direction of George H. Cady. Cady obtained his Ph.D. in Chemistry at University of California at Berkley with Joel Hildebrand. Cady is recognized as a pioneer in fluorine chemistry. Professor Hildebrand was a pioneer in physical chemistry and is known for his work with solubility parameters for predicting compatibility of substances, particularly solvents and polymers. The link back to Cady and Hildebrand is significant and helps to explain the research interests of Professor Porter.

Roger Porter moved on to California Research Corporation (Chevron Research Company), Richmond, California and remained there until 1966. In this period Roger Porter collaborated quite extensively with Edward M. Barrall II and Julian F. Johnson where they published in the area of Cholesteric liquid crystals and binary phase diagrams in addition to other pioneering research in polymer science. (Please refer to a first hand account of the Chevron days summarized by Ed Barrall II, Polymer Engineering and Science, Feb 2001, Vol. 41, No. 2). In 1966 Professor Porter moved on to the University of Massachusetts, Amherst, Massachusetts where he remained until the end of his career. While at UMASS he served as professor, chairman and director of the materials research institute. He also served as a visiting professor at Royal Institute of Technology, Stockholm (1972) and University of London, Queen Mary College (1972–1973). In 1983 he accepted an adjunct professor position at the University of Southern Mississippi, Hattiesburg, Mississippi. One additional position that he personally cherished was acting as science advisor to Lee Iacocca at Chrysler.

Professor Porter was well recognized during his career. Some examples include the Bingham Medal from the Society of Rheology, the Mettler Award from the North American Thermal Analysis Society, International Award from the Society of Plastics Engineers, the Education Award from the American Chemical Society, and the Plastics Hall of Fame. He served as editor of the journals Polymer Composites and Polymer Engineering and Science.

Professor Roger S. Porter passed away on August 25, 1998 after a long battle with cancer.

III. LITERATURE REVIEW

1. The Early Years

Porter's graduate work involved reactions of trifluoromethyl hypofluorite, the perfluorinated analog of methanol. Particularly interesting were the reactions to form perfluorinated peroxides and also the polymerization of tetrafluoroethylene in the presence of the hypofluorite under certain conditions. The initial connection with macromolecules was established.

At the California Research Institute, work was typical of what might be expected from an oil company. He performed basic research in the area of specific heats and thermal conductivities of aviation fluids, thermal decomposition of phosphates, measurements on surface areas and pore sizes of cracking catalysts, and one study that led to the development of antistatic agents for polymer liquids and solutions. This led to demonstrations of "bouncing liquids". Barrall, Johnson, Cantow and Porter were the first to correlate many of the relationships between polymer molecular weight and entanglement theory to the performance of motor oil. They established chromatography and rheology labs at Chevron to bring the fundamental understanding of polymer science to a new level.

2. Liquid Crystals

Porter, Johnson, and Barrall along with several Porter graduate students published a plethora of papers on the order and flow of liquid crystals. The work focused almost exclusively on esters of cholesterol. In this work they demonstrated the structural characteristics of cholesteryl ester liquid crystals that led to smectic, cholesteric, and nematic mesophases. They established structural characteristics that led to monotropism. This is the phenomenon where metastable mesophases are observed only upon cooling from the isotropic melt but not observed after melting the crystalline solid. Porter and coworkers established the rheological characteristics for this homologous series where nematic mesophases exhibited lower viscosities than the isotropic melt whereas smectic and cholesteric mesophases were more like crystalline solids and exhibited higher viscosity. They correlated this to "degree of liquid crystallinity" and quantified it by the ratio of the mesophase transition heat of fusion divided by the total heat of fusion from crystalline solid to isotropic melt.

Some of this work on liquid crystals can be applied to atherosclerosis as explanations for initial formation of blockages in arteries linked to cholesterol and to the chemistry of steroids.

Schell closed out the work on liquid crystals with more traditional studies on dielectric properties of liquid crystals. The eutectic mixture of liquid crystal pairs is often used as the liquid in liquid crystal displays.

