

17th ANNIVERSARY ISSUE

From the Director

Dear Friends,

In 2006, HfMI will be celebrating the 17th anniversary of its founding at Stevens Institute of Technology. This newsletter will give you a quick update on what we have done in 2005 and the new research areas that we will venture into during 2006, including



plans to increase our efforts in the general area of rheology and processing of complex fluids which exhibit strong processing, structure, rheology, property, and interrelationships.

Once again, all of us at HfMI thank you for your continuing support and wish you a great new year.

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Recent projects and capabilities

During 2005, we have completed a number of projects funded by various corporate and government sources in myriad areas.

Soaps and surfactants

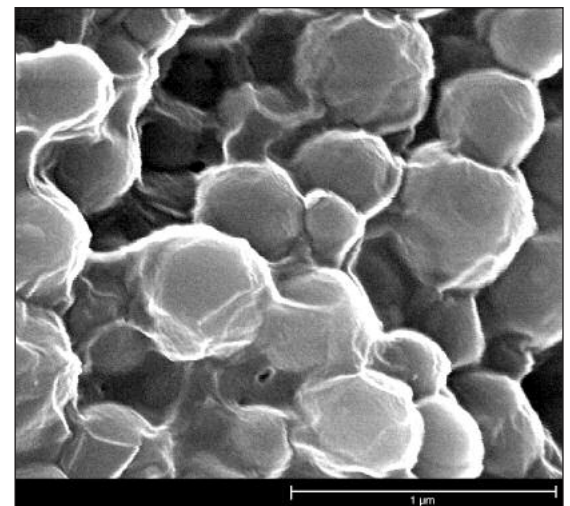
We have completed a number of major projects in this area, especially focusing on the continuous processing of various structured complex fluids. The results of our research into self-assembly and structuring of concentrated surfactants under pressurization and shear will be reported in collaboration with Procter & Gamble researchers.

Some interesting findings on the wall slip behavior of bar soaps were published jointly with Unilever researchers:

D. Kalyon, H. Gevgilili, R. Yazici, A. Post and G. McFann, "Flow and Structure Development Behavior of Bar Soaps Containing Synthetic Detergent," *Rheologica Acta*, 43, 396-405 (2004).

Particle size distribution analysis

We also expect to make extensive use of our technology base for the determination of the particle size distribution of particles and nanoparticles on the basis of our proprietary technology summarized in our patent:



Shear induced self-assembly based formation of multilamellar vesicles. (Photo by Rick Bacon, P&G)

R. Yazici and D. M. Kalyon, "Method and Apparatus for x-ray Analysis of Particle Size," U.S. Patent, #6,751,287 issued on June 15, 2004.

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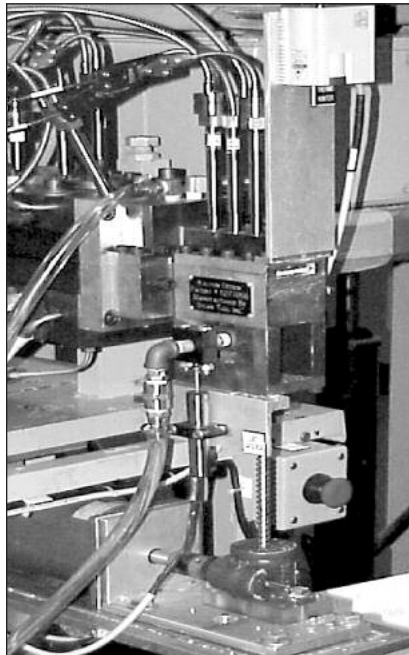
Recent projects and capabilities continued from page 1

Energetics manufacture: NSWC/Indian Head

During the last two years, we have continued to collaborate with the Indian Head Division of NSWC in the areas of processing of double base propellants and co-extruded grains which have resulted in the following papers:

C. Gonsalves, R. Muscato, S. Prickett, C. Cline, D. Kalyon, M. Malik, J. Kowalczyk, **"Development of Co-extrusion Process for Propellant Manufacture,"** 2005 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, Cincinnati, OH, November 3, 2005.

S. Prickett, W. G. Thomas, C. Raddack, D. Kalyon, M. Malik, and J. Kowalczyk, **"Twin Screw Extrusion Processing of Double-base Propellant,"** 2005 Annual Meeting of American Institute of Chemical Engineers Annual Meeting, Cincinnati, OH, November 3, 2005.



