A society of volunteer professionals dedicated to fundamental and applied research in science, technology, and the engineering of fibers, and their use as engineered/engineering materials in the service of humanity.

The Fiber Society 2006 Fall Annual Meeting and Technical Conference

October 10–12, 2006

Conference Chair
Dr. Gajanan Bhat
University of Tennessee Knoxville

Sponsors
Department of Materials Science and Engineering
College of Engineering
University of Tennessee Knoxville

e-Spin Technologies
Chattanooga, Tennessee
PROGRAM

Monday, October 9

8:30 AM–5:30 PM  Advanced Fiber Process Development Short Course
10:00 AM–5:00 PM  Governing Council Meeting, the Hilton Knoxville
5:30 PM–7:30 PM  Early Bird Registration, the Hilton Knoxville
7:30 PM–9:00 PM  Early Bird Reception, the Hilton Knoxville

Tuesday, October 10

7:30  Registration and Continental Breakfast
8:20  Welcoming Remarks, Business and Announcements  Gajanan Bhat, Program Chair
       Phil Gibson, President, Fiber Society
       Way Kuo, Dean, College of Engineering, University of Tennessee
8:40  Plenary Talk: Wesley Hoffman, US Air Force Research Laboratory
       How Well Does Carbon Handle Stress?

Day Chair: William Haile, Missing Octave Insights

<table>
<thead>
<tr>
<th>Session 1 A</th>
<th>Session 1 B</th>
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<tbody>
<tr>
<td><strong>High-performance Fibers:</strong>  &lt;br&gt; Andy Campbell, Sunoco Chemicals</td>
<td><strong>Fiber Assemblies:</strong>  &lt;br&gt; Rakesh Gupta, Eastman Chemical Company</td>
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<tr>
<td>9:25</td>
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<tr>
<td><strong>PP and PET Nanocomposites Fibers: Process, Structure and Some Applications</strong>  &lt;br&gt; A. Ajji, J. Denault, M. Bureau, D. Trudel-Boucher and S. Dimitrievska, Industrials Materials Institute</td>
<td><strong>Enhanced Wicking Behavior in Two-Ply Fibrous Composites</strong>  &lt;br&gt; Haskell Beckham¹, Johannes Leisen¹, Patrick McDonald², Fred Ahrens² and Tim Patterson², ¹Georgia Institute of Technology, ²George W. Woodruff School of Mechanical Engineering and Institute of Paper Science and Technology</td>
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<td>9:55</td>
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<tr>
<td><strong>Elongated Rheology and Orientation Number of Polymer Melts and Solutions</strong>  &lt;br&gt; John R. Collier and Simioan Petrovan, University of Tennessee</td>
<td><strong>Hydrophobic-Hydrophilic Surfaces via Modification with Natural Products</strong>  &lt;br&gt; You-Lo Hsieh, University of California Davis</td>
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<td>10:25</td>
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<tr>
<td><strong>Mechanical Properties and Fatigue Analysis of Heat Treated Poly(ethylene Terephthalate) Nanocomposite Fibers</strong>  &lt;br&gt; Rodney Averett, Mary L. Realff and Karl Jacob, Georgia Institute of Technology</td>
<td><strong>Designing Islands in the Sea Nonwovens to Meet Pore Size Specifications</strong>  &lt;br&gt; Glen E. Simmonds, John D. Bomberger and Michael A. Bryner, E. I. DuPont de Nemours &amp; Co.</td>
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<td>11:10</td>
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<tr>
<td><strong>Morphology of In Situ Microfibrillar Composite and Formation of Thermoplastic Micro- and Nanofibers</strong>  &lt;br&gt; Dong Wang and Gang Sun, University of California Davis</td>
<td><strong>Non-particulate Protective Fabrics: New Insight on the Protection/Adsorption Mechanism</strong>  &lt;br&gt; Utkarsh Sata, Senthil Chinnasami, Lohit Shastri and S. S. Ramkumar, Texas Tech University</td>
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*Pitch-based Carbon Fiber Spinning*
### Session 2

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<tr>
<th>Time</th>
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<tr>
<td>1:40</td>
<td><strong>Student Paper Competition</strong></td>
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<td><strong>Session Chair: Dominique Adolphe, ENSITM</strong></td>
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<td></td>
<td><strong>Special Session to Honor Prof. J. E. Spruiell</strong></td>
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<td></td>
<td><strong>Session Chairs: J. E. Spruiell, University of Tennessee</strong></td>
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<td><strong>Bhuvenesh Goswami, Clemson University</strong></td>
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<td>2:40</td>
<td><strong>Structural Hierarchy Developed in Polypropylene/Clay Melt Spun Fibers</strong></td>
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<td>J. H. Fujiyama-Novak and Miko Cakmak, University of Akron</td>
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<td>3:05</td>
<td><strong>Thermal Bonding of Polyolefin Fibers</strong></td>
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<td>Rajen Patel, Wenbin Liang, Gert Claasen, Karin Katzer, Jesus Nieto and Thomas Allgeuer, Dow Plastics</td>
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<td>3:30</td>
<td><strong>Superparamagnetic Flexible Substrates Based on Submicron Electropsun Estane® Fibers Containing MnZnFe-Ni Nanoparticles</strong></td>
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<td>Pankaj Gupta, Ramazan Asmatulu, Rick Claus and Garth Wilkes, Virginia Polytechnic Institute and State University</td>
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<td>3:55</td>
<td><strong>Break</strong></td>
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<tr>
<td>4:05</td>
<td><strong>Fiber Spinning Behavior of Biodegradable Polymers</strong></td>
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<td>Eric Bond, Procter &amp; Gamble Company</td>
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<td>4:30</td>
<td><strong>Morphological Investigation of Fibrous Materials by Multiaxially Stretched Films</strong></td>
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<td>Kyung-Ju Choi, AAF International</td>
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<td>4:55</td>
<td><strong>Fibrous Form of Bioabsorbable Polymers in Surgical Applications</strong></td>
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<td>Jack Zhou, Ethicon, Inc.</td>
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<tr>
<td>5:20–7:30</td>
<td><strong>Poster Presentations and Table Top Exhibits — Refreshments Provided</strong></td>
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### Wednesday, October 11

- **7:30** Registration and Continental Breakfast

**Day Chair: Kevin Kit, University of Tennessee**

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<tr>
<th>Session 3 A</th>
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</table>
| **Fiber Formation:**
Elizabeth McFarland, INVISTA | **Fabrics and Modeling:**
Phil Gibson, US Army, Natick |
| 8:30 | **Preparation of Superhydrophobic Surface Using Lotus Effect** |
| | Hoon Joo Lee and Stephen Michielsen, North Carolina State University |
| 9:00 | **Development of Orientation in Extruded Cellulose Fibers** |
| | Ramsis Farag¹, Roy M. Broughton¹, Fatma Kilinc-Balcı¹, Weijun Wang¹, Richard Swatloski² and Robin Rogers², ¹Auburn University, ²University of Alabama |

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**A clothing modeling framework for uniform and armor system design**

Xiaolin Man, Colby C. Swan, Jun Yang and Salam Rahmatalla, University of Iowa
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<thead>
<tr>
<th>Time</th>
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<th>Speaker(s)</th>
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<tbody>
<tr>
<td>9:30</td>
<td>Wet Spinning of High Molecular Weight Aliphatic Polyamides</td>
<td>Amit Gupta, M. Afshari, R. Kotek, A. E. Tonelli and Nad Vasanthan, North Carolina State University, Long Island University</td>
</tr>
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<td>Tension and Bending Performance of Single Muscle Fiber in the Pig</td>
<td>Yuqing Liu and Ke Wang, Donghua University</td>
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<td>10:00</td>
<td>Break</td>
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<tr>
<td>10:15</td>
<td>Solvent Properties of Aqueous ZnCl2 Solution Toward Stereoregular Poly(acrylonitrile) Prepared by Urea Clathrate Polymerization</td>
<td>Masatomo Minagawa, Toru Kawase, Yosuke Kobayashi, Tomochika Matsuyama and Nobuhiro Sato, Yamagata University, Kyoto University</td>
</tr>
<tr>
<td></td>
<td>Ballistic Penetration Simulation of Textile Fabrics</td>
<td>Youqi Wang, Yuyang Miao and Bryan A. Cheeseman, Kansas State University, U. S. Army Research Laboratory</td>
</tr>
<tr>
<td>10:45</td>
<td>Spinability and morphology of melt-spun nanocomposite fibers</td>
<td>Rudolf Hufenus, Giuseppino Fortunato, Paul Brühlwiler, Jörn Lübben and Marcel Halbeisen</td>
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<td>Transplanter Water Transport Tester for Fabrics</td>
<td>M. K. Sarkar, J. T. Fan and X. M. Qian, Hong Kong Polytechnic University</td>
</tr>
<tr>
<td>11:15</td>
<td>Elastis Fibers Based on Polyolefin Elastomers</td>
<td>Hongyu Chen, Debbie Chiu, Ben Poon, Ashish Sen and Shih-yaw Lai, Dow Chemical Company</td>
</tr>
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<td>Hemp Fiber Preparation: Designing and Calculating a Full Size Production with Process Oriented Analysis</td>
<td>Daniel Gantner, Institute for Manufacturing Automation</td>
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<tr>
<td>11:45</td>
<td>Elastic Nonwoven Fabrics from Specialty Polyolefin Elastomers</td>
<td>Raja Dharmarajan, Smita Kacker, Vincent Gallez, Bruce A. Harrington and A. D. Westwood, ExxonMobile Chemical Corporation</td>
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<td>Plasma Surface Modification of Textiles</td>
<td>M. M. Hossain and Dirk Hegemann, Empa</td>
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<td>12:15</td>
<td>Lunch</td>
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### Session 4 A

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<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker(s)</th>
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<tbody>
<tr>
<td>1:30</td>
<td>The Effect of Orientation on the Thermal Behavior of Nylon 6 Fibers</td>
<td>F. Dadashian, Amirkabir University of Technology</td>
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<td></td>
<td>Thermocurrents from spider dragline silk</td>
<td>Xiaomeng Shi and Michael Ellison, Clemson University</td>
</tr>
<tr>
<td>2:00</td>
<td>Fundamental Study of the Driving Mechanisms for Cross-Section Shape Change in Highly Noncircular Fiber Spinning</td>
<td>Donggang Yao, Georgia Institute of Technology</td>
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<td>Textile Heart Valve Prosthetic: In Vitro Performance of the Fabric</td>
<td>F. Heim, B. Durand and N. Chakfe, ENSITM</td>
</tr>
<tr>
<td>2:30</td>
<td>Modeling the Torsional Behavior of Human Hair</td>
<td>Prasad Potluri, Don Harper, Yash Kamath, University of Manchester, TRI/Princeton</td>
</tr>
<tr>
<td></td>
<td>Phosphate Glass Fibres as Reinforcement in Resorbable Composites for Medical Implant Devices</td>
<td>A. J. Parsons, S. Cozien-Cazuc, Jing Yang, C. D. Rudd, and Ifty Ahmed, University of Nottingham</td>
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### Session 4 B

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<thead>
<tr>
<th>Time</th>
<th>Title</th>
<th>Speaker(s)</th>
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<tbody>
<tr>
<td>1:30</td>
<td>Fiber Structure: Kevin Kit, University of Tennessee</td>
<td>Financial Fibers: Stephen Michielsen, NC State University</td>
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<tr>
<td></td>
<td>The Effect of Orientation on the Thermal Behavior of Nylon 6 Fibers</td>
<td>Thermocurrents from spider dragline silk, Xiaomeng Shi and Michael Ellison, Clemson University</td>
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<tr>
<td>Time</td>
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<td>Session B</td>
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<td>3:00</td>
<td><strong>Break</strong></td>
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<tr>
<td>3:15</td>
<td>Optical hairiness- and profile-meter for flexible surfaces</td>
<td>Composite Material Fabrication and Testing for Galactic Cosmic Radiation Shielding</td>
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<td>Marie-Ange Bueno and Laurent Bigué, University of Mullhouse</td>
<td>Raj Kaul, NASA, Marshall Space Flight Center</td>
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<tr>
<td>3:45</td>
<td>Objective and Subjective Handle Evaluations for Disposable Diaper’s Top Sheets and Reusable Diaper’s Fabrics</td>
<td>Improvement of Dentist Gowns: New Constraints and New Risks</td>
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<td>Mitsuo Matsudaia, Jun-ichi Yoshida and Toshiyasu Kinari, Kanazawa University</td>
<td>Marie Schacher(^2), Stéphane Berger(^1), Dominique Adolphe(^1), Youssef Haïkel(^2),</td>
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<td>(^1)ENSITM, (^2)Faculté de Chirurgie Dentaire</td>
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<td>4:15</td>
<td>Experimental methods to evaluate the fiber length and orientation distribution of long glass fibers in</td>
<td>Synthesis of Cellulose Carbamate in Supercritical Carbon Dioxide</td>
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<td>injection molded thermoplastics</td>
<td>Cuiyu Yin(^{1,2}), Tianlai Xing(^2) and Xinyuan Shen(^1), Donghua University, (^1)Donghua</td>
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<td>Gregorio Vélez-Garcia(^1) and V. Kunc(^2), (^1)Virginia Polytechnic Institute and</td>
<td>University, (^2)Henan Textile College</td>
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<td>State University, (^2)Oak Ridge National Laboratory</td>
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<td>4:45</td>
<td><strong>General Body Meeting: Open to Fiber Society Members Only</strong></td>
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<tr>
<td>6:30</td>
<td><strong>Reception and Banquet</strong></td>
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**Thursday, October 12**

7:30   Registration and Continental Breakfast

**Day Chair:** Larry Wadsworth, University of Tennessee

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<th>Time</th>
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<tr>
<td>8:30</td>
<td>Correlation of Electrospun Fiber Structure to Jet Properties</td>
<td>Multifunctional Polymer Carbon Nanotube Composites for Light-Emitting and Photovoltaic Applications</td>
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<td>Samira Farhoodmanesh, John J. Mooskian and Julie Chen, University of Massachusetts Lowell</td>
<td>Bin Hu, University of Tennessee</td>
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<tr>
<td>9:00</td>
<td>Aligned Polymeric Nanofiber Arrays Using Biased AC Electrospinning</td>
<td>Electroluminescent White-Light Emitting Devices Constructed of Dye-Doped Colloidal Polymer</td>
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<td>Soumayajit Sarkar and Gary Tepper, Virginia Commonwealth University</td>
<td>Particles Prepared Using a Mini-Emulsion Technique</td>
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<td>Christopher Huebner, David Evanoff, Jr., Yurong Ying and Stephen Foulger, Clemson University</td>
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<tr>
<td>9:30</td>
<td>An Integrated Model of the Electrospinning Process</td>
<td>Flexible Electrochromic Devices Using Inkjet Printed Electrochromic Layer</td>
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<td>Yuki Imura, Leslie LaNieve, George Collins, Sheldon Wang, Wei-Kuo Lee and Michael Jaffe, New Jersey Institute</td>
<td>Goo Hwan Shim, Moon Gyu Han and Stephen Foulger, Clemson University</td>
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<td>of Technology</td>
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<td>10:00</td>
<td><strong>Break</strong></td>
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<td>10:15</td>
<td>Dynamic Performance Analysis of a Weaving Mill</td>
<td>Simon Zeier, Swiss Federal Institute of Technology</td>
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<td>10:45</td>
<td>Supercritical Carbondioxide Dyeing of Polyester</td>
<td>Ajay Shankar Joshi, Tanveer Malik and Shivendra Parmar</td>
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<tr>
<td>11:15</td>
<td>The Study of Improving the Tearing Strength of Coated Fabrics with Silicone</td>
<td>Yiren Chen(^1,2), Jianyong Yu(^1) and Caiyuan Chu(^1), Donghua University, Wuhan Institute</td>
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<td>11:45</td>
<td>Analysis of Sulfonated Azo Dyes: Effect of Structure on Enzymatic Decolorization</td>
<td>Yiping Lu and Ian R. Hardin, University of Georgia</td>
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### Poster Presentations

