

21st ANNIVERSARY ISSUE

From the Director

Dear Friends,

It is a pleasure to write to you again in 2011, on the 21st anniversary of the founding of HfMI. During the last few years the activities of our research center have gone significantly beyond the boundaries of the highly filled materials. As you will see from our list of publications and presentations, we have applied our core competencies on mathematical modeling, rheology, mixing and processing, microstructural analysis and ultimate properties of polymeric suspensions and other complex fluids to very diverse applications, including biomaterials development and tissue engineering. For example, during the last five years four PhD theses were offered in the area of tissue engineering (co-advised with Professors Wang, Yu and Ritter of our Biomedical Engineering Department)—two of which have already been completed. I am very happy to report that we have managed this significant broadening of our research (a byproduct of which is my recent appointment as a joint affiliate professor of biomedical engineering at Stevens) without



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hampering our existing capabilities in the highly filled materials area. All of us at HfMI thank you for your support and look forward to continuing to work with you on your important projects.

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Recent Projects and Capabilities

Biomaterials Research and Development

Four processes that rely on the application of the variants of the twin-screw extrusion process were developed to fabricate biodegradable scaffolds for tissue engineering. The methods are suitable for the fabrication of novel scaffolds

with interconnected open pore structures that are graded in multiple directions with gradients in porosity, mechanical properties and bioactive concentrations.

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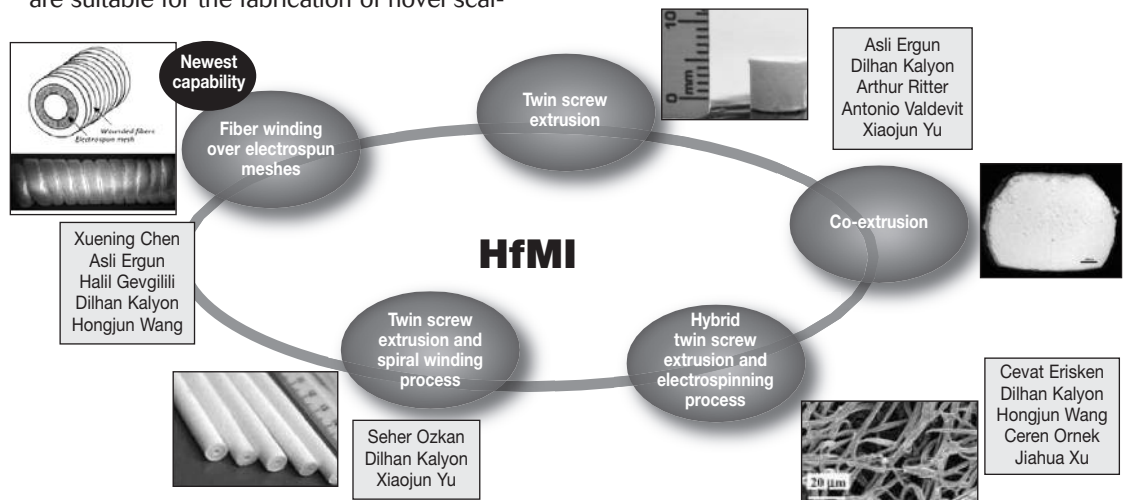


Figure 1. Overview of HfMI's scaffold fabrication capabilities and collaborations.

Biomass Conversion Using Reactive Extrusion

The twin-screw extrusion process was adopted to generate a reactive extrusion process to allow the pretreatment of cellulosic biomass via shear stress application and chemical reaction

(Fig. 2). An invention disclosure was submitted to Stevens and was accepted for conversion into a provisional patent.