3. Chromatography and Fractionation

Porter and Johnson were active in the area of gas chromatography where one of their first papers in 1959 established a method to determine the amount of fuel that leaked into lubricating motor oils. They published two papers in the journal **Nature**, where they developed a circular gas chromatograph and subsequent methods for its use.

R.J.P. Williams introduced the gradient elution method for polymer fractionation in 1952. Porter and coworkers optimized this method. The original gradient elution method started by coating beads with the polymer to be evaluated. Temperature and solvent gradients were then established to progressively separate the polymer into its molecular weight fractions. Porter optimized the controlled introduction of various solvent mixtures, ran several columns in parallel, and established combined temperature gradient, solvent strength combinations. Cantow, Porter and Johnson published an additional paper in **Nature** on large-scale polymer column fractionation in 1961.

Around 1965 gel permeation chromatography became the method of choice for establishing molecular weight and molecular weight distributions for polymers. At the time, there was various nomenclature used for the method including gel filtration, exclusion chromatography, restricted diffusion chromatography, molecular sieve filtration, and molecular sieve chromatography. Porter was instrumental in establishing criteria for the porous beads, how to calibrate the columns with standard polymer distributions, establishing the final nomenclature as well as optimizing and determining sources of error in the new GPC method. Several other publications on chromatography followed including a method to determine comonomer content via pyrolysis-gas chromatography.

4. Rheology and Processing

Porter, Julian Johnson, and others were early in studying the rheology of polymers and polymer solutions with a particular emphasis on correlating flow behavior to polymer structure. They studied the fundamentals of lubricating oils and the effects of polymers in base oils. They elucidated many of the fine details of controlling polymer viscosity versus shear rate and molecular weight (MW).

Prest, Porter, and others contributed to the understanding of entanglement molecular weight and the transition from Newtonian to non-Newtonian flow behavior. For example, they established that the dependence of entanglement chain length might be separated into contributions due to backbone composition and pendant group volume and polarity. Above the critical molecular weight and upon addition of high molecular weight components, there was an exponential dependence of viscosity on MW. Concomitantly, the polymer shear-thinned and showed minimal dependence on temperature. Opposite effects were observed below the critical MW. The characteristic entanglement composition, a product of molecular weight and volume concentration was observed in several distinct ways:

–The onset of the rubbery plateau in viscoelastic measurements
–Changes in relaxation times from NMR
–The change in power dependency of viscosity
–Location of the onset of non-newtonian flow at low shear

Optimized viscosity shear-rate curves for motor oils were established in order to minimize power loss and yet maximize oil life. Reversible and irreversible non-Newtonian behavior was separated into shear thinning and to shear degradation components and established criteria for each. They became experts at high shear viscometry and helped to optimize that method. The basic concepts of polymer rheology were applied to paint and food systems.

Porter helped to elucidate pressure effects on the melt viscosity of a broad range of polymers. Above a critical molecular weight polymers exhibit shear thinning with increasing shear rate but in capillary flow above a critical pressure there can be observed an increase of viscosity versus shear rate. This was explained via free volume theory and has direct practical correlation to extrusion of polymers in industry.

The paper by Kiss and Porter, “Rheology of Concentrated Solutions of Poly (gamma-benzyl-glutamate)” deserves a special note. They observed a negative first normal stress difference under certain conditions of liquid crystallinity and shear rate. Concomitant with the observation of the unique rheology they also discovered a morphological phenomenon of shear induced band structures perpendicular to the direction of shear. This was the first such observation. Secondly, they observed nematic like viscosities in cholesteric mesophases attributed to a shear induced cholesteric/ nematic transition. They observed that in these lyotropic solutions the critical concentration for liquid crystal formation predicted from Flory’s theory occurred at a higher concentration via viscosity measurements. This was attributed to liquid crystal formation prior to the drastic drop in viscosity.

5. Thermal Analysis

Thermal Analysis as a characterization technique permeates much of Professor Porter’s work. We include a separate section on thermal analysis because Porter worked to develop and optimize several techniques. These included an improved differential thermal analysis and expansion apparatus, microboiling point determination, the application of differential scanning calorimetry to purity measurements, and applying fundamental thermodynamics to the melting of semicrystalline polymers.