**Session Chair: Allan Stahl, University of Tennessee**

- *Functionalized Nanofibers for Defense Applications*
  S. S. Ramkumar, Presenter

- *Relationship Between Cotton Breeds and the Comfort of Knitted Fabrics: A New Understanding*
  S. S. Ramkumar, Presenter

- *High Efficiency Encapsulated N-Octadecane for Thermoregulated Textile*
  Kishor Gupta, Presenter

- *Evaluation of Self-Decontamination Textiles for Chemical Protective Clothing*
  Ellan Spero, Presenter

- *Melt spun thermoresponsive shape memory fibers from polyurethane: effect of drawing and heat-setting on fiber morphology and properties*
  Jasmeet Kaur, Presenter

- *Stent for Percutaneous Textile Heartvalve Implantation: First Results*
  Coralie Marchand

- *Bamboo Charcoal Fiber and Related Application*
  Fuli Zhang, Presenter

- *Synthesis of Reactive Dye Resist Agents for Cellulose Based on Maleic Anhydride Copolymers*
  Kee Jong Yoon, Presenter
- Effect of Thickness Variation on the Chemo-physical Properties of Electrospun PLGA Nanoweb
  Tae Jin Kang, Presenter

- Electrostatic characteristics of the washer-dryer combination under various drying conditions
  Chung Hee Park, Presenter

- Effect of spinning conditions on the nonwoven properties of electrospun polyurethanes
  Chung Hee Park, Presenter

- Simulation of Injection Molding Thermoplastic Reinforced with Micro- and Nano-Particles
  Gregorio Vélez-Garcia, Presenter

- Convergent synthesis of poly(methyl methacrylate) (PMMA) based electroluminescent colloids
  Shane Hayes, Presenter

- Propargyl poly(acrylate) photonic bandgap composites
  Ryan Roeder, Presenter

- Structure and Properties of Nanoclay Reinforced Polypropylene Fibers
  M. G. Kamath, Presenter

- Thermal Bonding of Polypropylene Films and Fibers
  Raghavendra Hegde, Presenter

- Structure and Properties of Latex Coated Nonwovens
  Prabhakar Gulgunje, Presenter

- Statistical approach to neural network model building for prediction of compressive properties of woven fabrics
  B. R. Gurumurthy

- Extrusion of Immiscible Polymer Blends for Fabrication of Highly Porous Fibers with Continuous and Size Adjustable Porous Structures
  Pratapkumar Nagarajan, Presenter

- A Novel Light-Emitting Fiber
  Indraneel Sen, Presenter

- The Use of Bacterial Cellulose as a Novel Nonwoven Fiber Matrix for Ceramic and Metal Deposition
  Stacy Hutchens, Presenter

- Recovery and Reuse of PVA Size Using Flash Evaporation
  Kishor Gupta, Presenter

- Acid Leuco Dyeing of Unmodified Polypropylene Fiber
  Murari Gupta, Presenter

- Effect of Molecular Weight and Polymer-Solvent Concentration on Electrostatic Processing of Chitosan
  Keyur Desai, Presenter

- Mechanical Properties of Fibers Using Nano-Tensile Testing
  Matt Kant, Presenter

- Shaped fibers and their applications
  Alex Lobovsky, Presenter
Session 1 A
Polymer nanocomposites (PNC) have attracted much attention during the past ten years because of their low cost and their ready availability. In comparison with the pure polymer or conventional (microscale) composites, the presence of nanoparticles in the polymer matrix can significantly increase moduli, strength, and heat resistance, and decrease gas permeability and flammability. Those properties will however depend strongly on orientation of both the matrix and reinforcement. PNCs also present an extraordinary opportunity for polymers to be used in tissue repair and reconstruction applications. Indeed, bone tissue for example is a hierarchically structured composite material formed of nanofibres containing a polymer phase, collagen type I, and a mineral phase, crystalline apatite nanoparticles, organized in a complex 3D structure. In this study, we investigated the effect of material and process parameters on the dispersion and orientation of the polymer and nanoparticles in polypropylene (PP)/Montmorillonite clay and polyethylene terephthalate (PET)/hydroxy apatite (HA) nanocomposites.

As potential biomaterials targeted for load bearing bone applications of the orthopaedic/dental implant field, we also evaluated PET/HA nanocomposites biocompatibility in vitro.

Fibers from PP and PNC as well as PET and PET/HA fibers were produced using a fiber spinning process. Various amounts of clay and HA were used. The structure of PP and PNC fibers was evaluated using field emission scanning electron microscopy (FE-SEM) and X-ray diffraction. The crystalline axes orientation factors were determined from wide-angle X-ray diffraction pole figures of (110) and (200) reflections for PP and of (001) for clay. The d-spacing of the (001) clay crystalline plane, indicative of the clay dispersion, was determined from the plots superimposed on the two dimensional X-ray diagrams. Similar characterization was also done on PET/HA fibers. The results obtained for the d-spacing at small angles indicated some partial intercalation of the clay in the PNC (d spacing of 2.52 and 3.15 nm). Results on orientation of the clay (001) axis (normal to the clay platelets plane) indicated that it was oriented in the normal direction, which is expected. Its orientation in machine and transverse directions were different, suggesting that clay platelets were not randomly distributed in the transverse plane of the fibers. This was confirmed in the SEM observations showing preferential alignment of the clays. The orientation of PP crystalline axes in the fibers was slightly higher for the nano-composite that the pure resin. Tensile modulus, strength and elongation at break for both PP and PNC fibers increased with increasing draw ratio. Flexural modulus and strength of laminates made by consolidation of the PNC fibers were higher than those of consolidated pure PP fibers as well as those of bulk PP or PNC.

For PET/HA nanocomposites, X-ray results indicated that PET matrix was amorphous. Lastly L929 fibroblasts cells cultured directly on the PET/HA fibres, demonstrated a dose dependent relationship with the amount of HA present in the fibers, up to 14 days in culture. This intimate response was quantified by Alamar Blue (93% and 72% cell viability for PET 10 and PET 0 respectively at day 3) and qualified by Field Emission Gun Scanning Electron Microscope (FEG-SEM).

The ability of the PET10 fibre matrices to support L929 attachment, spreading and growth in vitro, combined with the compatible degradation extracts suggests potential use of these composites as load-baring bone biomaterials.
Oriented fiber and film formation is dominated by elongational deformation and relaxation during processing. Elongational rheology of polymer melts and solutions has been measured with hyperbolically convergent dies at typical processing temperature, elongational strain rates and Hencky strains. Some of the same melts were evaluated in other laboratories on Meissner type devices. Characteristic relaxation times were also determined for these materials by dynamic shear rheology.

An Orientation Number that is modified Weissenberg type number is suggested to explain the agreement and disagreement between the elongational viscosity data measured by different techniques. This Orientation number is the product of the Hencky strain, the elongational strain rate, and the average relaxation time. According to the value of this number, the flow regime can be divided in three domains. At low orientation numbers, the flow is in a relaxation dominant regime, as contrasted to the orientation dominant flow generally at orientation numbers higher than one, and an intermediate transition regime. When the fluid is at an orientation number somewhat greater than one it appears that slip at the wall occurs in the hyperbolic dies and pure elongational flow is approached as it is in most free surface deformation processes. When the extrudate exits the die in the transition region, the extrudate surface is disrupted whereas in the relaxation dominant and orientation dominant regions the extrudate is smooth. Agreement exists between the hyperbolic die and Meissner type devices when the sample passes through the transition region at low deformation, but if the transition is reached at higher deformations in the Meissner type, indicating significant thinning the sample fails, however the hyperbolic dies do not have that problem.

Commercial melt spinning and melt blowing nonwovens tend to operate well into the orientation dominant region and typically are solidified rapidly while under tension. Therefore, high degrees of orientation can be imparted and retained since relaxation is impeded by line tension and inhibited after solidification.
PET control fibers (diameter \(\sim 24\pm3\mu m\)) and heat treated polyethylene terephthalate (PET) fibers with embedded vapor grown carbon nanofibers (VGCNFs) (diameter of \(\sim 25\pm2\mu m\)) were exposed to monotonic tensile tests and cyclic loading. The control fibers were processed through a typical melt-blending technique and the PET/VGCNF samples were processed with approximately 5 wt.% carbon nanofibers present in the sample. When tested at a strain rate of 6.69E-03s\(^{-1}\), the mechanical properties of the PET/VGCNF samples outperformed the control samples; elastic modulus, hardening modulus, yield strength, yield strain, fracture strength, and tensile energy of the PET/VGCNF samples were all higher than the control samples. The constitutive response of both the pristine PET control samples and PET/VGCNF samples was modeled as a Ramberg-Osgood solid [1] with significant strain hardening.

When cycled above the static yield point for a few cycles, the PET/VGCNF samples absorbed \(\sim 79\%\) more energy in the form of hysteresis.

From Figure 1, the equation that appropriately describes the energy dissipated during one load-unload cycle (1.1) is:

\[
\Delta E = \int_{\sigma} \sigma \varepsilon \, d\varepsilon = \int_{\sigma} \sigma \varepsilon \, d\varepsilon - \int_{\sigma} \sigma \varepsilon \, d\varepsilon
\]  

(1.1)

More appropriately, the integration from the standpoint of energy absorbed during one cycle in terms of angular frequency is [2]:

\[
\Delta E = \int_{0}^{2\pi/\omega} \sigma \frac{d\varepsilon}{dt} \, dt
\]  

(1.2)
Under uniaxial fatigue conditions for a large number of cycles, the fibers were subjected to a maximum stress that was approximately 80% of the yield stress of the sample at a strain rate of 6.69x10^{-3}s^{-1} in uniaxial tension. The equation that appositely describes this dynamic loading condition is:

\[
\begin{align*}
\varepsilon &= (\Delta \varepsilon) \sin \omega t \\
\sigma &= (\Delta \sigma) \sin (\omega t + \delta)
\end{align*}
\]  

(1.3)

In 1.3, \(\varepsilon\) is the instantaneous strain, \(\varepsilon_a\) is the strain amplitude, \(\omega\) is the angular frequency, \(\sigma\) is the instantaneous stress, \(\sigma_a\) is the stress amplitude, and \(\delta\) is the phase lag component of the strain. For same stress dynamic loading conditions under fully reversed loading, the phase lag component of the PET/VGCNF samples was higher than the PET control samples. The average value of the time lag differential for the PET/VGCNF samples was 0.018s, whereas the average time lag differential for the PET control samples was 0.014s.

Subsequent to non-destructive fatigue conditions, the fibers were tested under uniaxial stress conditions. The fracture strength and tensile energy of the samples in uniaxial tension subsequent to fatigue were shown to be dependent on the cyclic stress amplitude (\(\Delta \sigma\)) and prior accumulated strain for a fixed frequency of 5Hz. For the PET/VGCNF samples, there were negligible effects on the static yield point and modulus subsequent to fatigue. The effects of the fatigue process on these mechanical properties have been hypothesized and supported through existing qualitative and quantitative analyses.

References


Hybrid blends of cellulose acetate butyrate (CAB) with thermoplastic polymers (TP) could form *in situ* microfibrillar composites by ram extrusion with regular dies. Most thermoplastic polymers including polyesters, polypropylene and polyethylene can be blended with CAB and produce the special composites. The composite materials demonstrated self-reinforcement properties resulted from the microfibrils of TP in the composites. Morphologies of the microfibrillar were studied and will be discussed in the presentation. More interestingly, the microfibrillar structures in the composites are similar to that of sea-island structures, which can be employed in production of microfibers of the TPs. Enhanced and repeated dispersions of the blends and subsequent extrusion could produce fibers in 100-200 nanometers.
There are a number of designs for blow spinning processes for thermoplastic materials. The fibers produced from such materials are relatively robust and can tolerate handling stresses present during spinning and collection. Low cost carbon fiber is produced from mesophase pitch in a new high speed melt blown (patent applied for) process that is using air at near sonic speeds. Green fiber produced in this process is continuously attenuated to the desired diameter in the spinning process. The fibers are then slowed down to a few feet per second at the collection zone. The green fibers are very delicate and cannot support large tensile and bending forces at this stage in their production process. In this paper we will discuss major design and associated operational aspects in this spinning process.
Session 1 B
Enhanced Wicking Behavior in Two-Ply Fibrous Composites

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Wicking of fluids into porous media is driven by capillary pulling forces, which are governed by the fluid-solid interaction. Capillary pulling forces per unit area are called capillary pressures and are used to characterize porous substrates. When the fluid-solid interaction is attractive, substrates with small pores exhibit stronger capillary pulling forces (i.e., higher capillary pressures) than those with larger pores, simply due to the higher surface areas for a given volume in which the fluid interacts with the solid. On the other hand, substrates with small pores present a higher resistance to fluid flow than those with larger pores. In other words, the permeability of small-pore substrates is lower than it is for large-pore substrates. In an upward "wicking race" between the same fluid in a small-pore substrate versus a large-pore substrate of the same material, the large-pore substrate can lead at short times due to its higher permeability, but the small-pore substrate ultimately wins due to its higher capillary pressure.

When a small-pore substrate is laid next to (and in contact with) a large-pore substrate, the wicking behavior is noticeably enhanced. This is shown in Figure 1 for paper hand sheets, all of which have the same basis weight. Why does this two-ply fibrous composite exhibit enhanced wicking behavior? Does the fluid wick along channels created at the inter-ply interface? Does one ply feed the fluid that leads the wicking? Does the fluid exchange between the plies to find the most expedient route? Can similar effects be mimicked in single plies with appropriately designed pore-size distributions? We have been addressing these questions with a multi-pronged approach: (1) bulk wicking studies to define the permeability-saturation-capillary pressure (k-S-Pc) relationships, (2) spatially resolved wicking studies using magnetic resonance imaging (MRI) to provide insight into the wicking mechanism, and (3) model development to predict wicking behavior in multi-ply fibrous composites. The results from these studies will be described in this presentation.

**Figure 1.** Bulk upward wicking in paper hand sheets. All three samples are approximately the same thickness (0.7 mm) and basis weight (~230 g/m²). For the 2-ply sample, two sheets were prepared (small-pore and large-pore) using half the amount of pulp, and then pressed together prior to drying.
Liquid interactions with a fibrous material are determined by its surface properties and inter-fiber pore characteristics. While the pore structures are controlled by the assembly methods, surface properties are dominated by chemical nature of the fibers and/or any topical modifications. Surface chemistry determines the wetting behavior of fibrous materials that in turns determines how the liquid is transported into the pores and absorbed by the fibers. This paper analyzes how surface wetting behavior can be altered in a highly controlled manner by liquid surface tensions and/or by modifying surface hydrophobicity via adsorption and chemical reactions.

The characteristic of an aqueous media can be altered by the presence of surfactants. Aqueous solutions of varied surface tensions between 26.66 and 38.02 dyne/cm² were made from 0.1% surfactant conc. and between 39.71 and 71.67 dyne/cm² were obtained from dilutions between 10⁻² and 10⁻⁵%. These liquids have hydrophilic-lipophilic balance values from 8.6 to 16.7. The molecular surface areas of individual surfactant molecules were derived from Gibbs’ adsorption isotherms. Analyses of cosine wetting contact angle, capillary liquid, and their correlations with liquid and HLB values provide insight into the hydrophilic-hydrophobic nature of these fibrous materials. In the range of higher than 38.02 where surfactant concentrations are less than the critical micelle concentration (CMC). The water retention dramatically decreased with decreasing surfactant concentrations below the CMC, or increasing.

