Highly Filled Materials Institute

at Stevens

Winter 2007

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A Business Resource at



19th ANNIVERSARY ISSUE

From the Director

Dear Friends,

In May 2008, it will be the 19th anniversary of the founding of Highly Filled Materials Institute at Stevens. The activities of our Center have significantly gone beyond the boundaries

of the highly filled materials area, as reflected in the listing of our current projects, recent papers and publications. The ability to work in multiple areas (with over 120 funded projects) has enabled us to apply the software and hardware tools that constitute our core competencies to multiple industries and to create real synergies across disciplines.

All of us at HfMI thank you for your support and look forward to continuing to work with you on your important projects.

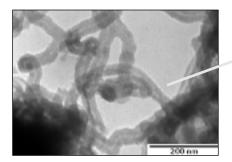
Dilhan M. Kalyon

Recent projects and capabilities

During 2007, we have completed a number of projects funded by various corporate and Government sources in myriad areas covering the nanocomposite, pharmaceutical, biomedical and tissue engineering, personal care, ceramic, energetic, packaging of electronic materials and environmental fields. We are also in the process of starting a number of new projects including one in the area of renewable energy.

Nanocomposites

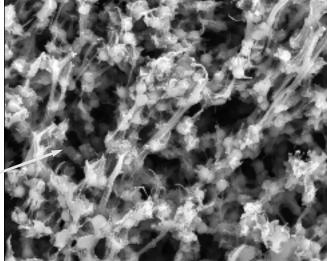
We have carried out multiple investigations in the area of the compounding and processing of various types of nanoparticles including the intercalation and exfoliation of nanoclays, incorporation of multi-walled C nanotubes into pyroelectric polymers, shear induced crystallization with nanoparticles, generation of nanocomposite membranes, and nanoenergetics. Our collaboration with Prof. Frank Fisher and his group through the mechanism of co-advicing of graduate students



has produced important results in the area of C nanotubes.

Some of our recent publications and manuscripts in these areas are included in our list of publications on page 4.

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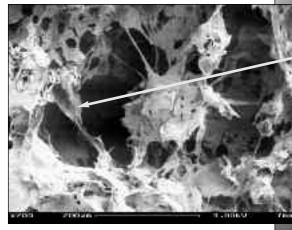
Compounding of C nanotubes into pyroelectric PVDF for generation of membranes with controlled porosity

Recent projects and capabilities continued from page 1

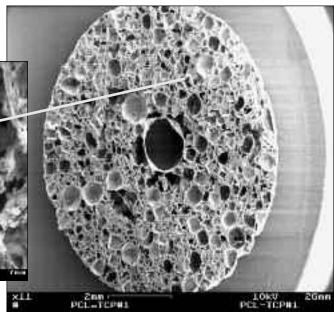
Medical Polymers and Tissue Engineering

We currently have three PhD projects being carried out in collaboration with Biomedical Engineering Professors Yu, Wang and Ritter of Stevens. We are especially pleased that

we have been able to generate myriad materials and processes, including the polymerization of various biodegradable polymers (for example, poly(lactic acid)), synthesis of nano hydroxyapatite and new processes for the processing of biodegradable poly-



mers into scaffolds that can be used in tissue engineering. Some of the recent publications and presentations are listed on page 4.



Tissue engineering scaffold with human osteoblast cells [inset].

Catalytic converters

We are in the first stage of a multi-year program funded by Corning Corporation in rheology and processing of complex fluids utilized in the area of catalytic converters world-wide. The project has the components of rheological characterization using multiple rheometers, mathematical modeling and validation at large manufacturing scale and process control using model-based control algorithms.

Prof. Kalyon has also given a short course in the area of rheology of complex fluids at Corning to 40 participants through the Professional Advancement Department of Stevens.

Energetic Materials

We have continued to collaborate with the Indian Head Division of NSWC and ARDEC in the area of processing of energetic materials. Some of the research findings were compiled into special issues of the Journal of Energetic Materials which were dedicated to the memory of our friend (and Advisory Board Member) Dr. Richard S. Miller, whom we have lost. We are extremely pleased that the issues could be published within two years of the passing away of Dr. Miller on January 7, 2006 and we especially thank Dr. Paul Marinkas for his support and encouragement of the special issues.