The broadening and/or depression of the melting transition are a function of crystal size distribution, the thermal conductivity of the sample, the response lag inherent to the measuring technique, and the presence of miscible “impurities”. Additionally, if one can assume equal crystallinity in the resin portion of the sample then the percentage of added components like second amorphous materials and fillers can be determined.

6. Solid State Extrusion

From the drawing of fibers to the biaxial orientation of films, solid phase processing has significant practical application. Polymer properties are a function of their chemistry, molecular weight, and orientation. Professor Porter challenged the then current paradigm of ultimate polymer properties. The original work on properties of highly oriented polyethylene started around 1967 when Porter studied the flow of partially crystalline polyethylene. This was followed by the work with Southern in making polyethylene strong and clear. Capiati committed this work to solid-state extrusion in 1975. Solid-state coextrusion was developed by Zachariades in 1978, and single crystal draw by Kanamoto and Sherman in 1979. In the papers titled "New Methods of Production of Highly Oriented Polymers by Solid State Extrusion" by Watts, Zachariades and Porter and "Solid state Extrusion" from the Encyclopedia of Polymer Science and Technology by Porter, Southern, Capiati, Kanamoto, and Zachariades much of the key work in the Porter lab on this subject is highlighted:

–Conventional solid-state extrusion
–Split billet extrusion and coextrusion
–Powder extrusion including precipitated single crystals
–Reversible plasticization and gel solid-state extrusion

A broad range of polymers were extruded just below their melting point for semi-crystalline polymers and above the glass transition temperature for amorphous polymers. Maximum orientation and crystallinity resulted in properties for some polymers approaching that of single crystals. Referencing the tensile properties of polyethylene single crystals as the quintessential target led to various methods to approach that goal. One idea was to extrude a compaction of precipitated crystals that reportedly minimized molecular chain entanglement. Other approaches such as the extrusion of gels and the use of reversible plasticizers were also utilized to minimize chain entanglement.

Several geometries were outlined that controlled the shear and elongational flow of various polymers during solid-state extrusion. This enabled the ultra orientation of a broader range of material not amenable to solid-state extrusion up to that time. Imagine producing polyethylene with a modulus and strength a minimum of 100X that of conventional processed polyethylenes. Other iterations of solid-state coextrusion included combinations of high and low molecular weight constructions of the same material and split billet "sandwiches" of different polymers that enabled lower pressures and higher draw down ratios.

Saraf and Porter attributed the limits of natural draw to the extension of the amorphous segments. The unraveling of lamellar crystals by deformation without chain rupture could lead to ultimate chain extensions for semicrystalline thermoplastics. They classified the possible ductility mechanisms for crystals and reviewed the theories of Flory, Peterlin and others to explain the phenomenon.

Biaxial orientation of polymers has been a common industrial practice with some of the pioneering work attributed to Miles at Dupont, McGlamery at Phillips, and Alfrey at Dow. Porter, Saraf and coworkers concentrated on the idealized uniplanar draw state as a means to optimize biaxial orientation in semicrystalline polymers.

Lastly, Porter and Zachariades published a paper titled “New Horizons” in 1996 that foresaw many of the polymer applications of the future. The key to property control was cited as a balance of chemistry, physics and rheology. In chemistry through monomer sequence, tacticity and branching, in physics through self-assembly, and rheology for morphology control. Some examples of prospects for polymer products were as barriers and membranes, for biomaterials (artificial organs, controlled drug delivery, and tubes used in non invasive surgery), and “smart” polymers used in nonlinear optics, electrical insulation, and lithography to name a few.

7. Polymer Stress Reactions and Mechanochemistry

Building upon the work in Russia by Baramboin and coworkers (summarized in the book “Mechanochemistry of Polymers”), the work by Watson and coworkers at the Natural Rubber Producers Research Association, and the work by Goodman, Bestul and coworkers at the National Bureau of Standards, Porter advanced the understanding of molecular weight breakdown and other changes in polymers subjected to a variety of external energy sources. He recognized that this had practical significance in processing effects on polymer properties, the recycling of polymers, the use of free radical generation to produce new polymer chemistry, and the effect of temperature, time, mechanical stress, environment, and various types of radiation on the long-term properties of polymeric systems. This culminated in Professor Porter teaching the first formal university course in the United States and co-writing (with A. Casale) a textbook of the same name on “Polymer Stress Reactions”.