Surface hydrophobicity can be modified via adsorption and chemical reactions. Adsorption of chemically similar vs dissimilar biologically derived compounds to the fibers and how this process affect wetting is demonstrated on cellulosic fibers, including pulp paper, cotton woven/nonwoven and cellulose nanofibers. Adsorption affinity of compounds is closely associated with their structural relationship to the fiber and clearly evident by the change in surface wetting. The figures show cellulose surfaces can become hydrophobic with the adsorption of low concentrations of hydrophobic compounds of chemical similar (right) and dissimilar (left) structures. Although the initial effects are similar between them, the chemically similar structure shows superior stability.
New nonwovens applications in areas such as filtration require a media designed to particular pore size specifications in the 3 to 20 micron range. We have found that spunbonded islands-in-the-sea nonwoven fabrics can be designed very precisely to achieve target pore diameters and porosity. Mathematical models can be used to develop fabric specifications in the standard manufacturing terms of basis weight and fiber diameter. A model developed by M.A. Bryner is presented and compared to a model developed by N. Lifshutz (2005). The two models presented diverge in terms of the influence of fabric thickness on the mean pore diameter. Measured mean flow pore diameters for a series of test fabrics showed excellent correlation to targeted mean flow pore diameters for both models. Experimental fit to the Bryner model is the better of the two, but requires specification of fabric thickness in addition to basis weight and fiber diameter to achieve actual mean pore diameters that closely match target values. Experimental validation of the influence of fabric thickness on the mean flow pore diameter at constant basis weight and fiber diameter remains open for further investigation. In addition, achieving complete separation of the island and sea polymers along with unbundling of the island fibers remain areas for improvement.
Non-particulate Protective Fabrics: New Insight on the Protection/Adsorption Mechanism

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The development of non-particulate sorbent decontamination wipe is a priority endeavor in the modernization strategy of the decontamination program of the US Department of Defense. The well known sorbent substrate is activated carbon and the principal mechanism behind its end use application is adsorption. Therefore, objective quantification of the adsorption characteristics of activated carbon fabrics is very important in the development of next generation decontamination wipes. This paper reports the results on the adsorption of a chemical agent simulant solutions such as pinacolyl methyl phosphonate (0.1 % w/v in butanol) by activated carbon nonwoven fabrics varying in their precursor materials and surface area.

A simple and novel protocol has been designed to understand the mechanism of adsorption of non-particulate protective fabrics. Thermogravimetric analyzer (TGA) has been used to objectively quantify the adsorption properties of various substrates. The TGA study gave precise weight gain values and has shown to be a reliable and objective method to quantify the adsorption characteristics of sorbent activated carbon nonwoven fabrics. For each experimental run 99.9 % ultra high purity nitrogen gas was passed through the TGA system for all the experiments. N₂ gas streams were controlled by flow controllers, where one of the N₂ streams (sheath gas-20 cc/min) was passed through a flat bottomed flask filled with the solution of the toxic chemical (PMP and butanol solution (0.1 % w/v). The results indicate that the Thermo-gravimetric analyzer (Pyris 1 TGA® Perkin ElmerTM), which is conventionally used for chemical composition analysis and oxidation behavior, when used in this setting, is an efficient analytical tool for quantifying the adsorption characteristics such as the saturation time and adsorption capacity. Results from the study show that non-particulate sorbent fabrics have higher adsorption capacity than particulate sorbent powders. A typical adsorption isotherm of nonwoven activated carbon is shown in Figure 1. As is evident from Figure 1, the activated carbon nonwoven can instantaneously adsorb toxic vapors. More importantly, the TGA based analytical method can serve as a good objective method due to its reproducibility.
Figure 1. Normalized adsorption curve indicating toxic chemical adsorption by M 291 Kit and nonparticulate nonwoven activated carbon.

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Development of an Antimicrobial and Breathable Microporous Polyurethane Membrane

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With the ever-growing public health awareness of disease transmission, crossinfections and malodors, barrier materials are having increasing applications as protective clothing materials for medical and chemical workers, sportswear and underwear. In our previous study, microporous polyurethane (PU) membranes were modified by grafting hydrophilic polymer onto the surfaces and within the pores of the membranes to enhance moisture transport while reducing the pore size to improve the barrier properties [1]. For enhanced performance, antimicrobial properties are desirable for use in health related applications.

Commonly used antimicrobial agents, such as antibiotics, silver ions, quaternary ammonium, the relatively new N-halamines and other biocidal agents, can be applied onto textiles and membrane materials by various chemical and physicochemical finishing techniques to protect the substrates from biological activities [2-4]. In this study, the effect of N-halamine chemistry on microporous polyurethane (PU) membrane was explored by grafting a hydantoin moiety onto PU membrane surface as an N-halamine precursor (Scheme 1). A combination of techniques was used to characterize this surface modification. Thermal and mechanical properties of the surface modified PU membranes were evaluated accordingly. Upon exposure to certain concentration of chlorine bleach, the hydantoin structures on the grafted PU membranes were transformed into N-halamines, which provided potent antimicrobial activities against both gram-negative and gram-positive bacteria (Scheme 2). A total reduction was observed for the TMIO modified PU membrane after a two hour contact period. Consequently, a microporous membrane that not only possesses good barrier and hygiene protections, but also maintains sufficient comfort properties in terms of water vapor transmission rate (WVTR) was developed. Detailed findings of this study will be discussed.


![Scheme 1: Two-step reaction scheme of: (A) HMDI activation of PU membrane surface, and (B) surface grafting of TMIO.](image1)

![Scheme 2: Chlorination of: (a) control PU membrane; (b) TMIO modified PU membrane.](image2)
Session 2
We have investigated the effect of clay nanoparticles on the crystallization and orientation behavior of melt-spun Polypropylene (PP) and PP/PPgMA (maleic anhydride modified polypropylene) fibers using WAXD, SAXS, birefringence and TEM techniques.

TEM analysis on the cross section of the fibers with no take up (A-surface) indicates tangential orientation of the platelets near the skin but near random in the center with plate normals oriented mostly in radial direction (Fig 1). The results of WAXD show that the crystalline chains in fibers with nanoparticles exhibit higher orientation than for unfilled PP/PPMA indicating that the presence of nanoparticles improves the amorphous and crystalline orientation at the same deformation levels. This behavior is attributed primarily to the suppression of chain relaxation in proximity to the clay platelets and enhancement of deformation in the polymer phase in the vicinity of nanoclay platelets. Measurements of the clay orientation in the fibers in molten state (Fig 2) reveal that the anisotropic nanoplatelets contribute positively to the total birefringence of the fiber.
Thermal bonding of Polyolefin fibers

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Polyolefins are extensively used to make monocomponent and bi-component spunbond fabrics for hygiene and medical applications. Spunbond fabrics are predominantly based on homopolymer polypropylene (hPP) due to excellent spinnability, broad bonding window and good tensile strength/abrasion resistance. Monocomponent spunbond fabric based on polyethylene resins exhibit excellent softness and drapeability but lower tenacity/abrasion resistance and narrow bonding window compared to spunbond fabrics based on homopolymer polypropylene (hPP) resins.

This paper will describe in detail thermal bonding characteristics of polyolefin fibers and various parameters governing thermal bonding of polyolefins fibers. Properties of such fibers and resultant fabrics will also be described. The fiber and fabric properties will be related to structural characteristics of the polymers and fabrication conditions.
Superparamagnetic Flexible Substrates based on Submicron Electrospun Estane® Fibers Containing MnZnFe-Ni nanoparticles

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Estane®, a segmented polyester based segmented polyurethane that is elastomeric in fibrous form, was dissolved in dimethylene acetamide, DMAc, at a concentration of 20wt%. Superparamagnetic ferrite nanoparticles (ca. 14 nm) based on MnZnFe-Ni and dispersed at 10wt% in ethanol were mixed into the 20 wt% Estane® solution at wt% different concentrations of the nanoparticles. These solutions were then electro spun at 17-20 kV, 20 cm and 3 ml/h for 20 min to produce fine fibrous mats that were collected on a steel wire mesh target. After easy removal from the target, the fibrous mats were subsequently dried in a vacuum oven for 8h at 60 °C to minimize any residual solvent. The flexible mats were characterized in terms of fiber morphology and magnetic properties. Field Emission Scanning Electron Microscopy (FESEM) indicated that the diameter of these composite fibers was ca. 300-500 nm. Furthermore, the back-scattered electron FESEM images indicated regions where some agglomeration of the nanoparticles at higher wt% (ca 17-26 wt%) loading on the electrospun fibers was apparent. Characterization of the magnetic properties was conducted by measuring the induced specific magnetization upon application of an external magnetic field at 300 K. The induced specific magnetic saturation and the relative permeability was found to increase linearly (R²=0.999) with increasing wt% loading of the ferrite nanoparticles on the submicron electrospun fibers. A specific magnetic saturation of 1.7 – 6.3 emu/g and relative permeability of 1.8-6 (μr) at 300 K indicated superparamagnetic behavior of these composite electrospun substrates.

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Fiber Spinning Behavior of Biodegradable Polymers

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A variety of biodegradable polymers have been spun into fibers and their properties evaluated for their usefulness in nonwoven applications. The basic categories of inherently thermoplastic biodegradable polymers will be highlighted and reviewed. Special emphasis in the review will be placed on Nodax PHA, a polyhydroxyalkanoates hexanoate copolymer resin. The presentation will focus on the fiber spinning behavior and properties derived from fiber containing Nodax PHA, neat and combined with other biodegradable polymers. The Nodax PHA used in this study contained 11.3mol% hexanoate copolymer (H11). Polylactic acid and polyethylene succinate were also evaluated alone and in combination with the Nodax PHA material. Monocomponent Nodax PHA fibers were extruded initially, but it was found they would not solidify in the spinline even in the presence of high concentrations of nucleating agents. Bicomponent (sheath/core) fibers were melt spun using a H11 core and a polylactic acid (PLA) sheath, as well as a polyethylene succinate (PES) sheath. The spinning results showed that H11 levels up to 30wt% could be incorporated with stable spinning. The incorporation of H11 was found to be controlled by the slow primary crystallization kinetics of the H11 in the spinline, even when using high amount (>3wt%) of nucleating agents. The neat and bicomponent fibers produced with Nodax PHA were found to have tensile strengths similar to polypropylene. The PES based fiber compositions were found to have elongation-at-break values similar to PP. Neat H11 copolymer fibers were found to be readily biodegradable at 25°C under aerobic conditions.
MORPHOLOGICAL INVESTIGATION OF FIBROUS MATERIALS
BY UNI- OR MULTI-AXIALLY STRETCHED FILMS

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ABSTRACT

The fibrous materials formed from uni-axially stretched polyethylene blown films and sequentially stretched PTFE films have been used in industry for two decades. Other materials may possibly stretch and form the fibrous materials. In air filtration markets, usage of these two fibrous materials has been rapidly growing due to the capability of highly electrically charged or of making very fine fibers. These fibrous materials were investigated by means of optical and scanning electron microscopes. Theoretical approaches were attempted to understand basics of film stretching characterization.

THEORETICAL BACKGROUND

Let us consider the steady state elongational flow of a fluid sheet. Here we take ‘1’ as the machine direction, ‘2’ as the transverse direction and ‘3’ as the normal or thickness direction in a Cartesian coordinate system, and the velocity field in the sheet as:

\[ v_1 = v_1(x_1) \]
\[ v_2 = v_2(x_2) \]
\[ v_3 = v_3(x_3) \]

The rate of deformation tensor is given by:

\[ \dot{\mathbf{d}} = \frac{1}{2} \left[ \dot{\mathbf{v}} + \left( \dot{\mathbf{v}} \right)^T \right] = \begin{pmatrix} \frac{\partial v_1}{\partial x_1} & 0 & 0 \\ 0 & \frac{\partial v_2}{\partial x_2} & 0 \\ 0 & 0 & \frac{\partial v_3}{\partial x_3} \end{pmatrix} \]

For a Newtonian fluid, a constituitive equation may be expressed as

\[ \sigma = -pI + 2\eta \dot{\mathbf{d}} \]

Here \( \sigma \) is the acting stress, \( p \) is a hydrostatic pressure, and \( \eta \) is the shear viscosity. In general the stress tensor can be given as

\[ \sigma = -pI + \mathbf{P} \]

where \( \mathbf{P} \) is an extra stress tensor associated with deformation. We shall assume that the constituitive equation for the viscoelastic fluid can be represented adequately by the convected Maxwell model. Accordingly, we have
\[ \sigma_{ij} = - p \delta_{ij} + P_{ij} \]

\[ P_{ij} = 2\pi G d_{ij} - \tau \frac{\partial P_{ij}}{\partial t} \]

where \( \frac{\partial P_{ij}}{\partial t} \) is the convected derivative defined by

\[ \frac{\partial P_{ij}}{\partial t} = \frac{DP_{ij}}{Dt} - \frac{\partial v^i}{\partial x_m} P_{mj} - \frac{\partial v^j}{\partial x_m} P_{im} \]

and \( G \) is the elastic modulus and \( \tau \) denotes the relaxation time. For a stable multiaxial elongation flow,

\[ P_{ij} = \tau G \left( \frac{\partial v^i}{\partial x_j} + \frac{\partial v^j}{\partial x_i} \right) - \tau \left( \frac{\partial P_{ij}}{\partial t} - \frac{\partial v^i}{\partial x_m} P_{mj} - \frac{\partial v^j}{\partial x_m} P_{im} \right) \]

or

\[ \frac{\partial P_{ii}}{\partial t} = 2G \frac{\partial v^i}{\partial x_i} + \left( 2 \frac{\partial v^i}{\partial x_i} - \frac{1}{\tau} \right) P_{ii} \]

For a uniaxial stretching;

\[ \sigma_{22} = \sigma_{33} = 0 \]

In a biaxially stretched sheet, this is subject to the boundary condition;

\[ \sigma_{33} = -p + P_{33} = 0 \]

then

\[ \sigma_{ii} = -P_{33} + P_{ii} \]

where I may be ‘1’ and ‘2’ and

\[ \frac{\partial v^1}{\partial x_1} = - \left( \frac{\partial v^2}{\partial x_2} + \frac{\partial v^3}{\partial x_3} \right) \]

for uniaxial stretching

\[ \frac{\partial v^3}{\partial x_3} = - \left( \frac{\partial v^1}{\partial x_1} + \frac{\partial v^2}{\partial x_2} \right) \]

for biaxial stretching.
Generally, the biaxial stretching machine operates in the following manner:

\[
L_i = L_o + v_i t_i \\
L_{21} = L_o + v_2 t_2
\]

The rate of deformation may be given as

\[
\frac{dv_i}{dx_i} = \frac{v_i}{L_i} = \frac{v_i}{L_o + v_i t_i}
\]

where \( L_i \) is the total deformed length along the \( i \)-direction, \( v_i \) is the constant velocity along the \( i \)-direction and \( L_o \) is the initial length.

References

Fibrous Form of Bio-absorbable Polymers in Surgical Applications

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The bio-absorbable polymers, especially PGA, PLA and their copolymers, have received much attention from both academic and industrial researchers because of their biocompatibility and absorbability with biological tissues. A wide range of medical products has used those special polymers, such as bone screws and anchors, tissue scaffolds, sutures, drug delivery device, and etc. with variety of processing forms (molded, foamed, extruded and compounded). This presentation will address the fibrous form of bio-absorbable polymers and their applications in surgical field, e.g. sutures and meshes. The key biological and functional requirements from the bio-absorbable polymers and their fibrous form will be discussed.
Session 3 A
A surface having a water contact angle greater than 150° is called a superhydrophobic surface [7]. A water droplet easily rolls off of such an excellent hydrophobic surface and keeps the surface clean [1, 4]. This self-cleaning ability has been termed the Lotus effect although this phenomenon is observed on other plants leaves besides the Lotus plant leaves. Since the Lotus effect is a significant function of a biological surface, a number of models have been developed for the improvement in hydrophobicity of artificial surfaces and these techniques have resulted in numerous technical applications [6, 8, 10].