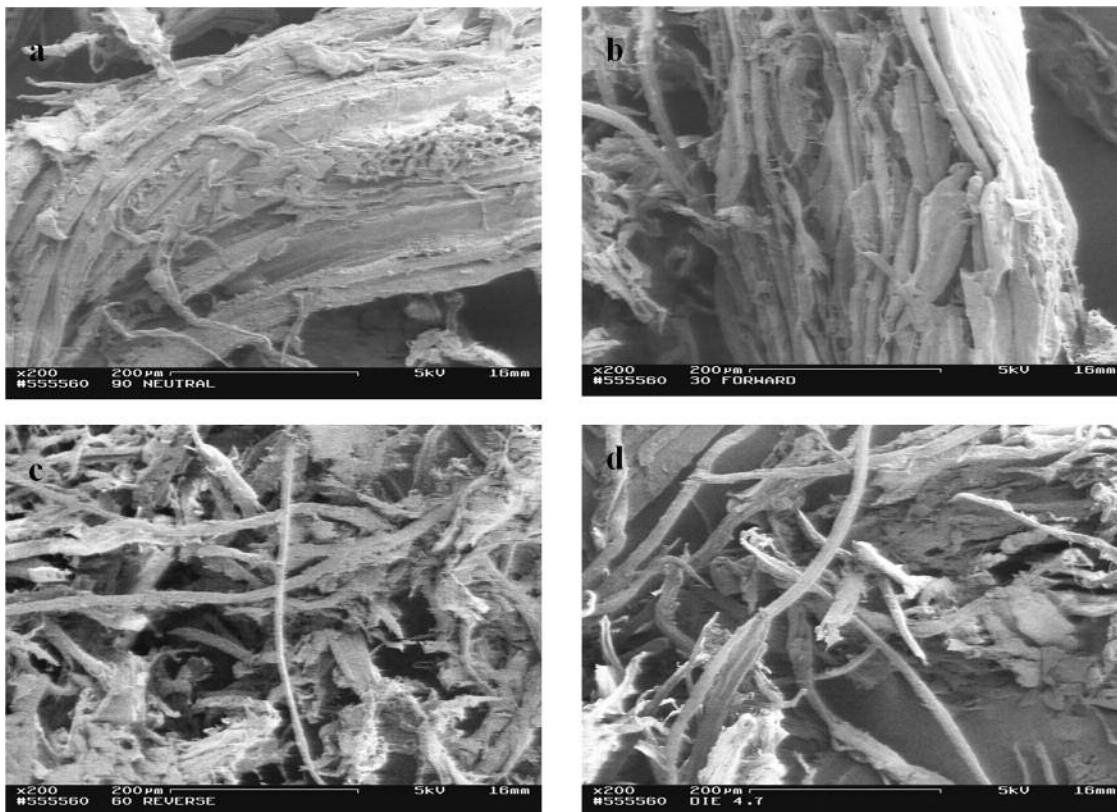


Figure 2. Untreated biomass [a, b] versus pretreated biomass via reactive extrusion [c, d].

Energetic Materials

We have been funded by ARDEC in the areas of advanced coatings and development of intumescent coatings and by Ensign Bickford in the area of controlled feeding and mixing of powders. We are continuing to maintain and develop our capabilities in the areas of rheology, processing, tool design and microstructural analysis of energetic materials.

Personal Care Products

Our funding and collaboration with P&G in the area of vesicle formation from concentrated surfactant suspensions has lead to interesting homogeneous multilamellar vesicles obtained upon dynamic assembly. Our joint manuscript: H. Gevgilili, D. Kalyon, E. Birinci, M. Malik, L. Goovaerts, R. Bacon and P. Mort, "Dynamic assembly of anionic surfactant into highly-ordered vesicles", is available in *Colloids and Interface Science* doi:10.1016/j.jcis.2011.01.011 (2011).

Synthetic Paper

We have received funding from PPG Corporation in the area of manufacturing of synthetic paper.

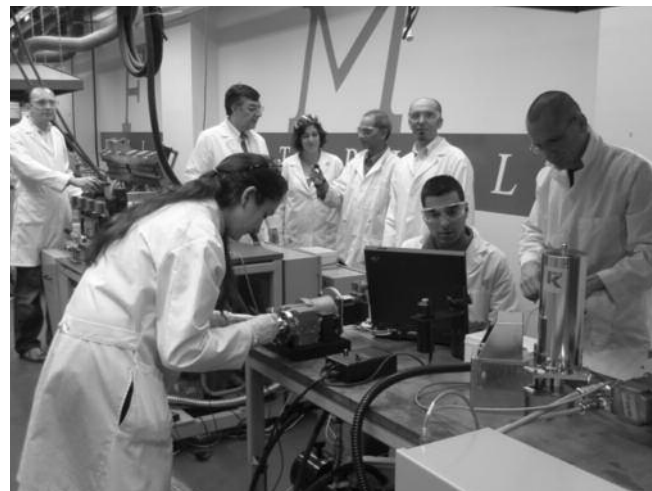


Figure 3. A view of HfMI's processing laboratory.