Porter studied stress reactions for amorphous polymer chains in dilute solution, in polymer melts, and in the solid state. Bond rupture in the solid state has practical application in predicting useful lifetimes of polymeric systems but also for producing new compositions that are useful and practical. Examples include the use of mechanochemistry in polymer-polymer mixtures to produce new copolymers, radical formation to bond to fillers (used in the tire industry) and to initiate polymerization of monomers to yield graft copolymers.

High shear rheometry and gel permeation chromatography advanced the understanding of polymer stress reactions. The working hypothesis for stress reactions relates to energy storage of entangled macromolecules, the competing processes of slip versus bond breakage, and the consideration of thermal and oxidative breakdown concomitant with mechanical degradation.

Highlights of the principals established in polymer stress reactions include

–The breaking of molecular bonds exhibit complex second order interactions with oxidation and temperature.
–Mechanical energy can mildly degrade polymers with little or no formation of low molecular weight products (longer molecules preferentially break).
–The presence of small amounts of moisture in condensation polymers will favor hydrolysis-activated stress reactions versus radical initiated stress reactions in the absence of moisture.
–The milling of broad molecular weight elastomers narrows the molecular weight distribution (MWD) tending toward Flory's most probable MWD of 2.
–The milling of narrow MWD polymers will often increase the MWD initially followed by a progression towards the most probable MWD with time.
–Once free radicals are formed via stress then all the normal reactions of free radicals are possible; recombination, disproportionation, and reaction with other materials.
–Molecular weight rapidly decreases upon mechanical degradation in the absence of crosslinking, slows down, and then reaches a limiting molecular weight.
–In polymer solutions, parameters such as temperature, concentration, MW, etc. influence the mechanical degradation proportionately to the way they influence the viscosity of the system.
–Negative temperature coefficients of degradation at relatively low shear and temperatures are well documented for a number of polymers.

The high shear concentric cylinder enabled a direct measurement of molecular weight loss versus shear stress and temperature. Porter and Johnson applied a reduced variables approach and found that absolute rate of change of MW increases markedly with increasing initial MW.

8. Liquid Crystal Polymers (LCP)

The liquid crystal phase in polymers can occur in solution (lyotropic) or in the melt (thermotropic). The phenomenon is associated with rigid molecules with high degrees of aromaticity or for polymers that form helixes in solution. Upon the practical application of making ultra high modulus fibers from lyotropic solutions of poly (p-phenylene diamine) by Kwolek at Dupont, research in the area of LCP became quite popular. The subsequent discovery of thermotropic LCP from copolymers of polyethylene terephthalate with p-hydroxy benzoic acid by Jackson at Tennessee Eastman fueled research activity.

Porter's work in LCP centered around two areas: lyotropic polypeptide solutions and thermotropic liquid crystal polyesters. In addition to the work on the rheology of poly (gamma-benzyl-l-glutamate) discussed in an earlier section, Kiss and Porter studied a range of polypeptides at different concentrations and molecular weight in the helicogenic solvent m-cresol. They observed different critical concentrations for the formation of mesophases as predicted by the Flory equations that correlate aspect ratio to critical mesophase concentration.

After the discovery of the negative first normal stress difference in a lyotropic polypeptide system along with the formation of band structures, Kiss and Porter reviewed the theory and experiments of several authors. The Doi theory adequately predicted the new phenomenon and their work could be credited with spawning a host of research that significantly expanded the working knowledge of liquid crystal solutions.

For thermotropic LCP, Porter and coworkers extended earlier work on the rheology and phase diagrams of traditional liquid crystals. The work centered on liquid crystal copolyesters, their rheology, blends with other polyesters, and blends with low molecular weight liquid crystals. It was recognized that transesterification must always be monitored in these systems both in blends and in the individual copolymers.