A hydrophobic surface can exhibit a contact angle up to 119° when the surface is flat and has a low surface energy. However, this surface can be superhydrophobic when it is roughened by mechanical or chemical modification [2, 3]. When the surface is roughened the minimization of liquid surface energy results in two possible contact angles, the Wenzel apparent contact angle and the Cassie-Baxter apparent contact angle. According to Wenzel, increasing the roughness of a hydrophilic surface increases its hydrophilicity and that of a hydrophobic surface increases its hydrophobicity [5]. This state describes a droplet of water that fills the cavities of a rough surface and remains even when the surface is tilted to a high angle. In contrast, Cassie and Baxter modeled a hydrophobic surface for which a water droplet does not fill the cavities. In this model, they determined the contact angle by considering only the surface area fraction of the highest points on the rough surface [9, 11].

In this research, short nylon fibers were electrostatically attached via the flocking process on a polyester surface upon which adhesive had been previously applied. This formed the substrate for a preparation of superhydrophobic rough surface. For the development of a superhydrophobic surface, an anionic polyelectrolyte, poly (acrylic acid) (PAA), was first grafted onto the nylon fiber surfaces to act as a mediator between nylon 6,6 and a low surface energy material. Next, 1H, 1H-perfluorooctylamine, was grafted onto the PAA that had previously been grafted onto the nylon [0] rough surfaces. The carboxylic acid groups of PAA covalently grafted to the amino groups of both nylon 6,6 and fluoroamine by a triazine-based condensing reagent. Finally, the water contact angles were measured.

Increasing the roughness of a hydrophilic surface increased its hydrophilicity as expected. Since nylon and PAA are hydrophilic, rough surfaces made of nylon or the PAA grafted nylon absorbed water into the structures and both surfaces became more hydrophilic. However, the rough surface became more hydrophobic when its smooth surface had a water contact angle less than 90°. The nylon rough surface grafted with a low surface energy material such as fluoroamine became superhydrophobic when the roughness and the top area fraction were in the possible design of the Wenzel and the Cassie-Baxter models. In this study, superhydrophobic surfaces were produced by a combination of mechanical and chemical surface modifications.
Literature Cited


Development of Orientation in Extruded Cellulose Fibers

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We have recently begun to investigate the extrusion of cellulosic fibers from ionic liquids (1,2) and other solvents (3). Cellulose from a variety of sources and various molecular weights has been extruded and properties measured. Properties obtained have been in line with those from commercial cellulose extrusion, and have varied in a reasonable manner with extrusion condition. These data are reviewed.

Since the main determinant of fiber strength is the molecular orientation in the fiber direction, it has been necessary to examine ways of determining that parameter. We have used birefringence and sonic modulus to examine orientation of molecules. Substantial work has been reported on the measurement of orientation previously by numerous authors (3) These techniques are reviewed. Initially sonic modulus was explored using a variety of available fibers including a couple of model systems. Then the technique was applied to commercial cellulose fibers and those extruded from ionic liquids. The relationships between these estimates of molecular orientation and the tensile properties of the fibers is explored and reported.

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Wet Spinning of High Molecular Weight Aliphatic Polyamides

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Introduction
Much effort has been concentrated on producing ultra-high modulus polymers [1]. It is well known that the highest elastic moduli reported for linear polymers are generally much smaller than their theoretical values. Only ultra-drawn, high molecular weight polyethylene, isotactic polypropylene and Kevlar showed moduli close to their theoretical values. The high modulus polyethylene fibers are obtained by gel spinning with an ultra high draw ratio of more than 200 times [2]. Polyamides are one of the most important commercial thermoplastic, particularly in the form of oriented fibers. Unlike polyethylene, polyamides have hydrogen bonds between molecular chains. These hydrogen bonds play an important role in making polyamides engineering plastics. At the same time, they prohibit high draw ratios in the polyamides, which have a maximum draw ratio of only 5 [3]. Aliphatic polyamide fibers achieve maximum moduli of about 1/20th of their theoretical value. Usually, polyamide fibers are obtained industrially by melt spinning, followed by drawing and heat treatment. Using this route, however, it is still difficult to produce tenacity and modulus above certain well-established levels, probably because of the low molecular weight of the parent polymer and the relatively low deformability of the crystalline polyamide fibers. Molecular weight has been shown to have a marked effect on the mechanical properties of oriented polymers, and is well known, generally, the higher the molecular weight, the higher the tensile strength [4]. However, it is very difficult to spin high molecular weight polymer via melt spinning route because the melt viscosity is very high due to the ultrahigh molecular weight of the polymer and the fluidity of a melt thereof is very low. Then if a higher spinning temperature is used to reduce the melt viscosity, a serious molecular weight drop occurs due to degradation. These problems have directed attention to a number of attempts that have been made for suppression of crystallinity or modification of the number and strength of the hydrogen bonds within the polymer crystals by using various processing techniques such as wet spinning, gel spinning, dry spinning, salts and Lewis acids for producing high strength/modulus polyamide fibers [5-11]. In the present study, the wet spinning method of high molecular weight polyamides also along with the use of Lewis acids has been employed in attempts to prepare high performance polyamide fibers.

Experimental
Nylon 6 chips (High~65,000, medium~50,000 and low viscosity~30,000 g/mole) were supplied by BASF Corp. High molecular weight nylon 6,6 (Mw=190,000 g/mol) was prepared by using solid state polymerization with commercial nylon 6,6 chips (Zytel®101, Mw=30,000 g/mole), which were supplied by Du Pont Corp.. GaCl₃, formic acid and nitromethane was obtained from Aldrich Co. The dry-jet wet spinning method was used for producing fibers. For complex solution concentration was 4 wt. % and for formic acid solutions 40 wt. %. For complex solutions coagulant was isopropanol and for formic acid solutions 10 wt.% of formic acid in water.

Results and Discussion
The following table shows some preliminary results for N6 and N66 fibers prepared from complexation with GaCl₃. SR is the GaCl₃:amide bond stoichiometric ratio.
Table 1. Physical properties of regenerated N6/GaCl3 and N66/GaCl3 fibers

<table>
<thead>
<tr>
<th>Sample</th>
<th>DR</th>
<th>Young’s Modulus (GPa)</th>
<th>Tenacity (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N6 (HV, SR=1:0.8, C=6%)</td>
<td>6</td>
<td>15.2</td>
<td>0.07</td>
</tr>
<tr>
<td>&quot;</td>
<td>5</td>
<td>18</td>
<td>0.09</td>
</tr>
<tr>
<td>&quot;</td>
<td>4</td>
<td>19.8</td>
<td>0.07</td>
</tr>
<tr>
<td>N66 (Mw=190,000 g/mole, SR 1:1, C=4.5%)</td>
<td>5</td>
<td>1.4</td>
<td>0.08</td>
</tr>
<tr>
<td>&quot;</td>
<td>4</td>
<td>2.3</td>
<td>0.06</td>
</tr>
<tr>
<td>&quot;</td>
<td>3</td>
<td>1.4</td>
<td>0.04</td>
</tr>
<tr>
<td>N66 (Mw=190,000 g/mole, SR=1:0.9, C=4%)</td>
<td>10</td>
<td>12</td>
<td>0.11</td>
</tr>
<tr>
<td>&quot;</td>
<td>8</td>
<td>12.2</td>
<td>0.10</td>
</tr>
<tr>
<td>&quot;</td>
<td>7</td>
<td>19</td>
<td>0.12</td>
</tr>
<tr>
<td>&quot;</td>
<td>6</td>
<td>11.1</td>
<td>0.09</td>
</tr>
</tbody>
</table>

References
The solvent properties of ZnCl₂ solution toward isotactic poly(acrylonitrile) (PAN) were studied by using a laser-optical instrument, FT-IR and Raman spectroscopy. This solvent showed an excellent solubility toward isotactic PAN. The best salt concentration for wet spinning was in a quite narrow salt range (56-60wt%), however. In the inverse plots of dissolution temperature (Tₜₜ) against the isotacticy, a breaking point appeared, which was connected to the existence of two different dissolution mechanisms. This breaking point did not appear for organic polar solvents, such as N,N-dimethylformamide (DMF) and dimethyl sulfoxide (DMSO). An extraordinarily large shift of the CN band to a higher frequency (2249→2276cm⁻¹) in FT-IR spectra was observed, which was explained in terms of the coordination bonding of ZnCl₂ toward CN groups. The significance of the coordination number (n) of H₂O molecules around a ZnCl₂ molecule was suggested from both a numerical calculation and CN band shift. Physical meaning of Tₜₜ master curve, which appeared in the plots of Tₜₜ against isotacticy as a function of salt concentration, was discussed in relation to the derivation of dissolution criterion of isotactic PAN in this solvent.
To enhance or add new properties to synthetic fibers, nanometer-sized particles of various shapes are used to produce nano-composites to be melt-spun into fibers. The materials of interest include functional nanoparticles, carbon nanotubes, natural nanofibers, and a variety of polymers. Our bicomponent plant enables the production of fibers consisting of two different polymers at laboratory scale with a throughput of up to several kg/h. Distinct features can be imparted to these fibers in taking advantage of functional properties of the sheath component and mechanical properties of the core component. Ongoing projects include TiO$_2$ nanoparticle doped photocatalytic fibers, nanocomposite controlled-release fibers, cellulose nanofibril reinforced biofibers and carbon nanotube reinforced LCP fibers (Fig. 1).

**Fig. 1: Melt-spun bicomponent nanocomposite fiber**

Photocatalytic activity of TiO$_2$ is well known (Kaneko & Okura, 2002). We produce enhanced TiO$_2$-based nanoparticles and nanocomposites to be melt-spun into photocatalytic fibers that eliminate malodor with light. Silver metal is well known to exhibit antibacterial activity (Yeo et al., 2003). Our respective project aims to assess the feasibility and application effectiveness of antimicrobial silver-silica nanopowders in polymer fibers. Technical challenges to be addressed relate to the incorporation of the nanopowder within various polymer matrices, characterization of the antimicrobial performance of the polymer composites, and assessing the influence of the additive on the fiber properties.

For the reinforcement of polymers, cellulose fibrils with diameters below 100 nm have been mechanically separated from wood pulp. Cellulose fibril reinforced biopolymers are favorable when biodegradable fibers with a high tensile strength are required, e.g. for medical applications (Zimmermann et al., 2004). Due to microstructural alignments between nanotubes, low concentrations of carbon nanotubes can enhance nucleation, crystal growth and, as a consequence, tensile properties of polymer nanocomposites (Zhang et al., 2004). The question of interest is if the tensile properties of liquid crystal polymers (LCP) fibers, where the molecular chains become highly oriented simply by subjecting them to shear and extension forces via melt extrusion, can be improved by adding carbon nanotubes.
In this study we present techniques that provide new knowledge on matrix-particle interactions (dispersion, orientation and migration of nanoparticles during melt-spinning), on how incorporated nanoparticles influence the morphology of polymer composites during melt-spinning into fibers, and how they affect the properties of produced mono- and bicomponent fibers.

**Literature**


In late 2002, Dow commercially launched Dow XLA* elastic fibers based on polyolefin elastomers. The unique performance advantages over the market incumbent, such as soft stretch, chemical resistance, thermal resistance and dimensional stability, are being validated with commercial success. In this talk, the properties of different types of elastic fibers will be compared. The difference in fiber properties will be rationalized based on the difference on molecular chain architecture and the solid state structure (orientation and morphology) of fibers.

* Trademark of The Dow Chemical Company.
Elastic Nonwoven Fabrics from Specialty Polyolefin Elastomers

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Vistamaxx™ Specialty Elastomers are a new generation of metallocene catalyst based specialty polyolefin elastomers (SPE) that can be processed in conventional spunmelt equipments to produce elastic nonwoven fabrics. The elastic nonwoven can be used in several applications such as personal care, adult incontinence, medical and industrial applications. SPE grades of varying melt flow rate have been developed for both spunbond and meltblown applications.

This paper examines properties of nonwoven fabrics comprising blends of SPE and an isotactic polypropylene (PP) oven an extensive blend ratio, and processed in conventional spunmelt equipment. The SPE grade selected for this study is an 80 MFR spunbond grade, while the isotactic PP is a 36 MFR commercial grade used extensively in spunbond applications. The blends were tumble blended and introduced directly into the extruder.

The processability of SPE is very similar to conventional polyolefin resins such as PP. At varying blend ratios of SPE to PP, fabric samples were made with changes in draw force and calendar bonding temperature. Fiber diameter is modified through either change of draw force at a constant blend composition, or by the addition of PP at constant draw force, both of which influence the tensile and elastic properties of the nonwoven fabric. For both SPE and SPE/PP blends, increasing bonding temperature from 75 ºC to 125 ºC diminishes tensile strength and elongation, while minimum bonding pressure is found to be optimal for achieving enhanced physical properties.

Non-linear regression models were constructed to correlate the change in physical properties with factors such as SPE/PP blend ratio, draw force, and calendar temperature and fabric basis weight. These models offer robust predictive capability over the design space in which the experiments were conducted. The surface feel properties of the fabric are also effectively tailored through the addition of PP, however at the expense of elastic performance.
Session 3 B
A clothing modeling framework for uniform and armor system design

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A relatively new motivation for studying the effects of clothing on a wearer’s physical performance capabilities is the analysis and design of military uniform and body armor systems. Toward this end, a clothing modeling framework, for quantifying the mechanical interactions between a given uniform or body armor system design and a specific wearer performing defined physical tasks is proposed. The modeling framework consists of three interacting modules: (1) a macroscale fabric model; (2) a collision detection and contact correction module; and (3) a human motion module. The macroscopic fabric model utilizes a large deformation continuum-degenerated shell theory representation. The collision and contact module enforces non-penetration constraints between the fabric and human models and computes the mutual contact forces between the two. While the human motion in this study is generated by imposing motion capture data onto kinematic human models of specified anthropometry. For captured human motion data, the simulation framework quantifies the contact forces exerted by the clothing system on the human and the internal deformations and stresses in the fabrics. These quantities are then integrated over space and time to estimate the amount of resistance that the closing exerts on the wearer during the specific activity considered. Current results from the framework are presented and the extended potential applications are discussed. Some extensions of the proposed framework are suggested and the key challenges involved are outlined.

Keywords: fabric modeling; structure-properties of textiles; contact, finite element methods, armor design

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TENSION AND BENDING PERFORMANCE OF SINGLE MUSCLE FIBER IN THE PIG
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Muscle fibres are not static structures, but easily adapt to altered functional demands, hormonal signals, and changes in neural input. Muscle fibres, once considered fixed entities, are in reality, dynamic structures with an extraordinary adaptive potential. Their dynamic nature makes it difficult to categorise them into distinct units. Their phenotypic profiles are affected by innervation/neuromuscular activity, exercise training, mechanical loading/unloading, hormones, and aging. Fibre types in pig muscle will probably be investigated with more advanced and sensitive techniques. Furthermore, single fibre dissection and quantitative loading and unloading analyses may increase the knowledge about metabolic and tension properties of the muscle fibre. In our recent research, the effects of Mechanical loading and unloading have been studied using tension and bending modes.