The list of our papers related to our energetics research, is included on page 4.

Electronics

The key issues in this field continue to be the ability to mix multiple ingredients efficiently and in a reproducible manner, the rheology control and tight constraints on continuous processing and the optimization of processing conditions and geometries.

We have been funded by Bergquist Corporation of Minnesota in the area of rheology, modeling and processing of conductive highly filled suspensions.

Some of our earlier work carried out with IBM in the development of suspensions for electronics packaging has been presented at the International Microelectronics and Packaging Society meeting in Scottsdale, Arizona.

Environmental

We have continued to collaborate with the Environmental Engineering Center at Stevens, especially in the area of treatment of Cr^{+6} contaminated residues and soil. A patent application has been filed.

Personal Care Products and their Characterization and Processing

We have continued to carry out research, principally through the funding of P&G, in the area of rheological characterization, processing and structure/rheological behavior control of complex fluids, including multilamellar vesicles, hydrogels and suspensions targeted for the personal care area.

When are our capabilities most useful?

Generally, we offer our capabilities when one or more of the following factors apply:

- 1. A product exhibits a complex and sensitive structure, which affects the ultimate properties of the product on one hand and is affected significantly by the thermo-mechanical history that the material experiences during processing. For such complexity, the trial and error methodologies cannot provide the requisite understanding. We have worked on the development of various such structured products, including many in the personal care, battery, energy and biomedical industries.
- **2.** The product is processed at rates that are very high and a detailed understanding of the process and the material that we generate provides savings in capital and operating

Current staff news

Dr. Mike Goekbora joined HfMI as a Research Scientist in the area of Biomaterials and Medical Devices. Mike started his career in the polymer processing and compounding area, has worked with major polyolefin companies and for the last twelve years he has been involved in the development of bioresorbable medical implants, including orthopedic and cardiovascular areas.

Ms. Seher Ozkan is a 2007-2008 recipient of a Merck Research Laboratories Fellowship in Chemistry, Pharmaceutical Science, Material Science, and Engineering and will spend three months at Merck as part of her PhD training. Congratulations!

Past staff and alumni news

Dr. Mel Allende is a researcher with P&G in Cincinnati, Ohio.

Dr. Birnur Aral is the Health, Beauty, and Environmental Sciences Director at the Good Housekeeping Research Institute in New York, NY.

Dr. Nezahat Boz is an assistant professor of chemical engineering at Kocaeli University.

Ms. Elif Burcoglu is with J&J.

Mr. Dharmesh Dalwadi is a process engineer with Jacobs Engineering in Houston, Texas.

Mr. Emre Demirkol has been working as a process engineer at Foster Wheeler Italiana in Milan.

Dr. Tom Fiske is a senior analyst, Enterprise Applications, at ARC Advisory Group.

Mr. Jason Garrow is the lead systems engineer for countermeasure systems at General Dynamics in Burlington, Vermont.

Dr. Hasan Gocmez is an associate professor at Dumlupinar University. His current work includes the synthesis, dispersion and stability of nanoparticles, and molecular modeling.

Dr. Hal Gokturk is a research scientist with Panasonic Electric Works Laboratory of America in San Jose, CA, where he is focusing on computer aided design of nanodevices using quantum chemistry tools. Our congratulations go to Hal who recently got married.

Dr. Alex Gotsis is a professor at the Technical University of Crete in Chania, Greece, which he joined upon spending 12 years in Delft

costs and reduces time to market. For example, in the area of extrusion we have optimized geometries and processing conditions for production rates that were as high as 10,000 lb/hour per extruder.

- **3.** The product involves very expensive ingredients so that raw material costs are important even at the lowest possible processing scale. Examples of such cases include some medicinal drugs which can cost as high as a million dollars a kilo but still need to be incorporated into various polymeric binders or some types of C nanotubes.
- **4.** The process and the materials are hazardous and requires the temperature and pressure conditions of the process need to be known apriori. This happens frequently in the pharmaceutical and energetics industries.