Nanoparticles and Nanocomposites

In conjunction with our collaboration with Prof. Frank Fisher of Mechanical Engineering of Stevens we are investigating the synthesis, surface functionalization and compounding of myriad nano-inclusions and the structure and ultimate properties of the polymer/nanoparticle composites. The projects of G. Mago, K. Dikovics, S. Vural focusing on C nanotubes are completed and research of S. Senturk, S. Modi, B. Kang, N. Degirmenbasi on synthesis and surface functionalization of nanoparticles is ongoing. The dispersion of the nanoparticles was identified as a major issue that needed to be addressed (see Fig. 4 on the effects of state of dispersion on elasticity of the resulting suspensions) as well as the stability of the matrix polymers into which the nanotubes are compounded.

We have received funding from Benet Laboratories and US Army in the area of compounding of engineering polymers with multi-walled carbon nanotubes and the functionalization of nanotubes (inside their lumens and outside surfaces) with various metallic nanoparticles including Ag, Pt, Pd and Co (Fig. 5).

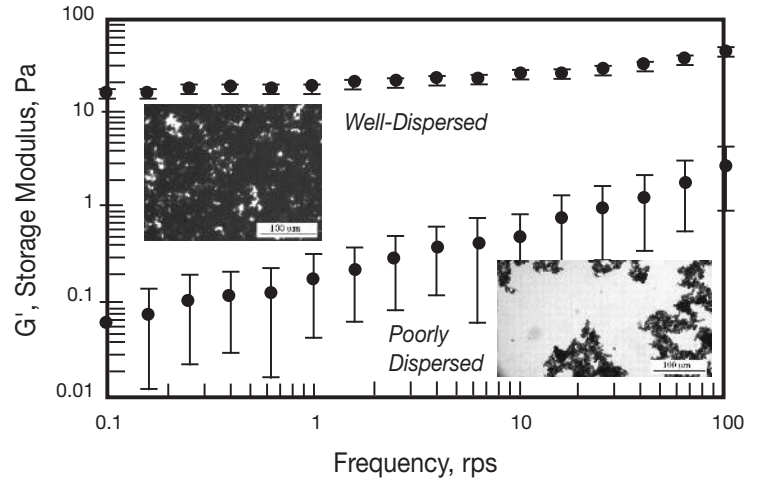


Figure 4. Storage modulus, $G'(\omega)$, as affected by the dispersion state of the carbon nanotubes in a Newtonian fluid [Vural et al., *Soft Matter*, 6, 3870 (2010)]

Catalysis and Environmental

We have collaborated with Kocaeli University in the area of biodiesel synthesis using novel nanoalumina based catalysts (Boz et al., *Applied Catalysis B: Environmental*, 89, 590-596 (2009)).

We have received additional funding from Environmental Technologies (Science & Technology) of Corning Incorporated in the area of mathematical modeling and simulation.

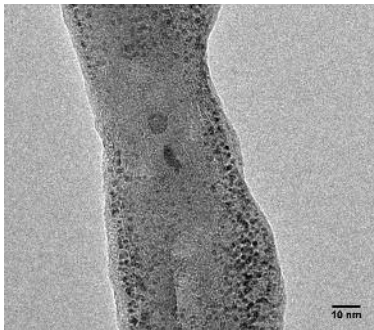


Figure 5. Cobalt-functionalized nanotubes.

Patents

During 2010 two patent disclosures were made to Stevens. Both disclosures were accepted for conversion into provisional US patent applications.

- S. Senturk, H. Gevgilili and D. Kalyon, "Novel Pretreatment Method for Delignification of Cellulosic Biomass via Flowability Control and Reactive Extrusion Process." (See Fig. 2)
- A. Ergun, D. Kalyon, H. Gevgilili, T. Valdevit, A. Ritter, "Integrated multi-zonal bioresorbable cage/core devices as bone graft substitutes and apparatus and method of fabrication of such cage/core devices", Provisional patent application # 61/353,468 filed June 10, 2010. (See Fig.6)

A third application for a US Patent was recently approved by the US Patent and Trademark Office.

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) accepted by US Patent Office (2010).