A unique characteristic of skeletal muscle is its diversity, which is created by its design, its fibre type, or cell type, composition and the heterogeneity of individual fibres (cells). We know that no skeletal muscle within an animal is identical with a second. Homologous muscles exhibit differences in fibre composition between species and between strains. Striated skeletal muscles are responsible for body and they are composed of multinucleated muscle fibre cells, may extend the entire length of the muscle and are arranged in myofibrils. Each myofibril is constructed of two types of longitudinal filaments. The thick type contains mainly the protein Myosin consists of two identical heavy chains and two pairs of light chains. Small globular projections at one of the heavy chains form the heads, which have ATP binding sites as well as an enzymatic capacity to hydrolyse ATP. The thin filaments contain the proteins actin, tropomyosin, and troponin.

The main components of muscle tissue in the pig are water, protein and lipid. Lean muscle tissue from a typical porcine muscle after rigor mortis consists on average of 75% water, 22% protein and varying amounts of lipid and carbohydrates. The muscle proteins can be classified into three main groups: myofibrillar, sarcoplasmic, and stroma proresponses teins. The myofibrillar proteins constitute the largest fraction and represent about 60% of the total protein content. The sarcoplasmic proteins represent about 30% of the total proteins. This fraction consists of myoglobin, which is a muscle pigment, and many different enzymes involved in energy metabolism. The stroma proteins represent about 10% of all proteins. The main components of the stroma probasis teins are collagen and elastin, which are the main components of connective tissue.

The muscle fibres used in this study were obtained from a female Large White pig. The pig was slaughtered at the usual commercial body weight of 100 kg by electrical stunning and exsanguination, in compliance with Chinese national regulations applied in commercial slaughtering. The samples were immersed in ice-cold skinning solution, separated into small bundles (70–150 fibres, ~10 mm long) and stored in skinning solution, containing 50% glycerol, at −20°C for 3–4 weeks. Single fibres were dissected under a stereomicroscope and pinned down at both ends. Fibres were treated with relaxing solution containing Triton X-100 (1%, v/v) for 30–40 min at 2–4°C to ensure complete removal of the sarcolemma and sarcoplasmic reticulum. A segment 5–6 mm long was cut from the fibre and the clips were mounted at its extremities for attachment to transducers. The force transducers used were strain gauge force transducers. The bending behavior and bending modulus of the muscle fibres have been measured by an axial-buckling method developed by the authors, which uses the fiber compression bending analyzer (FICBA). The bending properties of single fibers were quantified by calculating the equivalent bending modulus and the flexural rigidity by measuring the protruding length and diameter of fibre needles and the critical force, $P_{cr}$, obtained from the peak point of the force-displacement curve.
A fabric is composed of yarns and a yarn is composed of fibers. Ballistic penetration resistance is determined by 1) fabric topology, 2) yarn structures, 3) fiber strength and modulus and 4) yarn-to-yarn and fiber-to-fiber frictions. In this paper, an explicit digital element approach is developed to simulate the ballistic penetration process through textile fabrics. The approach enables investigation into fabric ballistic penetration resistance based upon the aforementioned fundamental physics. The paper includes three parts. Firstly, the explicit digital element approach used in dynamic simulation is explained. Then, a dynamic digital element relaxation procedure with periodic boundary conditions is developed to generate the detailed fabric geometry. Finally, the explicit digital element approach is used to simulate ballistic penetration of textile fabrics.

**Explicit Digital Element Approach**

A textile fabric is composed of many yarns and a yarn is composed of many fibers. In the digital element approach, a fiber is modeled as a digital chain. The digital chain is composed of many rod-elements, defined as “digital elements”. Frictionless pins connect rod elements. As the length of rod elements approaches zero, the digital chain becomes fully flexible. Each pin is modeled as a node in digital element analysis.

The contact of digital chains is modeled by contact elements. When the distance between two chains is smaller than the diameter of the digital chain, a contact element is added between them. The contact element can support both compressive force and shear (friction) force.

A yarn is modeled as an assembly of digital chains. In reality, a yarn is composed of hundred of fibers. In numerical simulation, 1-50 digital chains are employed to represent a yarn.

A numerical algorithm for an explicit digital element approach is spelled-out for dynamic simulation of textile fabrics. The process divides into three steps as shown in the following equations:

\[ m_i a_i = f_i , \quad \Delta v_i = a_i \Delta t , \quad u_i = v_i \Delta t \]

where \( i \) denotes the nodal number, \( f_i \) the nodal force, \( m_i \) the nodal mass, \( a_i \) the nodal acceleration, \( v_i \) the nodal velocity, \( \Delta t \) time interval and \( u_i \) the nodal displacement.

**Generation of Textile Fabric Geometries**

The geometry of a textile fabric is generated using a dynamic relaxation approach. The procedure is shown in Figure 1. The picture on the left includes nine unit cells. The unit cell in the center is called the primary cell. The other eight are images of the primary cell. An initial micro-geometry is first established based upon fabric topology. A small initial-fiber-tensile-strain is applied. During each time step, nodal forces applied to the primary cell are calculated. The explicit code is used to calculate both nodal acceleration and displacement of the primary cell. A periodic boundary condition is adopted. Then, nodal positions of the primary cell are mapped to the eight image cells. Thus, a time step is completed and a new time-step starts. The process continues until all nodal forces and yarn
tensions vanish. As a result, a relaxed unit cell is generated. In order to prevent fabric oscillation, approximately 50%-75% of kinetic energy is removed during each time step. During the whole relaxation process, unstressed lengths of all fibers within a yarn must be identical.

![Figure 1 Generation of Textile Fabric Unit Cell](image)

**Ballistic Penetration Simulation of Textile Fabrics**

The explicit code is used to simulate the ballistic penetration processes of 2-D woven fabrics. Firstly, it is assumed that the failure strain of the fiber is $\infty$, i.e. fibers don’t fail. Bullet initial velocity is 800m/s. If the friction coefficient is small, the bullet penetrates through fabric by pushing fiber aside, called “wedge out”. Figure 2 shows a complete “wedge-out” process. Penetration resistance is the function of friction coefficients. The greater is the friction coefficient, the greater the penetration resistance. The effect of friction coefficients becomes much less significant when fibers fail at a strain of 4%. The bullet penetrates fabrics due to fiber failure. In order to examine the digital mesh effect, 1, 2, 5, 10, and 19 digital chain yarn models are used in numerical analysis. The friction coefficients between fibers and the friction coefficient between the bullet and fabric are assumed as 0, 0.1, 0.3 and 0.5, respectively. Details will be presented in the presentation.

![Figure 2 “Wedge-out” Penetration Process](image)
Transplanner Water Transport Tester for Fabrics

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This paper reports on an instrument suitable for measuring the water transport behavior in the trans-planner direction for all kinds of woven & nonwoven textiles and paper materials. The instrument is unique in that it can measure the initial absorption in the trans-planner direction of the entire fabric surface tested as well as the amount of evaporation from the fabric surface after complete wet saturation at various water temperatures as per the end use of the materials. This instrument can measure the absorption for various kinds of liquids depend on the end use of materials. This instrument can overcome the drawback of various other principles like spot test, strip test and siphon test and give results in more realistic in nature. It is also possible to compare the effect at various temperatures of the absorbing liquid on the initial absorption and surface evaporation for an individual textile or nontextile material as well as within different varieties.

The Instrument mainly comprises of two sections. One section kept on a sensitive electronic balance contains a small water reservoir, whose level of water can be adjusted & constantly maintained using a small flow-controlled water pump with the aid of water vessel placed underneath. Another section contained a perforated sample podium to simulate the human skin above another water vessel, attached with the water reservoir by means of a pipe. When a sample is placed on the sample podium, it absorbs the water and the amount is recorded in computer, with which the balance is attached.

The main advantage of this instrument is that, the water supply to the fabric sample is continuous by maintaining constant water level underneath the fabric sample, because of which the hydrostatic water pressure doesn’t change during testing and gives accurate measurement.
Hemp fiber preparation: Designing and calculating a full size production with Process-Oriented Analysis

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Introduction
The environment is loaded by the consumption of fossil sources of energy and materials. The use of regenerating raw materials in the industry could reduce this environmental impact. Therefore, the native raw materials are promoted in Europe and Switzerland by research and agricultural politics. They open new production opportunities and new sources of income, both for the native agriculture and for the local industry.

In contrast to the seed hair fibers of the cotton, the bast fibers are embedded as multi-cellular fiber bundles inside the plant. To process these fibers industrially, the fiber bundles must be extracted from the plants (decortication) and separated (fiber preparation).

Fiber preparation
The wet stages of cotton processing, such as washing, bleaching and dyeing, are mostly done on the fabric. In contrast, the wet processes for hemp occur at the very beginning of the processing chain, during the separation of the fibers from the bast. Therefore, a direct comparison between the fiber costs of hemp and cotton is not meaningful. The drying process following any wet treatment represents most of the cost, for cotton as well as for hemp. Hence, it makes sense to combine the bleaching and dyeing of hemp with the fiber preparation. It should be possible for hemp to omit further wet processes and the associated drying processes thereafter.

Analysis and specification of production processes
The first step of designing a plant is the analysis and specification of the processes and resources needed to produce the hemp fibers. The main processes and the product flow are drawn.

In the next steps, the equipment, layout (space), operators (labor), and material and other resources needed are added to this model. Closed loops are introduced and completed.

In the flow and process specification, time, quantities, and costs are defined. Based on this, the costs for the product, the various resources, and the waste as well as the value added can be determined.

The following (Value) Flow Diagrams show the production process. All costs are calculated on the basis of 1 kg hemp fibers.

This figure shows three levels of detail of the hemp production.
Profitability and cost calculation

An industrial use of hemp fibers has only a chance if it is profitable for farmer to cultivate this plant, and if fibers of adequate quality and price for textile applications can be made available by a sustainable preparation process. It must be possible to process these fibers according to the cotton spinning process.

The industrial production for hemp fibers (preparation) should be calculated to see, how much the product will cost. Answers for questions like the following are of interest:

- What are the costs for the hemp fibers relatively to other short stable fibers?
- What are the costs relatively to other traditional manufacturing technologies for hemp and linen production?
- What are the costs for the hemp fibers produced by different fiber preparation versions?
- What are the primary cost drivers?

The Value Flow Diagram makes it easy to visualize and compare the costs of different versions of processing hemp.
Plasma Surface Modification of Textiles

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High frequency cold plasma, so-called low pressure plasma, is a very effective means of modifying surface properties of textiles while preserving the bulk properties. Different surface strategies can be used to modify textile surfaces: ablation (etching), surface activation (non-polymer forming plasma), and surface deposition (polymer forming plasma). Surface wettability is one of the most important properties for textiles. The hydrophilicity can be improved by incorporation of some hydrophilic groups such as –OH, –COOH, –C=O etc. on the surface using non-polymer forming gases. The introduction of polar groups is attributed to the improved soil resistance and a level dyeing as well. The modification was found to be less permanent due to restructuring of the polar surfaces. However, the modified surfaces can be used for subsequent processes such as lamination, coloration, composite textiles etc.

By contrast, ammonia with hydrocarbon gaseous mixtures facilitates to obtain nitrogen-incorporated-hydrogenated amorphous carbon (a-C:H:N) films. The deposited films strongly reduce hydrophobic recovery and preserve nitrogen-functionalities in the coating for a long time. Thus, a permanent functional coating can be obtained, which is one of the milestones of this work. As the coating contains polar functions, it alters the surface energy significantly from hydrophobic to hydrophilic. Addition of nitrogen contributes to nanoporous and crosslinked coating. The functionality in the coating facilitates respective dye-molecule access and form a dye-fiber bond. Hence, nanoporous a-C:H:N films facilitate to obtain a substrate independent surface dyeing which allow to characterize the surface properties such as accessible amino functionalities, coating purity and uniformity etc.

These findings introduce new advanced functionality in fabrics which can be potentially used in various fields such as incorporation of bio-molecules, proteins, UV absorbers etc. while preserving strong hydrophilicity. New added functions in fabric systems are desirable for the next generation of wearable chemical and biological protection.
Session 4 A
The Effect of Orientation on the Thermal Behavior of Nylon 6 Fibres

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Introduction
The thermal behavior in most material is simple. As the temperature rises, the material expands, and then in a certain temperature melts. With fibers, it is more complex. As the temperature rises, they may contract instead of expanding. This contraction may be reversible and, when a critical temperature is reached, it may become very pronounced. Temperature change can lead a material through different transitional phases. These can cause the specimen to expand and contract, to melt and re-crystallize or to go through major structural changes. During extrusion and further processing, stresses are imposed on fibers at elevated temperature, and some residual stresses remain as the fiber is cooled. If the fiber is heated, shrinkage is observed. The most interesting parameter in structural responses via mechanical measurements is temperature. The behavior of melt-spun fibers changes rapidly with changing temperature, particularly when the material undergoes a transitional phase. The application of thermo-mechanical analysis allows prediction of the strain response to any stress history and vice versa at varying temperature. Applications of thermo-mechanical analysis to the solution of complex problems in fiber-processing have been discussed [1]. Thermal behavior of lyocell fibres was reported by using TMA [2]. Observations of multiple melting, middle endotherms, crystallization peaks, etc., help to be aware of the different thermodynamic and structural interpretations of various melt-spun fibers by DSC. Ribnick [3] postulated that the thermal shrinkage of an oriented polyester yarn was composed of a very fast rate of contraction followed by monotonically decreasing rate, which eventually vanish. He proposed that the mechanism of thermal shrinkage is one in which the intermolecular bonds formed between neighboring extendedness chains become loosened by thermal energy, allowing very rapid contraction along the length of the chain, followed by moderation of shrinkage caused by intermolecular bond re-formation. As a man-made fiber is produced, a complex organization of polymer molecules takes place that involves developing both orientation and crystallinity. This organization results in molecular stress in the fiber. By raising temperature the polymer molecules may become sufficiently mobile to relieve the stress by disorientation and re-crystallization. This process occurs at the molecular level but is manifest macroscopically as fiber shrinkage. Consequently, measurements of fiber shrinkage may provide information about molecular structure. Nylon 6 fibers like other synthetic fibers often undergo a thermal treatment intended for purposes such as reduction or elimination of residual stresses, enhancing dimensional stability, improvement of physical properties in some process such as texturizing, dying, and etc. In this paper, the thermal behavior of nylon 6 with different degree of orientation has been studied by TMA and DSC.