Key features of the rheology of thermotropic LCP included

–The absence of a low shear Newtonian plateau with pronounced shear thinning.
–Lower viscosity in the nematic mesophase versus the isotropic phase.
–Lower (perhaps negative) die swell versus traditional polyesters.

George and Porter established the conditions that led to eutectic phase diagrams of LCP mixed with LC diluents. They applied the Flory-Huggins theory to obtain theoretical heats of fusion for perfect crystals for new thermotropic LCP and also demonstrated the practical use of low heats of fusion leading to a sharper decline in melting point depression accompanied by lower processing temperatures. The melting point depression of a model LCP-diluent system was compared to that of a series of copolymers of decreasing comonomer. The LCP-diluent system was a much better fit with the Flory-Huggins theory attributed to the copolymers having progressively disrupted crystal structures.

A novel approach for processing LCP was invented when George, Griffin, and Porter invented a novel bi-functional low molecular weight LC ester utilized in a first step to lower the melting point and viscosity of the LCP for lower practical processing temperatures. A post-processing step reacted the polymer and diluent to produce a final product that exhibited a higher melting point and improved physical properties.

George and Porter extended the "degree of liquid crystallinity" concept to those LCP-diluent systems that exhibited isomorphic phase diagrams by taking the ratio of the nematic-isotropic heat of fusion to the total heat of fusion in blends with progressively higher polymer concentration. Extrapolating to 100% polymer yielded a good measure of order in the mesophase. This confirmed that the LCP exhibited more order in the nematic phase versus low molecular weight liquid crystals and that low molecular weight liquid crystals in binary miscible blends are aligned with the LCP. This approach matched results of other researchers using x-ray diffraction and also when the theoretical heat of fusion of the LCP was known.

9. CO2 and Polymers

Wang demonstrated that CO2 permeability was decreased upon solid-state orientation of both amorphous polystyrene and crystalline polyethylene. Beckman extended the work on CO2 with polycarbonate and is best described with a quote from reference 246. "Because CO2 is both the pressure transmitting medium and a diluent whose properties vary significantly with pressure, pressure and temperature combine to affect the crystallization rate, degree of crystallinity, and the melting temperature of the crystals so formed". Beckman was able to produce polycarbonate with crystallinity levels up to 20 percent.

Beckman, Koningsveld, and Porter followed the work of Schottky, utilizing a lattice of filled and vacant sites to model compressible fluids. A host of lattice models could be reduced to one equation of state. This area of research is referred to as mean field lattice equations of state. Professor Eric Beckman went on to champion CO2 and its phase behavior with materials leading to new applications of this technology.

10. Characterization of Polymers

The characterization of polymers permeates all the work of Professor Porter. In this section we attempt to highlight methods that have broader utility and also work that is not covered in one of the other categories. While at Chevron, Porter, Johnson, Barrall and Cantow collaborated on a variety of methods. They developed improved gas chromatography methods for determining contaminants in petroleum streams; utilized rate of pyrolysis and gas chromatography to determine composition of polybutenes; improvements in osmotic pressure molecular weight methods via thermoelectric determination of vapor pressure lowering; and an early study of physical property trends in stereoregular versus atactic polymers.

Complimentary to the work on solid-state orientation of polymers, several techniques to quantitatively measure macroscopic and molecular orientation in polymers were developed and optimized. This included methods to measure surface versus bulk orientation as well as amorphous versus crystalline orientation. The work on shrinkage as a measure of orientation is practically significant in that it can be applied to heat shrink tubing. The paper on surface orientation as measured by contact angle was the first report of differential contact angle in machine versus transverse direction and also along with orientation measurements via FTIR (Dichroism) and birefringence helped to delineate surface versus bulk orientation. The FTIR study (with Hsu, Ito, and Pereira) of coextrusion drawn polyethylene terephthalate is an elegant treatment showing that the change in dichroic ratio of particular absorption bands can describe overall orientation complemented by detailed information of increased trans conformations of C-O bonds in the crystalline phase. Interestingly, there was no change in the gauche isomer of the methylene-rocking band attributed to limited orientation in the amorphous region.