In-line adjustable gap rheometer (Stevens patent US # 5,277,058) licensed to MPR Inc. for commercialization

HfMI has made many important contributions to the research and manufacture of energetic materials at the Indian Head Division of Naval Surface Warfare Center. Energetic materials involve very high degrees of solid fill and the problems associated with such high degrees of solid fill. HfMI has directly contributed to the area of the safe processing of energetic materials at Indian Head and was involved in the design and manufacture of hardware, including dies, selection of geometries and operating conditions for the manufacturing and processing of many energetic formulations and the testing of extruded grains for concentration homogeneity and migration effects.

*Constance Murphy
Naval Surface Center, Indian Head
Division, Indian Head, Maryland*

Processing of energetics: US Army ARDEC

Picatiny ARDEC: Our collaboration with Picatiny ARDEC continues to be strong, with two current on-going projects in the areas of generation of functionally graded propellants and the continuous manufacturing of PAX 3 propellants. A number of joint papers were presented at various meetings and one joint patent application was filed on the manufacture of novel functionally-graded propellants:

D. Fair, D. Kalyon, S. Moy and L. Manole, **"Cross-sectional Functionally-graded Propellants and Method of Manufacture,"** US application filed on February 11, 2005 (application number 10/906274).

Since 1985 ARDEC has partnered with Stevens Institute of Technology. Stevens has introduced new devices for rheological analysis such as the squeeze flow and the adjustable-gap slit rheometers and new methods including rheological data interpretation and die/extruder modeling into the industry. Research into the quality of mixing has provided insight into how process affects sensitivity and performance. The black art previously associated with energetic manufacture has been replaced with science through Stevens' academic leadership. The insight into processing technology has inspired new concepts and technologies in energetics. This has been a progressive partnership between Stevens and ARDEC, with each discovery leading to new opportunities.

*David F. Fair
United States Army Armaments Research,
Development and Engineering Center (ARDEC)*

Electronics packaging

We have completed two projects for two major corporations and are moving in the direction of setting up new projects, which will introduce novel processing capabilities to the electronics packaging industry. Some of our results in this area can be found in a joint paper with IBM researchers:

C. Feger, J. Gelorme, M. McGlashan-Powell, and D. M. Kalyon, **"Mixing, Rheology, and Stability of Highly Filled Thermal Pastes,"** *IBM Journal of Research and Development*, 49, 4/5 699-707 (2005).

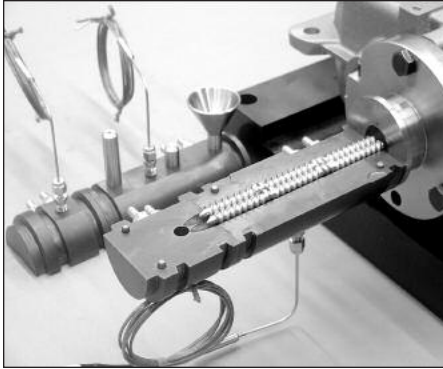
Environmental

Our collaboration with the Environmental Engineering Center at Stevens, funded by Honeywell Corporation, gave rise to a new technology and a patent filing in the area of treatment of Cr⁺⁶ contaminated residues and soils:

M. Wazne, D. Dermatas, M. Kauris, X. Meng, J. Morris and D. Kalyon, **"Method of Treatment, Stabilization, and Heave Control for Chromate Ore Processing Residues (COPR) and Chromium Contaminated Soils,"** US provisional patent filed on March 31, 2005.

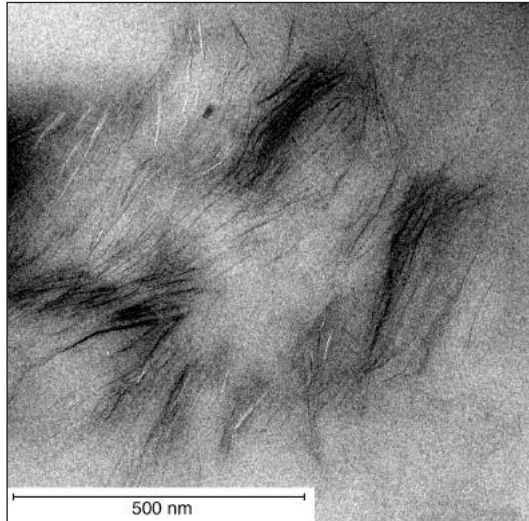
Nanocomposites

Our research in the area of incorporation of nanoparticles into polymeric binders or gels has received the support of Material Processing & Research, Inc. (MPR) of Hackensack, NJ, by giving us access to the "smallest twin screw extruder" in the World (7.5 mm), which was developed with US Army ARDEC funding to MPR. This extruder has allowed us to process nanocomposites at rates which are as low as 10 g/hr.