Experimental
The filament fibers have been used in this study are non-oriented (NOY), partially–oriented (POY) and fully–oriented (FOY) nylon 6 fiber with 72, 40, and 19 denier count, respectively. Shrinkage measurements as a function of temperature were carried out using a Dupont 943 thermo-mechanical analyzer in standard atmospheric condition (%65±2 relative humidity and 20±2 °C). In the TMA, a fiber sample which is placed between two cleaved aluminum balls is held in tension by a probe assembly. Movement of the probe is linked via a linear variable differential transformer to a recorder. The TMA samples, 10 mm in length, were heated at 10 °C/min rate from room temperature up to approximately 250°C. The thermo-analytical investigations were performed by means of the Perkin Elmer DSC-4 differential scanning calorimeter. The samples of about 11 mg were crimped in an aluminum pan and then heated at 10°C/min rate.
Results and Discussion

Figure 1 shows the dimensional changes of NOY fiber heated from room temperature up to approximately 250°C. As shown there, the initial rate of thermal extension is high. The region of 60-92.86 °C exhibits no shrinkage that may be related to the Tg temperature. Polymers have almost dimensional stability in Tg temperature. With rising the temperature, the expansion is continued, because of high vibration within molecular chains and increasing distance between crystalline regions, so the rupture in the chains takes place. The length of the fiber has increased up to % 26 at 250°C. TMA thermo-gram of POY fiber has been revealed in figure 2. The fibers expand between 32.35-48.53°C up to %0. 2. Since there is no shrinkage in the region 48.53-57°C, it is supposed as the Tg region. The sample shrinkage begins after the Tg region in 57-78.68°C. After these regions the fibers have no dimensional changes. It is known that when a polymer is heated, the molecular chains loosen so the distance between them increase that termed as an elongation. When heating was continued, the vibration of the chains accelerates and it may be done on the contrary movement of each other that cause the shrinkage. In other hand, melting begins and the polymer chains loosen, thus elongation and shrinkage occurs at the same time, which balanced, and fibers have dimensional stability in 78.68-148.53°C[7]. The range of these temperatures is applicable for dyeing, finishing and etc. processes. As shown in figure 3, the TMA thermo-gram of FOY fiber exhibits some expansion and the Tg region occurs at 54.8-69.5 °C. The FOY fiber begins to shrink after the Tg region at 87.8-97.5°C. Since the FOY fiber is fully oriented, it has more ordered and perfected crystallites, the spaces of vibrating between crystals reduce and the fiber has lower shrinkage than that of POY fibers. All fibers were heated up to 250°C, NOY and POY were tolerated the temperature but FOY ruptured at 226°C. Khanna [4] studied about TMA thermo-gram of the oriented and un-oriented nylon 6 yarn. He reported that TMA result of oriented nylon 6 reveals shrinkage between the Tg and Tm due to the disorientation of the amorphous fraction as well as disorientation of the crystallites. Figure 4 shows the DSC thermo-grams of NOY, POY and FOY fibers. They reveal two endothermic peaks. The first represents the loss of moisture and the second is due to the melting. There is also a third endothermic peak which appears between those two and refers to as a middle endothermic (MEP). This peak represents melting of the crystallites formed in the amorphous regions of the fiber microstructure during the heating process. The DSC thermo-gram of POY fiber has smaller and narrower middle peak than that of NOY fiber. According to Khanna's [4] proposal it is related to the higher orientation of POY fiber. The DSC thermo-gram of FOY fiber shows three endothermic peaks, which the middle endothermic peak was being almost, disappeared. It might be responsible for high orientation and very low micro crystals in FOY fiber.

References

Fundamental Study of the Driving Mechanisms for Cross-Section Shape Change in Highly Noncircular Fiber Spinning

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Highly noncircular fibers with a precision microprofile are highly demanded in the emerging biochemical, biomedical, and telecommunication technology. As compared to a circular filament, a microprofile possesses second-order effects caused by its highly noncircular cross-section. The cross-section shape influences significantly the mechanical, physical, chemical, and biological functions of the fiber. However, highly noncircular shapes are difficult to produce using the current melt spinning technology. Figure 1 shows some examples of the typical shape change from the spinneret orifice to the fiber. It is thus the objective of this study to obtain fundamental understanding on the basic shape changing mechanisms in highly noncircular fiber spinning. With increased knowledge on the driving mechanisms, new processes or methods may be developed to alleviate or even suppress the driving forces for fiber shape change.

Figure 1: Shape change from die orifices (yellow) to extruded filaments (blue).

Die swell and surface tension are considered to be the main causes for shape change. There are two types of driving forces for die swell; one is velocity redistribution and the other is viscoelastic recovery. For a Newtonian liquid, die swell is caused by velocity redistribution (thus also called Newtonian die swell), while for a viscoelastic liquid, both mechanisms for die swell coexist. In the literature, the die swell effect, particularly the Newtonian die swell, is used widely for shape correction in profile extrusion with relatively a large cross-section, e.g. a window trim. For fibers/filaments with a much smaller cross-section, the surface tension effect becomes important. There have been very few studies addressing the understanding of the shape changing process in noncircular filament extrusion. Kim et al.\(^1\) studied the effect of Newtonian die swell on the fiber shape change, but their results did not even qualitatively predict the filament shape. Takarada et al.\(^2\) considered the surface tension effect and developed a semi-analytical solution which is able to show the correct trend of surface minimization. However, their approach is only applicable to simple cross-sections, i.e. rectangular shapes and annular shapes.

In this study, both analytical and numerical analyses were carried out to study the different shape changing mechanisms during noncircular fiber spinning. An analytical solution governing the shape change of an elliptical cross-section was developed, as given in Equation (1):

\[
\frac{d\chi}{dt} = \frac{\sqrt{\pi}}{2\lambda} \left(3 - 3\chi^2\right) \sqrt{\chi},
\]

where \(\chi\) is the ratio of the long radius over the short radius of the ellipse, \(A\) is the area of the ellipse, \(t\) is the time, and \(\lambda = \eta\sqrt{A}/\gamma\) is defined as the characteristic surface tension relaxation time. This characteristic time can be useful in estimating the representative time scale for shape evolution in non-circular fiber spinning when the surface tension is the main driving force.

For numerical simulation, a trilobal cross-section was chosen (with the characteristic dimension shown in Figure 2). Zero drawdown was applied. The effects of Newtonian die swell and surface
tension were studied and compared. A typical viscosity of 1000 Pa-s and a surface tension of 0.03 N/m were chosen. The simulation results are shown in Figure 2. The shape changes due to the two mechanisms are very different. For this specific case, the result from the surface tension approximates the actual microprofile shape very well except a smaller curvature in the center section. The surface tension continuously evolves the microprofile until the shape becomes almost circular after 10 s. From Figure 2B, the Newtonian die swell effect tends to enlarge the curvature in the center section. Combination of both effects offers better shape prediction.

![Figure 2: Comparison of the effects of die swell and surface tension on shape change: A (die orifice), B (die swell effect) and C (surface tension effect).](image)

References

The torsional behavior of human hair has been studied with the use of a single fiber torsion pendulum. Shear modulus and the average log decrement have been found to depend on the area of cross section of the fiber. Since human hair consists of cortex and cuticle, forming a bicomponent fiber, and since the cuticle is known to be more rigid compared to the cortex, this behavior can be expected. The apparent decrease of the shear modulus with the increase in cross-sectional area of the fiber suggests that the fibers with smaller cross section have a greater proportion of the rigid cuticle compared to the softer cortex. This has been confirmed from the measurements. The second aspect which is relevant is the dependence of shear modulus on fiber ellipticity. Using this property we were able to calculate the shear modulus of the cortex. Using this experimental background we are in the process of modeling the shear mechanical properties of hair as a function of ellipticity and the cuticle to cortex ratio. The extended abstract will have this information.
Optical hairiness- and profile-meter for flexible surfaces

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Summary

In this study, an optical device has been developed in order to evaluate hairiness and surface profile for a flexible material, mainly for textile surface analysis. This device is able to separate hairiness and structure information, therefore it can give information about hairiness or roughness according to the image processing.

1. Optical setup

When looking at a textile fabric with beam tangential to the material, the shadow of the structure relief and the hairiness can be seen. An edge extraction permits to eliminate DC-component and to obtain hairiness and fabric relief.

The optical setup implemented includes two parts: the drive of the sample and the optical assembly. The assembly includes a diode laser which illuminates the fabric with a zero angle incidence. An image with fibers appearing in dark on a bright background is then obtained. The use of a DC-stop in the back focal plane of a lens allows to remove the direct component of the image (it now appears with external fibers and structure relief in bright on a dark background) and also to attenuate strongly the low-frequency component of the image. The hairiness and relief information are focused with a lens and directed onto a CCD camera (Fig. 1a).

Therefore, structure and hairiness can be separated with an appropriate image processing.

2. Hairiness-meter

Hairiness is considered in two different ways: hairiness emerging from the structure and emerging fibers without considering the structure.
In the first case, a movie with about 200 images, like image from figure 1, is directly processed. The light energy is calculated line by line and reported onto a curve (Fig 2 in black). This curve shows the repartition function of the fibers in the structure. A statistical interpretation of this function is done. In the second case, the image processing determines the structure envelope and cancels it in order to keep only hairiness information (Fig 1b). Then, as previously, the light energy is calculated line by line and reported in a curve (Fig 2 in gray).

3. Profilemeter

In this case, the image processing is the exact opposite of the one above described: only the structure envelope is considered (Fig 1a in red). That is reported on a 3D graph (Fig. 3) with varying time. The standard topographical parameters are then computed.
Objective and Subjective Handle Evaluations for Disposable Diaper’s Top Sheets and Reusable Diaper’s Fabrics

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1. INTRODUCTION

Objective evaluation equations for fabric handle using basic mechanical parameters of the fabrics have been developed by Kawabata and Niwa1,2), and several equations for different end-uses have been used widely now3). Similar objective equations for Futon (bedclothes) fabrics are also reported by Matsudaira et al.4-6), and original primary hands for disposable diaper’s top sheets have also been defined by Matsudaira et al.7) such as “NAMERAKASA”, “SOFUTOSA”, “KOSHI” and “FUKURAMI” based on opinions of consumers. Objective evaluation equation for total hand value is also derived from those primary hands8-9).

In this paper, applicability of those objective evaluation equations are examined and compared with the results of reusable diaper’s fabrics.

2. EXPERIMENTAL METHOD

2.1 Primary Hands of Disposable Diaper’s Top Sheet

Primary hands of disposable diaper’s top sheets defined for their original primary hands are shown in Table 17). “NAMERAKASA” is the peculiar primary hand for the top sheet.

2.2 Samples

Top sheets samples (A, B, C and D) are representative samples used widely now (2003-04) in Japan for disposable diapers. Fabrics for reusable diapers are also representative plain weaves before use (E) and after repeated use (F) of 400 cycles (2 years used sample).

2.3 Calculation of the Primary Hands and Total Hand Value

Primary hands were calculated from the basic mechanical parameters measured by KES-System using the objective evaluation equation (DPCM-17)) for the original primary hands. Total hand values (THV) were calculated by DPCM-28) and DPCM-39).

2.4 Subjective Evaluation of the Primary Hand and Total Hand Value of the Top Sheet

In order to evaluate the primary hands and total hand value (=THV) of the samples, paired comparison method was carried out. To assure the results, the coefficient of agreement in paired comparison is calculated and examined. The coefficient means the degree of agreement between the subjects whether or not they are right in absolute sense. If the results are assured, the data are compared with objective data.

3. RESULTS

3.1 Subjective Evaluation

Results of paired comparison were checked their agreement and it was shown that all the results (four primary hands and total hand value) between judges agreed very well by the coefficient of agreement using Chi-square distribution. Therefore, it is worth to use these subjective values for comparison with objective data.
3.2 Objective Evaluation of Primary Hands

Results of a primary hand “NAMERAKASA” obtained by DPCM-1 equation are shown in Fig.1 comparing with subjective values. In the case of top sheets, although the absolute values are different, objective values decreased in the order of spunbond (B), air-through (A), meltblow (C) and spunlace (D) and this tendency agreed well with that of subjective values. Objective values of reusable weaves (E and F) showed extremely smaller values than subjective values. This means DPCM-1 equation is not useful for woven fabrics.

Table 1 Terms and Definitions of Original Primary Hands of Disposable Diaper's Top Sheet

<table>
<thead>
<tr>
<th>No.</th>
<th>Term</th>
<th>Definition</th>
<th>Remark</th>
<th>Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>NAMERAKASA (Smoothness)</td>
<td>A mixed feeling come from smooth and dry feeling having slippery touch without hitching to fingers.</td>
<td></td>
<td>28 %</td>
</tr>
<tr>
<td>2</td>
<td>SOFUTOSA (Soft feeling)</td>
<td>A soft feeling, mixed feeling of bulky, flexible and smooth feeling.</td>
<td>Same definition as HESC</td>
<td>26 %</td>
</tr>
<tr>
<td>3</td>
<td>KOSHI (Stiffness)</td>
<td>A feeling related to bending stiffness. Springy property promotes this feeling.</td>
<td>Same definition as HESC</td>
<td>21 %</td>
</tr>
<tr>
<td>4</td>
<td>FUKURAMI (Fullness &amp; softness)</td>
<td>A feeling come from bulky, rich and well formed feeling. Springy in compression with warm feeling.</td>
<td>Same definition as HESC</td>
<td>12 %</td>
</tr>
</tbody>
</table>

HESC*: Hand Evaluation and Standardization Committee in the Textile Machinery Society of Japan

Reusable weaves examined here showed quite a large value of SMD (13μm for new sample E and 12μm for used sample F) compared to that of top sheets (3 – 5μm). It is easy to know that large SMD brings smaller values of “NAMERAKASA” from the coefficient of DPCM-1 equation. Yokura also reported that diaper’s reusable cloths had a large value of SMD compared to top sheets. It is interesting that used fabric for reusable diaper has a larger “NAMERAKASA” for both subjective and objective values.

Results of “SOFUTOSA” are shown in Fig.2. It is clear that objective values did not agree with subjective values. Results of other primary hands and THV are shown in the presentation.
References

Experimental methods to evaluate the fiber length and orientation
distribution of long glass fibers in injection molded thermoplastics

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Long fiber injection molded thermoplastics have been proposed as a strong and lightweight material to be used in cars and light trucks, to reduce the fuel consumption, improve mechanical properties, eliminate corrosion, and reduce cost. Mechanical properties and dimensional stability of these composites depend on their three-dimensional fiber architecture. Characterization of fiber length and fiber orientation is crucial for validation of models and development of reliable parts.

In this paper, we develop procedures to measure long glass fiber length distribution in two different injection molded configurations (isoplacs and center gate disks). We evaluate the length of the fibers by burning-off specimens obtained from various locations in the samples, followed by image analysis. The burn-off experiments showed that these composites have several distinct regions, which causes different amount of elastic energy and porosity within the polymer matrix. The image analysis showed a severe attrition along the molded parts, but approximately 50% of the fibers are greater than 1 mm.

Traditional methods for determination of fiber orientation as well as a state-of-the-art non-destructive X-ray computer tomography (XCT) used in the course of an ongoing research will be discussed.
Session 4 B
Nephila clavipes can produce six kinds of silk. Among these silks, the dragline silk, produced by the major ampullate glands, has been the focus of most studies because of its impressive mechanical properties. Actually, the secondary protein structure in the dragline silk not only contributes to these mechanical properties, but also plays a significant role in the piezoelectricity and thermoelectricity of silk. In prior studies, we conducted some preliminary measurements of the piezoelectric response of dragline silk by employing a lab-made instrument. The piezoelectric signal of dragline silk fell into the range of 10 mv. In this study, we investigated the thermoelectric property of silk through thermally stimulated current (tsc). An axial anisotropy in the thermo-current was observed, presumably a consequence of a structural directionality within the dragline silk. Two peaks of thermoelectric current in the order of 10-12a presented in the temperature current spectrum, one in the range of 12-16°C and another at 81-85°C.
TEXTILE HEARTVALVE PROSTHESIS: *In Vitro Performances of the Fabric*

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INTRODUCTION

In order to replace faulty human valve, there are mainly two types of valve prostheses for which there’s currently good experience. Mechanical prostheses proved good durability but require long term anticoagulant medication for the patient. Biological valves respect the hemodynamic of the human, but their durability is nevertheless limited due to tissue degeneration. Next to these standards, surgeons are exploring heartvalve replacement without the need for open – heart surgery. The new, less invasive procedure, known as percutaneous, need a valve that is completely flexible as well as durable.

We describe a concept of heart valve prosthesis, based on the use of textile material. This material allows at the same time to have very low bending stiffness due to its discrete structure (essential for good fatigue resistance and thereby durability of the valve cusps during the cyclic opening and closing phases), and good orthotropical traction stiffness (essential for bearing the diastolic membrane stress during closing).

The goal of this work is to test the short and long term in vitro performances of a polyester fabric prosthesis.