Two additional studies to characterize molecular details of highly oriented polymers were small angle neutron scattering (SANS) and optical birefringence studies on oriented polystyrene. The SANS was complemented by thermal shrinkage and birefringence studies mentioned earlier. Increasing draw ratio had a direct effect on the radius of gyration in the machine direction but an indirect effect in the transverse direction. PET was oriented close to perfect orientation as measured by birefringence (a value of 0.21 was achieved versus a maximum theoretical birefringence of 0.236) if extruded below its glass transition temperature via a coextrusion split billet technique. Thermal expansion studies on PET revealed that non-crystalline tie chains could exert an entropic effect at higher temperatures resulting in a negative expansion coefficient.

The work by Jonza on thermal flash diffusivity of polyethylene determined that increasing crystal and chain alignment was accompanied by an increase in thermal conductivity in that direction.

Parmer and Mjordjevic utilized polymer/low molecular weight mixtures to simulate analogous polymer/polymer interactions of polymer blends.

11. Polyethylene, Polypropylene and Polyolefins

While most of Professor Porter's work on polyolefins is covered in the sections on solid-state extrusion and characterization, much work concentrated on individual polymers versus individual methods.

Barrall II, Johnson, and Porter published an interesting calorimetry study on copolymers polymerized via stereo-selective and non-selective catalysts with mixtures of ethylene and propylene. In the early 60's it is interesting to note they utilized DTA versus DSC and were using a planimeter to measure peak areas and subsequently heats of fusion as measures of crystallinity. This calorimetry method enabled the calculation of ethylene/propylene ratios in copolymers. This represents some early research on ethylene propylene rubber (EPR) materials in that random copolymers showed no crystallinity and were more elastic materials.

Mori, Weeks and Porter adopted a method from Keller and coworkers using nitric acid etching followed by GPC to determine crystal size distributions. These distributions identified up to 18% extended chain polyethylene morphology produced via solid-state extrusion with crystal thicknesses greater than 1000 Angstroms.

Porter and coworkers studied the rheology of linear and branched polyethylene and correlated flow activation energies and low shear viscosity behavior to degree and type of branching. The branching in polyethylene versus other polymers resulted in a steeper increase in viscosity versus molecular weight than predicted by the standard Graessley entanglement theory.

Weeks, Capiati, and Krzewki identified negative coefficient of expansion values along the chain axis of highly extended polyethylene. The expansion coefficient perpendicular to the orientation exhibited positive expansion coefficients as expected. The authors made the case that this is indeed another method of characterization to elucidate the morphology in these systems.

12. Polyesters and Polycarbonates

There was an extensive amount of research devoted to understanding polyester blends and transesterification between polyesters and polycarbonates in the Porter laboratory. This raised the awareness both of trans reactions taking place in single copolymers as well as the need to control transesterification in polyester blends. Indeed, polyester/polycarbonate blends used in industry today are most often modified with specific ingredients to prevent these trans reactions. Secondly, there was additional work on understanding and characterizing solvent/vapor induced crystallized polycarbonate, a polymer only utilized practically in its amorphous state. The combination of vapor-induced crystallization followed by annealing produced a polycarbonate with a melting point of 317 Degrees Celsius. Extrapolation via the Hoffman-Weeks method predicted an equilibrium melting point of 335 degrees C.

One pair of studies cited poly (butylene terephthalate) (PBT) and Poly (ethylene terephthalate) (PET) both blended in binary systems with a Bisphenol A based polyarylate. In the absence of interchange reactions the PBT was miscible with the polyarylate in the amorphous phases while the PET was immiscible. This was confirmed via the presence or absence of two glass transition temperatures. Interestingly, the polyarylate promoted higher crystallinity in the PET perhaps serving as a nucleating agent at concentrations of about 25%. Upon interchange reaction to a sufficient extent random copolymers were formed for each binary pair preventing crystallization of either the PET or the PBT.