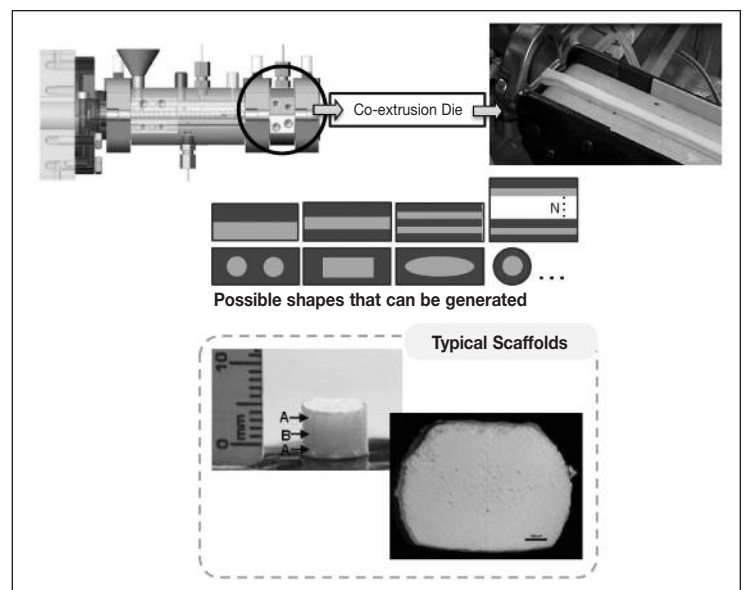


Figure 6: Multizonal biodegradable device for spinal fusion and its manufacturing method

Contributions to the Literature and Presentations (2009-2010)