Dr. Moinuddin Malik has collaborated on two publications in the area of granular flows:

- A. Mujumdar, M. Horio, P. S. Robi, R. Swarnkar, and M. Malik, "Experimental Investigation and Validation of Mixing and Segregation Behavior of Granular Flow in a Sectorial Container," *Journal of Chemical Engineering of Japan, 40,* 652-657 (2007).
- A. Mujumdar, P. S. Robi, M. Malik, and M. Horio, "Artificial Neural Network (ANN) Model for Prediction of Mixing Behavior of Granular Flows," International Journal for Computational Methods in Engineering Science and Mechanics, 8, 149-158 (2007).

Dr. D. Kalyon has been elected a Fellow of American Institute of Chemical Engineers.

University of Technology. His research focus is on the structure and property relationships of polymers/composites.

Dr. Luisa Lao has passed the patent bar exam and is currently a patent examiner of US PTO.

Dr. Guangyu Lu is Manager of the Plastics Process Development group at Teleflex Medical in Pennsylvania

Dr. Ugursoy Olgun is an assistant professor at Sakarya University.

Mr. Paul Redner has recently completed his MBA degree at NYU and is working at the Picatinny Arsenal of ARDEC/US Army.

Mr. Harish Sangani is with PolyClean Technologies, Inc., a company which he has started. PolyClean provides customized solutions in the improvement of cleanliness and purity of virgin polymers used in critical applications and recovery/recycle of high value polymers.

Dr. John Suwardie is a research engineer at Axel Plastics Research Laboratories in Queens, New York.

Dr. Hansong Tang is an assistant professor at City College of the City University of New York.

Dr. Alan Wagner is the Director of Technology for the Polymer Additive business of Cytec Industries Inc. at Stamford, CT.

Dr. Piraye Yaras is the Technical Manager-TPE of Solvay Engineering in Arlington, TX.

Dr. Jeong Su Yu is Director and Vice-President of I & E Materials Research Institute at LG Chem Research Park in Korea.

Contributions to the literature and presentations

(since the last newsletter in 2006)