The binary blend of the polyester poly (caprolactone) and bisphenol A polycarbonate yielded quite novel results. The binary blend exhibited a single Tg indicating amorphous phase miscibility. However, the Chi parameter calculated via melting point depression from the Flory Huggins equation yielded a slightly positive value. Selective solvent extraction followed by spectroscopy evaluation showed the presence of thermooxidative branching but the absence of transesterification.

Yokoya and Porter completed a practical coating study of PET and a copolymer of PET with isophthalic acid and their relative adhesion to Chromium-coated steel. The copolymer was more amorphous and additional chain mobility promoted better surface adhesion compared to PET. Overheating of either material led to embrittlement attributed to increased crystallinity and degradation of the ester linkage. This type of work that bridged the gap between polymer science and practical applications was a key talent of Professor Porter.

Brady and Porter studied the adsorption and crystallization of polycarbonate in composites with carbon fibers. Interfacial adhesion and crystallization were affected by the time the polymer melt was in contact with the carbon fiber. This was also studied in aromatic polyketone composites and will be covered in that section.

13. Polystyrene, Acrylics, and Styrene Copolymers

Jenkins and Porter utilized light scattering and GPC to determine molecular weight and intrinsic viscosity of isotactic and syndiotactic poly (methyl methacrylate) (PMMA) fractions. The unperturbed dimensions were obtained via viscosity plots. They concluded that isotactic poly(methyl methacrylate) was about 30% more extended than the syndiotactic confirmation of PMMA. This paper also included a thorough review of alternative statistical treatments to the Mark-Houwink relation and found quite similar results.

Several studies including those by Lucas were completed on the photooxidation of polystyrene and it was discovered that polystyrene exhibited surface autoinhibition at the later stages of oxidation at some UV wavelengths. The degradation mechanisms were elucidated and included the formation of peroxides, carbenes, vinyl groups and evolution of benzoic acid.

Dynamic mechanical properties of styrene-butadiene-styrene tri-block copolymers were determined to be a function of the interfacial thickness at the styrene matrix interface and also to the relative dispersion of styrene in the butadiene matrix. Studies at various frequencies led to elucidation of the finer details of the tri-block copolymer macroscopic morphology.

Wang and Porter studied the differential orientation after solid-state coextrusion of polystyrene and poly (2,6-Dimethyl-1,4-phenylene Oxide) (PPO) in their miscible blends via birefringence, infrared Dichroism, and density measurements. Both polymers decreased in orientation with increasing PPO content and upon annealing the PPO chains disorient more slowly than the PS chains. It would be interesting to apply this method to LCP/low molecular weight liquid crystal binary blends in order to follow differential orientation behavior.

14. Aromatic Polyketones

Porter and coworkers studied the poly (aryl ether ketone), poly (oxy-1,4-phenyleneoxy-1,4-phenylenecarbonyl-1,4-phenylene) known commercially as PEEK. They studied multiple melting peaks, crystallization behavior in the presence of fibrous reinforcement, sulfonated and nitrated PEEK, subsequent evaluation of physical properties and changes in phase transitions, the effect of these functionalities on compatibility with polyimides, and explored ultimate PEEK properties upon solid state extrusion.

Lee and Porter studied the crystallization of PEEK/carbon fiber composites. Heating the PEEK well above its melt point at longer times reduced homogeneous nucleation sites and yielded a propensity to heterogeneously crystallize on the carbon fibers. This led to significant improvements in transverse composite tensile strength, an indication of improved adhesion at the matrix/fiber interface.

Porter, Karcha, Shibuya, and Chen extended earlier work on miscibility of PEEK with poly etherimide to include miscibility studies with sulfonated PEEK and reported thermodynamic miscibility of sulfonated PEEK with both the polyetherimide (Ultem) as well as with a polyamideimide (Torlon). These studies are significant since PEEK, Ultem, and Torlon are commercially successful polymers today. Sulfonation and nitration reaction rates were studied and identified as routes to increase the glass transition temperature. Modern applications use this work to etch the PEEK surface to improve surface adhesion.