MPR 7.5 mm twin screw extruder used in the processing of nanocomposites

TEM micrograph of organoclays melt compounded with maleic anhydride-co-polypropylene



HfMI has been a leader in developing state of the art rheological characterization equipment and materials characterization techniques and mathematical models that allow for realistic elucidation of the interaction between the process material and the processing hardware. Physical characteristics of this interaction, such as pressurization rate and thermal excursions can now be quantified before any die or extruder hardware is built. These capabilities allow significant cost and time savings.

*James E. Kowalczyk
VP Technology and Development,
Material Processing & Research*

Medical plastics and tissue engineering

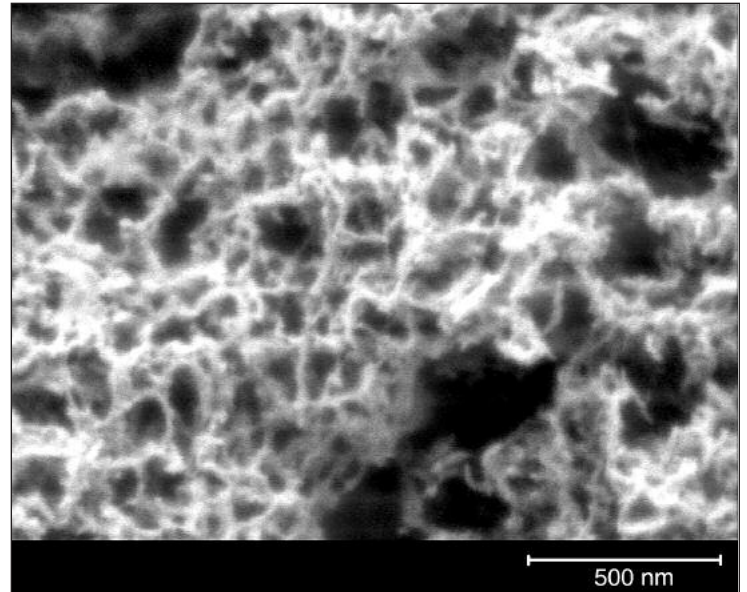
A PhD project in the area of filled medical polymers was carried out in collaboration with Becton and Dickinson together with Professors I. Yilgor and E. Yilgor of Koc University of Istanbul. The use of resins without any processing modifiers in the medical field renders such studies particularly difficult to carry out. The study resulted in the following publications:

G. Lu, D. Kalyon, I. Yilgor and E. Yilgor, "Rheology and Processing of BaSO₄ Filled Medical-grade Thermoplastic Polyurethane," *Polymer Engineering and Science*, 44, 10, 1941-1948 (2004).

D. Kalyon, G. Lu, I. Yilgor and E. Yilgor, "Rheology and Extrusion of Medical-grade Thermoplastic Polyurethane," *Polymer and Engineering Science*, 43, 12, 1863-1877 (2003).

We have initiated collaboration in the area of development of biomedical devices and materials with the Integrated Manufacturing Technology Institute at London/Ontario through the leadership of Dr. Gordon Campbell. Shortly, we will publish jointly in the area of PVA gels used in heart valve applications.

During the last two years we have carried out multiple projects in the areas of processing of nanocomposites including nanoclays upon intercalation and exfoliation, incorporation of nanoparticles of alumina into hydrogels, *in situ* synthesis of nanoparticles of hydroxyapatite



SEM micrograph of porous poly(vinyl alcohol), collagen and hydroxyapatite biocomposite manufactured at HfMI for tissue engineering applications

in collagen and hydrogels with the ultimate objective of gaining a foothold in the area of tissue engineering using polymers and nanoparticles. We currently have three PhD students being co-supervised by Professors H. Wang and X. Yu of Biomedical Engineering and Prof. F. Fisher of Mechanical Engineering in the area of tissue engineering.

Contributions to the literature (2005)