- D. M. Kalyon and H. Tang, "Inverse problem solution of squeeze flow for parameters of generalized Newtonian fluid and wall slip," *Journal of Non-Newtonian Fluid Mechanics*, 143, 133-140 (2007).
- S. Ozkan, H. Gevgilili, D.M. Kalyon, J. Kowalczyk, and M. Mezger, "Twin screw extrusion of nano-alumina based simulants," *Journal of Energetics Materials*, 25, 3, 173-201 (2007).
- E. Demirkol and D. M. Kalyon, "Batch and continuous processing of polymer layered organoclay nanocomposites," *Journal of Applied Polymer Science*, 104, 1391-1398 (2007).
- D. M. Kalyon and M. Malik, "An integrated approach for numerical analysis of coupled flow and heat transfer in co-rotating twin screw extruders," *International Polymer Processing*, 22, 293-302 (2007).
- M. Allende, D. Fair, D. M. Kalyon, D. Chiu and S. Moy, "Development of particle concentration distributions and burn rate gradients upon shear-induced particle migration during processing," *Journal of Energetic Materials*, 25, 49- 67 (2007).
- J. E. Kowalczyk, M. Malik, D. M. Kalyon, H. Gevgilili, D. F. Fair, M. Mezger and M. Fair, "Safety in Design and Manufacturing of Extruders," *Journal of Energetics Materials*, 25, 247-271 (2007).
- S. Ozkan, D. Kalyon and X. Yu, "Surface patterning of poly (L-lactide) upon melt processing: in vitro culturing of fibroblasts and osteoblasts on surfaces ranging from highly crystalline with spherulitic protrusions to amorphous with nanoscale indentations" accepted to appear in Journal of Biomedical Materials Research A (2007).
- H. Gevgilili, D. Kalyon and A. Shah, "Processing in continuous shear roll mills," accepted to appear in Journal of Energetic Materials (2007).
- 9. G. Mago, J. A. Dutreuil, F.T. Fisher, and D.M. Kalyon "Structural formation in poly(butylene terephthalate) and PBT nanocomposites during uniaxial deformation," *Proceedings of the ASME International Mechanical Engineering Conference and Exposition, Seattle, WA* (2007).
- G. Mago, D. M. Kalyon and F. T. Fisher, "Membranes of Polyvinylidene fluoride (PVDF) and PVDF Nanocomposites with Carbon Nanotubes via Immersion Precipitation," submitted to Journal of Nanomaterials (2007).
- C. Erisken, D. Kalyon and H. Wang, "A hybrid twin screw extrusion/ electrospinning method to process nanoparticle-incorporated electrospun nanofibers" submitted to Nanotechnology, 2007.
- G. Mago, F. T. Fisher and D. Kalyon, "Effect of shearing on crystallization behavior and morphology of PVDF nanocomposites," submitted to Journal of Nanoscience and Nanotechnology (2007).
- D. M. Kalyon and H. Tang, "Unsteady circular tube flow of compressible polymeric liquids subject to pressure-dependent wall slip," accepted to appear in J. Rheology (2007).
- 14. H. Tang and D. M. Kalyon, "Time-dependent tube flow of compressible suspensions subject to pressure dependent wall slip: Ramifications on development of flow instabilities," submitted to J. Non-Newtonian Fluid Mechanics (2007).
- 15. D. Kalyon and H. Tang, "Development of flow instabilities associated with microchannel flows of polymers and polymeric suspensions: Experimental and modeling studies," 60th Annual Meeting of American Physical Society Meeting, Division of Fluid Dynamics, Salt Lake City, Utah, November 18, 2007.
- D. M. Kalyon, D. Dalwadi, M. Erol, E. Birinci and C. Tsenoglu "Rheological Behavior of Concentrated Suspensions as affected by the Dynamics of the Mixing Process," *Rheologica Acta*, 45, 641-658 (2006).
- N. Degirmenbasi, D. M. Kalyon and E. Birinci, "Biocomposites of Nanohydroxyapatite with Collagen and Poly(vinyl alcohol)," Colloids and Surfaces B Biointerfaces, 48, 42- 49 (2006).
- E. Birinci and D. M. Kalyon, "Development of extrudate distortions in poly(dimethyl siloxane) and its suspensions with rigid particles," *J. Rheology*, 50, 3, 313-326 (2006).
- Z. Peralta-Inga, N. Degirmenbasi, U. Olgun, H. Gocmez and D. M. Kalyon, "Recrystallization of CL-20 and HNFX from Solution for rigorous Control of the Polymorph type: Part I, Mathematical Modeling using Molecular Dynamics Method," *Journal of Energetic Materials*, 24, 69-101 (2006).
- N. Degirmenbasi, Z. Peralta-Inga, U. Olgun, H. Gocmez and D. M. Kalyon, "Recrystallization of CL-20 and HNFX from Solution for rigorous Control of the Polymorph type: Part II, Experimental Studies," *Journal of Energetic Materials*, 24, 103-139 (2006).
- R. Schefflan, S. Kovenklioglu, D. Kalyon, M. Mezger and M. Leng, "Formation of aluminum nanoparticles upon condensation from vapor phase," *Journal of Energetic Materials*, 24, 141-156 (2006).
- 22. C. Feger, M. McGlashan-Powell, I. Nnebe, and D. M. Kalyon, "Rheology and stability of highly filled thermal pastes," *Proceedings of the International Microelectronics and Packaging Society, Scottsdale, Arizona, March* 23 (2006).