15. Polyamides

Zachariades, Kanamoto, and Porter proposed a new concept for the reversible plasticization of polyamides with ammonia, with particular work on Nylon 6 and 11. Nylons up to that point had been hard to solid state extrude to high extrusion draw ratios due to hydrogen bonding between amide groups. The ammonia intercalates between amide groups in the amorphous phase that enabled maximum physical properties not seen before. An important aspect of this approach was the removal of ammonia after processing. This yielded a final product that was not plasticized therefore yielding improved tensile properties versus traditional plasticized systems. The equilibrium absorption of ammonia is proportional to the amide group concentration in the particular polyamide. Chuah and Lee extended this reversible plasticizer concept to the use of Iodine that allowed intercalation within both the amorphous and crystalline phases. They also reported some quite unique crystalline morphologies, initial amorphous material that was subsequently strain induced crystallized.

Shimada and Perkins extended typical non-plasticized solid-state extrusion studies to Nylon 6, 66, 11, and 12. This confirmed some of the unique properties of the plasticized Nylon studies. Mathias and others worked with Porter to more fully characterize several polyamide materials particularly with the use of ^{15}N-NMR.

This worked foreshadowed the commercialization of Nylon 12 and Nylon 12 copolymers as important materials used as catheter jackets in the medical industry. Perhaps the reversible plasticization technique will find unique medical applications.

16. Fluoropolymers

Mead, Zachariades, Shimada, and Porter determined that poly (vinylidene fluoride) (PVDF) converted from the alpha to beta crystal form upon solid-state extrusion. This is significant in that the beta form of PVDF is piezoelectric and has practical utility.

Porter, Ting and George along with Porter's former colleagues at Chevron, Barrall II and Cantow studied the high shear viscosity behavior for perfluoropolyether fluids by two different techniques. The first involved direct measurement by a high shear viscometer, and the second utilized the time-temperature superposition principle to establish master curves from viscosity measurements at low shear rates and temperature. The two methods were comparable. These fluids are ideal for lubricating disc drives in computers. They do not break down in MW upon high shear, are lubricious, and have a high density.

Porter collaborated with a team of leading scientists in Japan (Kanamoto, Okuyama, Wantanabe, Uehara, Ito, Jounai, and Endo) to study the polymer morphology of poly (tetrafluoroethylene) (PTFE) before and after solid-state extrusion. Maximum draw ratios were achieved at temperatures approaching the PTFE melting point and yielded materials with mechanical properties 10X that of any commercially available materials.

They reported that PTFE reactor powder is virtually 100% crystalline and that crystal-crystal phase transitions present at 19°C and 30°C enable PTFE to be solid state extruded and drawn in a second stage even at temperatures 200°C below the crystalline melting point of 340°C. Interesting trends were noted in mechanical properties and thermal transition behavior as a function of draw ratio, maximum properties and crystallinity obtained at intermediate draw ratio. This can be attributed to an initial unwinding of crystals at low draw ratio followed by subsequent unwinding and recrystallization at intermediate draw rations and finally degradation and breakage of fibrils beyond a critical draw ratio.

Professor Porter would be happy to know that there are now large commercially successful companies based on the solid-state extrusion and stretching of PTFE and other polymers. In graduate school Porter discovered the polymerization of PTFE by a new route and in the last year of his life co-authored key papers on the finer details of PTFE morphology.

IV. ACKNOWLEDGEMENTS

To Professor Roger S. Porter for honoring us with his presence, for mentoring us, for advising us, for teaching us, for preparing us, and for being there when we needed you.

We miss you!

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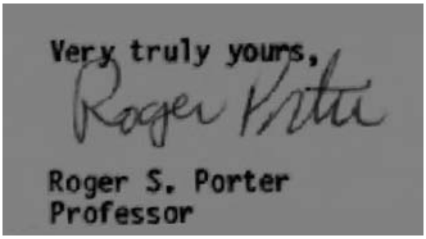
reading and helping to summarize the publications. It should not be surprising that this project has helped us do our research more effectively. Lastly, to Professor Darryl DesMarteau for providing information on George Cady and Joel Hildebrand.

Lastly, I saved a copy of Professor Porter's characteristic signature.


Very truly yours,
Roger Porter

Roger S. Porter
Professor

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