1. C. Feger, J. D. Gelorme, M. McGlashan-Powell and D. Kalyon, "Mixing, Rheology and Stability of Highly Filled Thermal Pastes," *IBM Journal of Research and Development*, 49, 4/5, 699-707 (2005).
2. D. Kalyon, "Letter to the Editor: Comments on the Use of Rheometers with Rough Surfaces or Surfaces with Protrusions," *J. Rheology*, 49, 5, 1153-1155 (2005).
3. D. Kalyon, "Apparent Slip and Viscoplasticity of Concentrated Suspensions," *J. Rheology*, 49, 3, 621-640 (2005).
4. R. Yazici and D. Kalyon, "Microstrain and Defect Analysis of CL-20 Crystals by Novel X-ray Methods," *Journal of Energetic Materials*, 23, 43-58 (2005).
5. M. Erol and D. Kalyon, "Assessment of the Degree of Mixedness of Filled Polymers: Effects of Processing Histories in Batch Mixer and Co-Rotating and Counter-rotating Twin Screw Extruders," *International Polymer Processing*, 20, 228-237 (2005).
6. M. Malik and D. Kalyon, "Three-dimensional Finite Element Simulation of Processing of Generalized Newtonian Fluids in Counter-rotating and Tangential Twin Screw Extruder and Die Combination," *Int. Polym. Processing*, 20, 398-409 (2005).
7. D. 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, On-line*, 1435 (2005).
8. U. Olgun and D. Kalyon, "Use of Molecular Dynamics to Investigate Polymer Melt-metal Wall Interactions" *Polymer*, 46, 9423-9433 (2005).
9. E. Birinci and D. Kalyon, "Development of Extrudate Distortions in Poly(dimethyl Siloxane) and Its Suspensions with Rigid Particles," *submitted to J. Rheology* (2005).
10. H. Gevgilili and D. Kalyon, "Determination of the Parameters of Integral-type Constitutive Equations with Time-strain Separable Memory Functions" *submitted to Rheologica Acta* (2005).
11. D. Kalyon and H. Tang, "Analysis of Squeeze Flow and Inverse Problem Solution for Parameters of Generalized Newtonian Fluid and Wall Slip," *submitted to Journal of Non-Newtonian Fluid Mechanics*, (2005).
12. N. Degirmenbasi, D. Kalyon and E. Birinci, "Biocomposites of Nanohydroxyapatite with Collagen and Poly(vinyl alcohol)," *accepted to appear in Colloids and Surfaces B: Biointerfaces* (2005).
13. E. Demirkol and D. Kalyon, "Batch and Continuous Processing of Polymer-layered Organoclay Nanocomposites," *submitted to J. Applied Polymer Science* (2005).
14. Z. Peralta-Inga, N. Degirmenbasi, U. Olgun, H. Gocmez and D. Kalyon, "Recrystallization of CL-20 and HNFx from Solution for Rigorous Control of the Polymorph Type: Part I, Mathematical Modeling Using Ab Initio and Molecular Dynamics Methods," *submitted to J. Energetic Materials* (2005).
15. N. Degirmenbasi, Z. Peralta-Inga, U. Olgun, H. Gocmez and D. Kalyon, "Recrystallization of CL-20 and HNFx from Solution for Rigorous Control of the Polymorph Type: Part II, Experimental Studies," *submitted to J. Energetic Materials* (2005).
16. H. Gocmez, U. Olgun, Z. Peralta-Inga, N. Degirmenbasi and D. Kalyon, "Control of Crystallite Size and Shape for HNFx and CL-20 Upon Recrystallization from Solution: Mathematical Modeling and Experimental Studies," *submitted to J. Energetic Materials* (2005).

Theses (2005):

17. M. Erol, "Interrelations Between Degree of Mixedness, Rheology and Ultimate Properties of a Conductive Composite Processed Using Batch and Continuous Mixers," M. Eng. Thesis, Stevens Institute of Technology, Hoboken, NJ (2005).
18. S. Ozkan, "Rheology and Processing of Hydrogels and Their Suspensions with Alumina Nanoparticles," M. Eng. Thesis, Stevens Institute of Technology, Hoboken, NJ (2005).
19. D. Dalwadi, "Study of the Effect of Specific Energy Input on the Rheological and Thermal Properties of Graphite-filled Thermoplastic Elastomer," M. Eng. Thesis, Stevens Institute of Technology, Hoboken, NJ (2005).
20. E. Demirkol, "Processing and Rheological Behavior of Organomodified Clay/polymer Nanocomposites," M. Eng. Thesis, Stevens Institute of Technology, Hoboken, NJ (2005).
21. A. Mirza, "Coextrusion of Concentrated Suspensions," M. Eng. Thesis, Stevens Institute of Technology, Hoboken, NJ (2005).

Patents:

22. D. Fair, D. Kalyon, S. Moy and L. Manole, "Cross-sectional Functionally-graded Propellants and Method of Manufacture," US application filed on February 11, 2005 (application number 10/906274).
23. M. Wazne, D. Dermatas, M. Kauris, X. Meng, J. Morris and D. Kalyon, "Method of Treatment, Stabilization, and Heave Control for Chromate Ore Processing Residues (COPR) and Chromium Contaminated Soils," US provisional patent filed on March 31, (2005).
24. R. Yazici and D. M. Kalyon, "Method and Apparatus for X-ray Analysis of Particle Size," U.S. Patent, #6,751,287 issued on June 15, 2004.

We are deeply saddened to learn of the sudden and unexpected passing away of our Advisory Board Member and friend, Dr. Richard S. Miller, on January 7, 2006. We will sorely miss his generous friendship and wise counsel.