- R. Schefflan, S. Kovenklioglu, D. Kalyon, P. Redner and E. Heider, "Mathematical model for a fed-batch process for energetic crystals to achieve targeted size distributions," *Journal of Energetic Materials*, 24, 157-172 (2006).
- D. Kalyon, H. Gevgilili, J. Kowalczyk, S. Prickett and C. Murphy, "Use of adjustable-gap on-line and off-line slit rheometers for the characterization of the wall slip and shear viscosity," *Journal of Energetic Materials*, 24, 175-193 (2006).
- G. Mago, F.T. Fisher, and D.M. Kalyon, "Effect of shearing on the crystallization behavior of poly(butylene terephthalate) and PBT nanocomposites," *Proceedings* of ASME International Mechanical Engineering Conference and Exposition (IMECE), November 5-10, Chicago, IL (2006).
- D. Kalyon, H. Tang and B. Karuv, "Squeeze flow rheometry," Journal of Energetic Materials, 24, 195-202 (2006).
- D. Kalyon, "An overview of the rheological behavior and ramifications on safety and product quality," *Journal of Energetic Materials*, 24, 213-245 (2006).
- E. Birinci, H. Gevgilili, D. Kalyon, B. Greenberg, D. Fair and A. Perich, "Rheological characterization of gels," *Journal of Energetic Materials*, 24, 247-269 (2006).
- 29. D. Kalyon, S. Ozkan, M. Malik, J. Kowalczyk and M. Mezger, "Twin screw extrusion of nanoenergetics with processing geometries that are tunable to the targeted thermo-mechanical history," Annual National Meeting of the American Institute of Chemical Engineers, Salt Lake City, Utah, November 8, 2007.
- 30. S. Ozkan, D. Kalyon and X. Yu, "Twin screw extrusion processing and shaping of biodegradable scaffolds with controlled morphologies for tissue engineering applications," Annual National Meeting of the American Institute of Chemical Engineers, Salt Lake City, Utah, November 7, 2007.
- 31. C. Erisken, D. Kalyon and H. Wang, "Nanocomposites for tissue engineering applications engineered using a novel twin screw extrusion and electrospinning process (SEEP-Screw Extrusion with Electrospinning," Annual National Meeting of the American Institute of Chemical Engineers, Salt Lake City, Utah, November 8, 2007.
- 32. G. Mago, F. T. Fisher, D. M. Kalyon, "Nanoparticle-enhanced processing-induced crystallization of PVDF and PVDF nanocomposites," 44th Annual meeting of the Society of Engineering Science, Texas A&M University, College Station, TX, October 21-24, 2007.
- G. Mago, F.T. Fisher, D. M. Kalyon, "Effect of shearing on crystallization behavior and morphology of PVDF nanocomposites," Virtual Conference on Nanoscale Science and Technology (VC-NST), Fayetteville, Arkansas, October 21-25, 2007.
- 34. D. M. Kalyon, "Continuous processing and shaping of energetic materials: Challenges with viscoplasticity, migration effects and wall slip," Texas Tech University Chemical Engineering Department Seminar Series: New frontiers in chemical engineering, Lubbock, Texas, September 14, 2007.
- D. Kalyon, "Rheology and processing of highly filled suspensions," Merck Company, West Point, PA, January 11, 2007.
- J. Kowalczyk, D. Kalyon, H. Gevgilili, "Flexible manufacturing technologies for the processing of energetic formulations," 2006 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November 16, 2006.
- D. Kalyon, H. Gevgilili, "Shear roll milling," 2006 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November 16, 2006.
- D. Kalyon, H. Tang, H. Gevgilili, C. Demir and J. Kowalczyk, "Squeeze flow rheometry for rheological characterization of energetic formulations," 2006 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November 16, 2006.
- S. Ozkan, D. Kalyon, J. Kowalczyk, H. Gevgilili, M. Mezger and C. Demir, "Processing of nanoenergetics," 2006 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November 16, 2006.
- D. Kalyon, H. Gevgilili, J. Kowalczyk, S. Prickett and C. Murphy, "Adjustable gap rheometry for the rheological characterization of energetic formulations," 2006 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, San Francisco, CA, November 16, 2006.
- 41. C. Feger, M. McGlashan-Powell, I. Nnebe and D. Kalyon, "Rheology and stability of highly filled thermal pastes," *IMAPS International conference on Device Packaging in conjunction with Global business Council Spring conference in Scottsdale, Arizona,* March 21, 2006.
- D. Kalyon, "Challenges associated with the processing of concentrated suspensions," *Bergquist Company, St. Paul, Minnesota,* July 27, 2006.
- 43. D. Kalyon and S. Prickett, "Rheological characterization of Gun Propellant Formulations for Co-Extrusion Applications," *Joint Army, Navy, NASA, Air Force* Workshop on Challenges for Fast-Core/Layered gun Propellants Joint Workshop, Aberdeen Proving Ground, Maryland, May 11, 2006.
- 44. D. Kalyon, "Issues, Challenges and Opportunities in Co-Extrusion Technologies," Joint Army, Navy, NASA, Air Force Workshop on Challenges for Fast-Core/Layered gun Propellants Joint Workshop, Aberdeen Proving Ground, Maryland, May 11, 2006.