MATERIALS AND METHODS

**Prosthesis manufacturing**

Tricuspid prosthesis is obtained by forming a fabric tube in a concentric way using three forming tools. The formed fabric is then heated over polyester vitreous transition temperature to fix the obtained geometry.
**Prosthesis testing**

*Valve performances*

The in vitro testing is done in term of static regurgitation (leakage versus pressure on closed valve) and dynamic regurgitation (leakage during closing). The performances of different fabric structures (weave, fabric saturation index (SI), yarn specific surface) are compared (under normal working conditions at 70 cycles per min) in order to assess the influence of textile parameters on the prototype in vitro behaviour.

*Fatigue testing*

In order to observe the fabric structure modification over time and its influence on the valve behaviour, a prototype with high bending rigidity (woven polyester used for vascular graft) is then tested in a accelerated fatigue testing device (at 5 Hz) over 10 Mio cycles.

**RESULTS**

*Valve performances*

In term of static regurgitation, we observe that in spite of a high saturation index, the satin weave, due to longer floats, is not enough floodtight. For plainweave, the higher the saturation index, the lower the leakage across the valve, and by same index, increasing yarn specific surface (microfibers) induces lower leakage.

Dynamic regurgitation is directly related to fabric bending stiffness. Satin weave as well as plainweave with microfibers yarns, offer low bending rigidity, leading to improved closing time (reduced closing volume). Increasing saturation index of plainweave leads to increased rigidity and delay in closing.

*Fatigue testing*

We observe that static regurgitation of the prosthesis increases much between 0 and 5 Mio cycles, showing that fabric relaxes over time under diastolic pressure (100 mmHg) becoming less watertight and less rigid. This is confirmed by a reduction of the closing volume (dynamic regurgitation) between 0 and 5 Mio cycles (faster closing of the valve).

Between 5 and 10 Mio cycles there’s only slight modification of the fabric structure. The material seems to become stable.
CONCLUSION

Fabric prosthesis shows encouraging behaviour in vitro (good flexibility, reduced permeability with adapted fabric parameters). By controlling manufacturing parameters and porosity of the material, future in vivo tests should lead to controlled tissue ingrowth, keeping the dynamic behaviour appropriate.

Fatigue testing shows that relaxing effect under pressure is to be considered when choosing the fabric structure so as to anticipate the phenomenon. First obtained results prove that fabric macroscopic structure is adapted to valve cyclic functioning. Tests currently in process at the level of the fiber, should assess the behaviour of the polyester material over time.
Phosphate Glass Fibres as Reinforcement in Resorbable Composites for Medical Implant Devices

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Introduction

Current resorbable medical implant devices are based on polymers such as PLA, PGA and PCL, sometimes self-reinforced, that can only provide low modulus materials of around 1-2 GPa. Compounding this, these polymers degrade through bulk hydrolysis, resulting in a rapid loss of mechanical properties before any significant loss of volume. As a result, current bioresorbable composites do not provide load-bearing support in the body. Structural implants rely on durable materials such as metal alloys and ceramics that exceed the modulus of parent bone tissue approximately tenfold. Because the bone structure constantly adjusts to local conditions, this tends to weaken surrounding tissues and can result in recurring damage after removal of the device.

Resorbable, high modulus glass reinforced polymer composites are garnering growing interest in application to medical materials. These materials offer potential as hard tissue implant materials to replace the use of durable components. These composites are produced using a resorbable polymer matrix and a resorbable or non-resorbable glass reinforcement and exhibit stiffnesses similar to cortical bone. Several resorbable polymer composites have been produced with Bioglass® reinforcement, a phosphate doped silica glass which has good bone bonding properties but that is essentially non-resorbable. However, silica-free phosphate glasses can be produced that possess appropriate stiffness values and are entirely resorbable. To date there are relatively few studies available on resorbable composites that have been produced using phosphate glass and only two have used long fibres of phosphate glass.

This paper reports on the development and testing of a range of phosphate glass fibres and their composites. Their mechanical properties, degradation rates and degradation profiles have been considered.

Materials and Methods

A wide range of phosphate glass compositions have been produced with varying content and type of low-toxicity metal cations including sodium, calcium, magnesium, zinc and iron. Phosphate content has been varied between 40 and 70%. The glasses were produced by melting phosphate salt precursors in platinum alloy crucibles at 1200°C. Formulations with promising degradation rates have been drawn into fibres using melt-drawing and pre-form drawing methods to provide continuous single filaments (see Fig.1). Single fibre properties have been determined using tensile testing methods (with Weibull analysis used to provide a statistical base) and interfacial sheer strength (IFSS) analysis. Bioglass® and E-glass fibres provided controls and comparisons to literature values have been made. Fibres have been used to produce PCL composite plaques by film stacking and in-situ polymerisation. Both unidirectional long fibre reinforced and random mat reinforced composites have been produced. Coupons of these have been subjected
to 3 point bending tests. The composites have been degraded for up to six weeks in deionised water and their mechanical properties monitored over this time. Fibre surface treatments have been undertaken using different coupling agents and their effect on IFSS and composite degradation rate assessed. Surface compatibilising treatments of the glass surface to improve coupling agent efficacy have also been considered.

Results

Single fibre testing provided tensile moduli for the phosphate glass fibres of between 20 and 50 GPa and tensile strengths of between 140 and 500 MPa (See examples Table.1). Weibull moduli are slightly below 4 for almost all of the phosphate glass compositions, indicating unpredictable failure behaviour. Composites produced using 40% Vf of the phosphate glass fibres exhibited moduli up to 19 GPa (c.f. bone 10-40 GPa) and strength up to 190 MPa (c.f. bone 90-140 MPa), with in-situ polymerised samples showing better mechanical properties. However, significant loss of properties (>50%) was observed after degradation in water for 7 days. Examination of recovered fibres revealed little change in fibre diameter but extensive breakdown of the fibres into short lengths. IFSS testing has shown that the coupling agent treatments significantly improve the fibre/matrix interface.

Discussion

The phosphate fibres demonstrated useful moduli which were close to what would be expected from literature but the strength offered scope for improvement. This was attributed to surface damage and moisture attack during drawing and subsequent testing and may explain the low Weibull moduli. This problem persisted in the composite leading to fragmentation which may suggest that water is attacking weak points in the fibre structure. Surface treatments of the glass fibres have been shown to provide significant improvement in the fibre/matrix interface. The quality of the interface is critical to the performance of the composite and is the focus for further research.

References

A Polyethylene fiber-reinforced epoxy matrix composite material is investigated for use as a multifunctional material in future space structures. The primary function of the material would be radiation shielding against biologically harmful radiation, with secondary functions including structural support and micrometeoroid impact resistance for space structures. Experimental results establish the composite as containing requisite properties, including mechanical strength, impact strength, and radiation flux reduction.

Although a significant progress has been made in developing efficient materials for space application, there is still a lack of protection against harmful galactic cosmic radiation (GCR). This limits the duration of human space flight, and must be remedied prior to engaging in long-term space flight missions. Composite materials offer a promising solution for space structure radiation shielding. The most effective radiation shielding is achieved by hydrogen rich composite materials, which is not present in metallics, but is relatively abundant in polymeric materials. Also, polymeric materials have the advantage of high strength to weight ratio, which is another advantage for space applications.

In this connection, polyethylene composite materials are being fabricated and evaluated. Polyethylene is the polymer with very high hydrogen content, and the strongest available fiber is made from polyethylene. Although the melting temperature of PE is low, for many space applications, this is not an issue. Composites from PE fiber in an epoxy matrix are produced and evaluated. PE fibers/fabrics impregnated with epoxy were obtained, and panels were prepared with a wide range of orientation arrangement of fibers in the matrix. These panels were vacuum bagged and cured in an autoclave. Fabricated composite samples were tested for a variety of mechanical properties, including tension, compression, shear etc. according to ASTM standards. The structures of these specimens were analyzed by microscopy and thermal imaging techniques. The samples were also evaluated for their radiation protection performance.

Polyethylene based composite materials showed high specific modulus, high strength, and a large reduction in radiation flux. These indicate the multifunctional advantage of these composites for space applications. Also, these can be fabricated fairly easily. Further evaluation of these materials is continuing to improve their performance in for use in extended deep space applications.
Nowadays, job classifications in which employees have potential for occupational exposure include dentists, dental hygienists, and staff with any instrument handling or patient care activities. An occupational exposure means a specific eye, mouth, nose, or skin contact with blood or saliva, other body fluids, human tissue, or contaminated instruments resulting from the performance of an employee’s job duties [FAY81]. As an example, even if there is no epidemiologic evidence to support HIV infection by saliva alone; in dental settings, saliva is considered potentially infectious because it frequently contains blood. Those risks can increase when using new device (high speed drills) which can increase non-percutaneous route susceptible to transmit the virus.

Gowns used for routine application in dentist care have always been considered as a relatively important workwear designed to fulfill many functions: easy disinfection, identifying or uniform appearance, protection against dirt and reasonable cost. They are usually made of routine apparel fabrics – polyester and cotton blends. But nowadays, the performance and protective levels of the gown must go up and there is a need of new Personal Protective Equipment (PPE) providing vital or safety protective function as well as comfort.

In this study we propose to address a list of these new constraints taking into account the new risks that the dentists face during its works, validating them through experimental experiences.

Based on these elements, improvement of the gown will be proposed. These improvements have to take into account the comfort aspect which is a factor not easily achieved.

We are faced to a dual demand: on one hand for an efficient bacteriological barrier, which necessarily necessitates a moister-repellent (hydrophobic) fibrous material, and on the other hand for optimum wearer comfort, which is provided only by a material which can allow heat and moister pass through it easily: in other word a hydrophilic material.

In a garment, there can be vastly different insulation properties depending on how its construction is carried out. The solution was to find a compromise by tailoring the gown so that the cut and make up presents protective (hydrophobic) zones where risks of contamination appears as critical, but with fit and openings of the garment able to create a micro-climate around the body which feels comfortable when the garment is worn.

First experiments were made to locate the impacts of drops susceptible to cause non-percutaneous cross-infection due to drill and other dental instruments and define the zones of the garment which are to be specifically protected.

The tested garments were commercially available routine cotton/polyester gowns currently used in Strasbourg dental hospital. They consist in two pieces: a jacket and a trouser.

The tests were firstly conducted using classical dental chair where water supply path was modified to dispense dilute dark dyes (successively food and reactive dyes). Dental drill was used on artificial denture (lab model) and real patient (fig.1). Image analysis of dye points on white dentist garment enables to precisely extract arms and torso zones with drops impacts lay-out.

Then, thermographical images, obtained under real conditions were made. They clearly confirm the zones previously defined (fig. 2).
Second set of experiments aimed to determine the mechanism of heat transfer through this specific clothing assembly as it appears as an important factor in enabling the development of clothing systems which helps the regulation and maintenance of thermophysiological balance.

Tests were conducted using wear trial protocols deliberately designed to reproduce usual conditions of activity and environment (dentist chair, light condition etc.) in order to provide comprehensive and informative data for assessing thermophysiological comfort.

The measurement of fabric surface temperature of the gowns was obtained via IR camera [BER05] in controlled environment condition. Thermographical images obtained (fig. 3 and 4) provide map of hot zones and valuable information of desired open space zone which will be designed for creation of preferential ventilation required for comfort improvement.

In parallel, some fabrics properties related with comfort such as air permeability, thickness and thermal resistance were measured using air permeability tester and Kawabata Evaluation Device [Y0084].

The new generation of routine dentist gowns can both meet protection standards and acceptable comfort. To achieve maximal comfort, improvement can to be drawn in garment design and fabrication which will provide good microbial protection in everyday practice.

The importance which will be attached to these aspects of comfort/protection in the next future is clearly dependent on the sales arguments of the companies marketing these products and moreover on the attitudes of the wearers themselves.

References
Synthesis of Cellulose Carbamate in Supercritical Carbon Dioxide

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Cellulose carbamate, an environmentally friendly material presents an interesting alternative to petroleum polymer because of its renewable, biodegradable, biocompatible, and efficiently alternative to the viscose process of producing fibers from cellulose, too.

In our laboratory, cellulose carbamates have been successfully synthesized from cotton linter pulps by supercritical CO\textsubscript{2}-assisted impregnation for the first time. In this method, urea was firstly impregnated into the cellulose pulp using SC CO\textsubscript{2} as a solvent and carrier at the ranging from 12 to 22 MPa and 50°C, followed by the esterification of cellulose at the temperature above the melting point of urea (132.7°C) for above 3h, resulting in cellulose carbamate. The nitrogen content of cellulose carbamates can be adjusted by the impregnating pressure, esterified temperature and esterified time.

At low pressures, solvent intensity of CO\textsubscript{2} was too poor to swell the cellulose substrate sufficiently to permit the rapid infusion of urea. At high pressures, CO\textsubscript{2} was a much better solvent for urea, and the partition of urea in the SC CO\textsubscript{2} fluid phase was also enhanced when it readily swelled cotton cellulose. However, higher temperature and longer time may result in the yellowing and pyrolysis of cellulose. So, we had chosen the temperature of 140°C as the practical esterification temperature, the esterification time of 4h. The effects of the impregnating pressure and esterification time on the nitrogen content of carbamate cellulose are shown in Fig. 1 and Fig.2.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figure1.png}
\caption{The nitrogen content of modified cellulose as a function of impregnating pressure after being treated in SC CO\textsubscript{2} for 6 h at 50°C.}
\end{figure}

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Fig. 2 The nitrogen content of modified cellulose as a function of esterification time at 140°C (Impregnation conditions: 50°C, 18MPa, 6h).

The structure and properties of cellulose carbamates were researched by Kjeldahl, Fourier transform infrared spectroscopy (FTIR), Wide-angle X-ray diffractometer (WAXD), $^{13}$C NMR spectrometer, and scanning electron micrographs (SEM). The results are shown in Fig. 3-6. It indicates that the production of cellulose carbamate esterification degree is high, the nitrogen content of it can reach 8.61 wt%. Compared with pristine Cotton linter pulps, cellulose carbamate becomes rough, loose and appears some imprint of violent bursting as a result of internal pressure, helical orientated along the direction of microfibrillar angle for 30°. Besides cellulose I, cellulose carbamate presents two types of cellulose, namely, cellulose II and cellulose III.

Fig. 3 FTIR spectra of the pristine cellulose (a) and cellulose carbamates with different nitrogen contents: 4.78% (b); 8.61% (c).
**Fig. 4** X-ray wide-angle scattering curves of the pristine cellulose (a) and modified cellulosics with different nitrogen contents: 1.98% (b) 4.78% (c) 8.61% (d)

**Fig. 5** $^{13}$C NMR spectrum of the pristine cellulose (a) and cellulose carbamates with different nitrogen contents: 8.61% (b)

**Fig. 6** SEM photographs of pristine (a) and esterified cellulose of cotton linter pulp (b).
Session 5 A
Correlation of Electrospun Fiber Structure to Jet Properties

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Micro- and nanofibers have been created from many different polymer solutions using the electrospinning process. This research will address the challenges of controlling and patterning electrospun fiber. Currently, control of electrospinning has been achieved primarily as a trial and error or Edisonian-type process, requiring offline and tedious electron microscopy imaging – e.g., scanning electron microscopy (SEM) or transmission electron microscopy (TEM) – to characterize the fibers. This research addresses techniques to quantify the jet properties and correlate them to fiber structure and process parameters. This will ultimately assist in development of patterned and oriented electrospun fibers.

The electrospun fiber diameter lessens 1000 times compared to the stable jet region at the pipette tip, caused by the instability or whipping motion of the jet. Therefore, the objective is to measure the fiber diameter, as well as the fiber velocity, prior to deposition on the target. It has been shown that by using a rotating disc as a target, the electrospun fibers will align along the rotational path. Figure 1 shows SEM images of electrospun 8wt% polyethylene oxide (PEO) in 80% ethanol and 20% water solution, on a 20 cm diameter disc at two different rotational speeds, 500 and 2000 rpm. As the target linear velocity approaches the jet velocity, fiber alignment improves, along with a reduction in and greater uniformity of fiber diameter. In addition, the SEM image in Figure 1 (b) demonstrates that as the target velocity (20 m/s) exceeds the jet velocity, some fiber breakage is evident.