1. H. Gevgilili, D. Kalyon, E. Birinci, M. Malik, L. Goovaerts, R. Bacon and P. Mort, "Dynamic assembly of anionic surfactant into highly-ordered vesicles," *Colloids and Interface Science*, doi:10.1016/j.jcis.2011.01.011 (2011).
2. S. Ozkan, D. Kalyon and X. Yu, "Functionally graded -TCP/PCL nanocomposite scaffolds for bone tissue engineering: In vitro evaluation with human fetal osteoblast cells," *Journal of Biomedical Materials Research - Part A*, 92, 3, 1007-1018 (2010).
3. C. Erisken, D. Kalyon and H. Wang, "Viscoelastic and biomechanical properties of tissue engineered constructs for osteochondral tissue regeneration," *J. Biomechanical Engineering*, 132, 091013-9 (2010).
4. S. Vural, K. Dikovics and D. Kalyon, "Crosslink Density, Viscoelasticity and Swelling of Hydrogels as affected by Dispersion of Multi-walled Carbon Nanotubes," *Soft Matter*, 6, 3870-3875 (2010).
5. D. Kalyon, "An Analytical Model for Steady Coextrusion of Viscoplastic Fluids in Thin Slit Dies With Wall Slip," *Polymer Engineering and Science*, 50, 4, 652-664 (2010).
6. G. Mago, D. Kalyon and F. T. Fisher, "Processing induced crystallization of semicrystalline polymer nanocomposites," *Proceedings of Society for the Advancement of Material and Process Engineering, SAMPE 2010*, May 17-20, Seattle, WA (2010).
7. S. H. Modi, K. B. Dikovics, H. Gevgilili, G. Mago, S. Bartolucci, F. T. Fisher and D. M. Kalyon, "Effects of carbon nanofibers on the thermo-oxidative degradation of poly(ether ether ketone)," *Polymer*, 51, 5236-5244 (2010).
8. K. B. Dikovics, M. Ocampo, M. Cheung, J. Li, H. Gevgilili, F. T. Fisher and D. M. Kalyon, "Viscoelasticity of poly(ether imide) incorporated with multi-walled carbon nanotubes," submitted to *Composites Part A*, July (2010).
9. C. Erisken, D. Kalyon, C. Ornek, H. Wang and J. Xu, "Osteochondral tissue formation through adipose-derived stem cell differentiation using biomimetic tissue scaffolds with graded stimulator concentrations," *Tissue Engineering Part A* doi:10.1089/ten.TEA.2009.0693 (2010).
10. A. Ergun, X. Yu, A. Valdevit, A. Ritter and D. M. Kalyon, "In vitro analysis and mechanical properties of twin screw extruded single-layered and co-extruded multi-layered poly(caprolactone) scaffolds seeded with human fetal osteoblasts for bone tissue engineering," submitted to *J. Biomedical Materials Research Part A*, August (2010).
11. N. Degirmenbasi, 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," *Journal of Biomedical Materials Research A*, 88A (1), 94-104 (2009).
12. S. Ozkan, D. M. Kalyon, X. Yu, C. McKelvey and M. Lowinger, "Multifunctional polycaprolactone (PCL) scaffolds integrated for controlled release and tissue engineering: In vitro evaluation of released protein secondary structure stability, release profile and biocompatibility," *Biomaterials*, 30, 4336-4347 (2009).
13. N. Boz, N. Degirmenbasi and D. Kalyon, "Conversion of Biomass to fuel: Transesterification of vegetable oil to biodiesel using KF loaded nano- Al₂O₃ as catalyst," *Applied Catalysis B: Environmental*, 89, 590-596 (2009).
14. G. Mago, F. T. Fisher and D. Kalyon, "Deformation-induced crystallization and associated morphology development of carbon nanotube-PVDF nanocomposites," *Journal of Nanoscience and Nanotechnology*, 9, 5, 3330-3340 (2009).
15. G. Mago, F. T. Fisher and D. Kalyon, "Polymer crystallization and precipitation-induced wrapping of carbon nanofibers with PBT," *Journal of Applied Polymer Science*, 114, 1312-1319 (2009).
16. D. H. Moon, M. Wazne, A. Koutsospyros, C. Christoudoulatos, H. Gevgilili, M. Malik and D. M. Kalyon, "Evaluation of the treatment of chromite ore processing residue by ferrous sulfate and asphalt emulsion," *Journal of Hazardous Materials*, 166, 27-32 (2009).
17. G. Mago, D. Kalyon and F. Fisher, "Nanocomposites of nylon-11 and carbon nanofibers: Development of microstructure and ultimate properties following solution processing" submitted to *Journal of Polymer Science Part B: Polymer Physics* (2010).
18. S. F. Bartolucci, G. Mago, H. Gevgilili, S. Vural, K. Dikovics, D. M. Kalyon and F. Fisher, "Investigation of the PEEK-Nanotube Composites Prepared by Solution Methods," *Proceedings of IMECE2009 ASME International Mechanical Engineering Congress and Exposition, Lake Buena Vista, Florida*, November 13-19, 2009.
19. S. Bartolucci, S. Modi, H. Gevgilili, K. Dikovics, F. Fisher, D. Kalyon, "Rheological and thermo-oxidative behavior of carbon nanofibers-poly(ether ether ketone) nanocomposites," *Material Research Society Fall Meeting, Boston*, November 30, 2010.
20. D. Kalyon, "Novel biodegradable scaffolds for tissue engineering and methods of fabrication: Applications to bone tissue engineering," special seminar at *Johns Hopkins University, Rangos Bldg*, 1-2 pm, November 29, 2010.
21. D. Kalyon, M. Malik and Halil Gevgilili, "Sensitivity Control of Energetics through Optimization of Processing Geometry and Conditions," *Annual Meeting of the American Institute of Chemical Engineers, Salt Lake City, Utah*, November 11, 2010.
22. J. Bolognini, C. P. Adam, D. Kalyon, H. Gevgilili and M. Malik, "Coating of Energetic Grains with Deterrents to Tailor Ballistic Performance," *Annual Meeting of the American Institute of Chemical Engineers, Salt Lake City, Utah*, November 11, 2010.
23. S. Prickett, C. Gonzalez, R. Muscato, H. Gevgilili and D. Kalyon, "Entrainment and Removal of Air During Continuous Processing of Energetics: Ramifications On Rheology, Processing and Safety," *Annual Meeting of the American Institute of Chemical Engineers, Salt Lake City, Utah*, November 11, 2010.
24. S. Ozkan and D. Kalyon, "Wall slip behavior of a sodium carboxyl methyl cellulose based hydrogel during drag and pressure induced flows," *Society of Rheology, 82nd Annual Meeting, Santa Fe, New Mexico*, October 27, 2010.
25. U. Yilmazer, T. Seyidoglu, and D. Kalyon, "Polypropylene nanocomposites reinforced with organobentonite," *Polymer Processing Society, PPS-26, Istanbul, Turkey*, October 22, 2010.
26. D. Kalyon, "Wall slip of complex fluids: Ramifications for rheology and processing," *Key Note, Polymer Processing Society, PPS-26, Istanbul, Turkey*, October 20, 2010.
27. D. Kalyon, "Rheology, processing and structure development of concentrated suspensions and nanosuspensions," *Chemical, Biological and Pharmaceutical Engineering Department of New Jersey Institute of Technology, Newark, NJ*, September 13, 2010.
28. D. Kalyon, "Rheology, Simulation and Process Development of Complex Fluids," *Corning Inc., Corning, NY*, August 6, 2010.
29. D. Kalyon, "A Retrospective on the Development of the Science and Technology Base for the Continuous Processing of Energetics: Accomplishments, Capabilities and Weaknesses," *16th JOCG Extruder and Continuous Mixer Users Group, Indian Head, MD*, July 21, 2010.
30. S. Vural, K. Dikovics and D. Kalyon, "Dispersion of multi-walled carbon nanotubes into crosslinkable polyethylene glycol monoacrylate: Effects on development of crosslink density, viscoelasticity and swelling," *Polymer Processing Society Annual Meeting, Banff, Alberta, Canada*, July 8, 2010.
31. D. Kalyon, H. Tang and M. Malik, "Development of flow instabilities during extrusion of flow instabilities during extrusion of polymer melts and polymeric suspensions: Importance of the use of pressure-dependent wall slip condition," *Key Note, Polymer Processing Society Annual Meeting, Banff, Alberta, Canada*, July 7, 2010.
32. D. Kalyon, "Application of core competencies of chemical engineering to multiple industries- Case studies utilizing extrusion of concentrated suspensions," *Plenary lecture, 9. National Chemical Engineering Congress, Ankara, Turkey*, June 22, 2010.
33. D. Kalyon, "Novel graded scaffolds, methods of fabrication and applications for bone tissue engineering," *Koc University, Istanbul, Turkey*, June 17, 2010.
34. D. Kalyon, "Challenges of continuous processing of highly filled suspensions," *PPG Corporation, Barberton, Ohio*, May 26, 2010.
35. G. Mago, D. Kalyon and F. T. Fisher, "Processing induced crystallization of semicrystalline polymer nanocomposites," *Proceedings of Society for the Advancement of Material and Process Engineering, SAMPE 2010, Seattle, WA*, May 17-20, 2010.
36. A. Ergun, H. Gevgilili, D. Kalyon, X. Yu, A. Valdevit, A. Ritter, "Multilayered biodegradable scaffolds for bone tissue engineering," *International Society of Pharmaceutical Engineering, Newark*, April 26, 2010.
37. D. Kalyon, "Dynamics of Continuous Mixing of Concentrated Suspensions," *Bergquist Corporation, Minneapolis, MN*, March 6, 2010.
38. S. Ozkan, D. Kalyon and X. Yu, "Functionally-graded poly(caprolactone) scaffolds: Fabrication via twin screw extrusion/spiral winding process and in vitro assessment of tissue engineering," *Material Research Society Meeting, Boston, Massachusetts*, November 30-December 4, 2009.
39. H. Tang and D. Kalyon, "Analytical solution for creeping channel flow of non-Newtonian compressible fluid subject to wall slip," *62nd Annual Meeting of the American Physical Society, Minneapolis, Minnesota*, November 23, 2009.
40. S. F. Bartolucci, G. Mago, H. Gevgilili, S. Vural, K. Dikovics, D. M. Kalyon and F. Fisher, "Investigation of the PEEK-Nanotube Composites Prepared by Solution Methods," *ASME International Mechanical Engineering Congress and Exposition, Lake Buena Vista, Florida*, November 13-19, 2009.
41. S. Prickett, R. Muscato, D. Kalyon, H. Gevgilili, C. Gonzalez, M. Malik, "Theoretical Analyses of Co-Extrusion Process for Co-Layered Energetic Extrudates and Experimental Investigations of Co-Extrusion," *Annual Meeting of the American Institute of Chemical Engineers, Nashville, Tennessee*, November 12, 2009.
42. D. Kalyon, M. Malik, M. Fair, D. Fair, "Imposition of the Pressure-Dependent Wall Slip Condition in Mathematical Modeling of the Continuous Processing of Energetic Materials: Twin Screw Extrusion and Die Flows," *Annual Meeting of the American Institute of Chemical Engineers, Nashville, Tennessee*, November 12, 2009.
43. K. Dikovics and D. Kalyon, "Nanocomposites of An Engineering Plastic, Poly(ether ether ketone) with Carbon Nanofibers," Poster presentation, *Annual Meeting of the American Institute of Chemical Engineers, Nashville, Tennessee*, November 9, 2009.
44. H. Gevgilili, D. Kalyon, S. Senturk, D. Chiu, S. Moy, "Mathematical Models of the Coating of Energetic Grains Upon Diffusion of Deterrents and Experimental Validation," *Annual Meeting of the American Institute of Chemical Engineers, Nashville, Tennessee*, November 12, 2009.
45. D. Kalyon, "Characterization, simulation and structure development in processing of concentrated suspensions," *LifeTek Solutions and Orthovita Inc., Malvern, PA*, July 17, 2009.
46. F. Fisher and D. Kalyon, "Local Polymer-Nanoparticle Interactions in Polymer Nanocomposites," *Benet Labs Materials Forum: Nano-Scale Materials and Modeling*, February 19, 2009.