Figure 2 – SEM image of electrospun PEO fibers on rotating disc with aluminum tape underneath the fibers. Disc rotation speed (a) 500 rpm, and (b) 2000 rpm.

In the literature, velocity calculations are typically based on conservation of fiber volume and diameter; hence, the evaporation rate of fiber-jet is mainly overlooked. Initial empirical studies, however, suggest that there is an inevitable conservation of mass within the jet and the evaporation rate. Figure 2 shows preliminary data of solvent evaporation versus time, which can be described as two regions. The first region, during the electrospinning, shows that as the distance between the target and the tip of the pipette (H) increases, from 9 cm to 16.5 cm, a greater mass loss, 55% to 80%, is measured. This mass loss is due to the greater time for solvent evaporation. The second region, demonstrates a more gradual evaporation rate after electrospinning, while the electrospun mat is on the scale.
Figure 3 – Electrospun jet/fiber mass change due to solvent evaporation versus time at two different disc height, 9 and 16.5 cm (3.5 and 6.5 in), (electrospun 8wt% PEO in 80-20 Ethanol-Water).

To obtain a more detailed analysis of jet properties and fiber structure, an online jet diameter measurement will be addressed – e.g., optical measurements and laser micrometer. Optical measurements are considered since they are non-contacting and do not interfere with the electrospinning process. These measurements, however, are limited, especially when the jet diameter decreases to nanometer dimensions. As an example, a laser micrometer with a high sampling rate to capture the unpredictable instability/whipping motion of the fiber-jet can be considered. These micrometers can measure a jet diameter as small as \(5 \pm 0.01 \ \mu m\). Note that during measurements, the jet will be considered continuous, since it has been observed and reported that the splaying or splitting of the jet rarely occurs during the spinning. These jet measurements can also be correlated with fiber diameter measurements from the field-emission scanning electron microscope and with pore size distribution measurements by porosimetry methods.

The objective of this research is to develop novel control methods and models for the charged polymer jet, its relation to fiber structure and process parameters. Electrospun fiber alignment has been achieved by Edisonian-type process and tedious characterization. To further advance the ability to control the fibers and fiber networks created, online jet diameter and velocity measurements will be addressed, as well as the relationship between porosimetry measurements and process parameters.

REFERENCES

Electrospinning has been used to produce polymer nanofibers with diameters ranging from a few microns to less than 100 nanometers. Due to mechanical oscillations of the electrically charged fibers during electrospinning, they are usually collected in the form of a non-woven mat without any significant fiber orientation. Recently new methods have been introduced with the goal of producing aligned arrays of polymer nanofibers [2-4]. These new methods usually require either a specially designed collecting electrode or externally applied electric field “lenses” to minimize the fiber oscillations.

We have developed a new method for making highly aligned arrays of polymer nanofibers by using an AC coupled DC field to drive the electrospinning process. This new “biased AC electrospinning” method can be used to deposit aligned arrays of polymer nanofibers onto virtually any substrate and without the need for an external “lens” electrode. Instead of the conventional DC voltage used in the electrospinning process, an AC voltage, biased to a DC value, is used as the driving potential. By varying the AC voltage, the biased DC value and the frequency of the AC voltage, the fibers can be aligned in any desired orientation on a conductive as well as an insulating substrate. This method eliminates the splaying and whipping effects observed in classical electrostatic fiber formation techniques. We have performed a systematic comparative study of biased AC, pure AC and conventional DC electrospinning. The applied AC voltage reduces the overall charge in the fiber during its formation, reducing the electrostatic repulsion forces responsible for the fiber instability and whipping. However, because the net charge on the fiber is reduced, the jet/fiber axial velocity is correspondingly reduced making it difficult to efficiently collect the fibers onto a substrate. Therefore, we DC bias the AC voltage in order to control and optimize both the fiber instability as well as the jet axial velocity. In this way, we are able to produce mats of highly oriented nanofibers with uniform diameter.

A 7% solution of Polyethylene oxide (PEO) in DI water was electrospun using AC with biased DC voltage, pure AC voltage and DC voltage. Figure 1 shows a series of SEM images contrasting the morphologies obtained under the three different processing methods. The fiber diameters are very uniform throughout the electrospun mat with sizes ranging between 100-200 nm. The DC bias value is chosen such that the bottom edge of the negative half-cycle of the AC voltage just crosses zero. This value of the AC voltage below zero potential is between zero and –2500 V. Also, for optimum fiber stability we have found that the AC frequency should be between 400 Hz and 1 kHz.
Similar results were obtained for PEO with carbon nanotubes, gold nanoparticles, and magnetic nanoparticles and also with polystyrene and polyisobutylene polymers.

We have found that it is also possible to produce advanced structures using the biased AC electrospinning method. For example, by shifting the angle of the target by 90° a double-layered wire mesh was obtained as shown in figure 2.

![Figure 2: Wire mesh of PEO with Single walled carbon nanotubes](image)

**References**

A mathematical model of the electrospinning process is being developed for both process control and property prediction. The electrospinning model focuses on the spinline stress critical parameters of solvent concentration, temperature, and viscosity. The descriptions of these parameters as a function of spinline position form the differential equations that describe the concentration, temperature, and stress profiles. The model utilizes an already published treatment of the electrostatic forces operating on the fiber. Characterization of the spin line stress profile is necessary for controlling the fiber solidification behavior. This, in turn, controls molecular orientation and is vital in achieving desired fiber diameter and morphology.

This unique electrospinning model was created based on the premise that the electrospinning process may, in many respects, be viewed as a variation of the dry spinning process. By integrating the electrostatic component of the process into an industry-proven dry spinning model, generation of the material profiles necessary for predicting fiber diameter, morphology, and uniformity has been achieved. Such a model should enhance process understanding and lead to improved process control strategies.

The model presented in this paper is a preliminary 1-dimensional electrospinning model. The fluid used was 27% by mass of cellulose acetate in acetone, modeled as a Newtonian fluid. The differential equations for the model where solved using a constant–step second order Runge-Kutta numerical method in FORTRAN.
Only a supplier who can offer its marketable products or services for competitive prices will have success on a long-term. The effects of internal changes concerning the product costs can be simulated best with computer programs based on a method like the Process Oriented Analysis (POA)\(^2\). POA has been developed and refined over the last ten years for the analysis of complex production systems. Its goal is to help engineers with conception, optimization, modernization, and maintenance. Existing instruments for the analysis of such systems with advanced technology that include all the technical and operational aspects are few in number and limited in performance.

POA investigates production systems on different levels of complexity. The analyzed production system can be an entire manufacturing plant, such as a sewing shop for garments, a production line for jeans, a single machine as for example a packaging machine, or a single process, such as weaving. This can be done either time-independently (statically) or time-dependently (dynamically). The concept of POA includes both viewpoints of the system, as well as the transition from one to the other. For this, the method comprises two diagram types in order to draw models for the static and the dynamic analysis.

Computer simulation belongs to the dynamic analysis part. Applied, for instance, on a typical weaving mill for towels, a simulation can answer two key questions:

- What are the possible reasons for substandard performance of production processes?
- Which amount of reserve capacity has to be made available on the different stages of the process chain, in order to maintain optimum productivity of the whole plant?

Only by the best available processes, it is possible to maintain a profitable production in a country with high labor cost. Since mass-produced goods are manufactured very economically in low wage countries, highly developed nations must bring in effect other advantages in order to upkeep the traditional industries, as textiles. This affords a wide range of high quality products, made just in time for a quickly fluctuating market. The quintessence of quick response on demand are small production lots. In the example of towel production, this means working either with quick style change systems and a large inventory of standard warp beams, or with short warps made to order. This second way has become more and more feasible since automatic sample warpers are offered on the market. A new alternative to those is the conventional sectional warper equipped for fully automatic operation. To find the best solution for making short warps, different procedures must be compared.

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Automatic Sample Warper</th>
<th>Sectional Warper with Automation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Manufacturer (examples)</td>
<td>Karl Mayer, Tsuzuki</td>
<td>Benninger</td>
</tr>
<tr>
<td>Characteristics</td>
<td>• cylindrical drum</td>
<td>• conical drum</td>
</tr>
<tr>
<td></td>
<td>• Rotating creel</td>
<td>• Stationary, conventional creel</td>
</tr>
<tr>
<td></td>
<td>• Limited number of bobbins</td>
<td>• Larger number of bobbins</td>
</tr>
<tr>
<td></td>
<td>• Up to 1500 m/min</td>
<td>• Up to 1200 m/min</td>
</tr>
<tr>
<td></td>
<td>• Little space required</td>
<td>• More space required</td>
</tr>
</tbody>
</table>

For this kind of optimization, computer simulation is a key tool. In the example given, an on-line calculation of operating cost is integrated to the simulation program, which leads to new insights in optimizing the performance of manufacturing plants.
In current textile dyeing water is used as a solvent for dyes. The large amount of water needed in the process (100kg water for 1kg textile Material) causes ecological as well as economic problems because of the wastewater treatment and an extra drying step necessary after dyeing the textile.

In order to reduce or even eliminate these disadvantages, it is proposed that water will be replaced by carbon dioxide as a solvent for dyes. In this new process high temperature and pressure are needed for dissolving the dyes. Under these conditions, the carbon dioxide is called supercritical carbon dioxide and, besides the reduction of water, has other advantages like high diffusion rates and low viscosities, which allows the dye to penetrate into the fiber shortening and improving the process. Moreover, by reducing the pressure at the end of the process dye and carbon dioxide can be recycled.

Reactive-disperse dyes for supercritical carbon dioxide dyeing has been specially designed and synthesized for this purpose. Besides their ability of being dissolved in supercritical carbon dioxide, they can react with the reactive site of the natural fibers forming a covalent bonding, which increasing the fixation between the dye and the textile material.

Several synthetic and natural textiles have been tried dyeing in supercritical carbon dioxide. The Laboratory scale showed that synthetic fibers were successfully dyed but natural fibers, like, cotton, wool and silk showed poorly dyeing. Here, In this article an attempt is made to review the advantages of Supercritical Carbon Dioxide Dyeing of Polyester.

Introduction

Since early 1990s, supercritical fluid dyeing that uses the supercritical fluid as an alternate solvent instead of water in conventional dyeing process had been arousing much interest in textile industry because of wastewater treatment. A supercritical fluid is a substance under pressure above its critical temperature. Under these conditions the distinction between gases and liquids does not apply and the substance can only be described as fluid (see graph no.1). The dyeing technique with supercritical fluid is an alternative one which has been developed without environmental contamination. Scholl Meyer and coworkers [1-4] had established the pioneering works for the process.
Conventional dyeing process of polyester fiber discharges much waste water that is contaminated by various kinds of dispersing agents, surfactants and unused dye. It is very difficult to treat the wastewater including many additives by the conventional process. The dyeing technique with supercritical fluid is an alternative one which has been developed without environmental contamination.

**Need of the Process**

In current textile dyeing processes, water is used as a solvent for dye. However, this causes environmental and economics problems. The water that remains after the process contains residual dye and its purification is costly. Furthermore, an energy demanding drying step is required after dying with water. Dyeing in supercritical (300 bar, 100°C) carbon dioxide instead of water solve these problems. The dye can be separated easily from the carbon dioxide and hence both can be recycled. No drying step is needed after the process. An additional advantage of working in supercritical carbon dioxide is that heat and mass is transported faster than in water. This makes the whole dying process potentially faster. Carbon dioxide (CO\(_2\)) can be successfully used as a replacement solvent for water in textile processing. Carbon dioxide can be successfully used in dyeing, applying UV stabilizer and for the application and removal of size. Large savings in energy and reduction in waste generated can be realized by switching to these green solvent.

Traditional Textile processing and production are based on aqueous processing. The use of water as the carrier solvent for processes, such as slashing (sizing) desizing, dyeing and finishing, consumes large quantities of water, results in large energy consumption for drying and results large volume of waste water requiring treatment. The non aqueous based Textile processing represents a radical redesign of the current process. The advantage of reengineering the process from the beginning is that a true “zero discharge” manufacturing operation can be achieved through the use of an environmentally benign, “green” solvent. Both liquid and super critical carbon dioxide can be used as a replacement solvent for water and exploit its property of exciting as a gas at room temperature. The substance which is used as a fluid in super critical carbon dioxide dyeing has critical temperature (T\(_c\)) and critical pressure (P\(_c\)) and which have particular values for particular substances, as shown in Table1.
TABLE 1
Substances useful as supercritical fluids, with critical parameters.

<table>
<thead>
<tr>
<th>Substance</th>
<th>Critical Temperature ($T_c$ / Kelvin)</th>
<th>Critical Pressure ($P_c$ / Bar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon dioxide</td>
<td>304</td>
<td>74</td>
</tr>
<tr>
<td>Water</td>
<td>647</td>
<td>221</td>
</tr>
<tr>
<td>Ethane</td>
<td>305</td>
<td>49</td>
</tr>
<tr>
<td>Ethene</td>
<td>282</td>
<td>50</td>
</tr>
<tr>
<td>Propane</td>
<td>370</td>
<td>43</td>
</tr>
<tr>
<td>Xenon</td>
<td>290</td>
<td>58</td>
</tr>
<tr>
<td>Ammonia</td>
<td>406</td>
<td>114</td>
</tr>
<tr>
<td>Nitrous oxide</td>
<td>310</td>
<td>72</td>
</tr>
</tbody>
</table>

By using this process the operating material and energy cost reduce & the efficiency of sizing & drying process is increased. This happen because of efficiently use of precious water stream.

**Supercritical Carbon Dioxide Dyeing Method**

A close system for supercritical fluid (SCF) dyeing was used to perform the dyeing on the polyester samples, which is shown in fig.1. The carbon dioxide is supplied to the system by a SFC or SFC/SFE grade carbon dioxide tank without helium head pressure. An ISCO model 100DX series syringe pump is used to fill and control the pressure of the entire system. The pump is programmable with the ability to control rate and levels of pressurization/depressurization. The ISCO pump jacket is cooled with a VWR scientific constant temperature bath to allow for the use of carbon dioxide tanks without a helium head. A HIP check valve to choose that there is no back flow that would allow the fluid to run from the system back into the pump. A T fitting divides the single line from the ISCO pump to allow for equal pressure at both the inlet and outlet ports of the recirculating pump. The process cell contains the polyester sample and the dye cell holds the disperse dye sample in a cellulose dye pack. The pressure block transducer holds a probe for monitoring the pressure and temperature inside the closed dyeing system.

A rupture cell creates a safety valve that will blow out if the system exceeds a pressure of approximately 500 atmospheric pressure. A three way pressure release valve allows the system to be vented. A micro pump corporation ultra high pressure fluid circulation pump model 1805 – C with leak free magnet drive head and variable DC drive is used to recirculate the fluid through the entire system to produce a flow from the dye cell through the polyester sample cell. Except for the insulated micro pump head and feed lines, the entire dyeing system is contained in a Yamato DN – 43 isothermal convocation ovens from Baxter scientific products. The oven temperature is monitored and controlled by a Watlow 1942 microprocessor base controller. Additional thermocouples are mounted on the inlet and outlet sides of the magnetic drive recirculating pump. A closed system for SCF dyeing was used to perform the dyeing on the polyester sample, fig.1. The carbon dioxide is supplied to the system by a SFC/SFE grade carbon dioxide tank without helium head pressure. An ISCO model 100DX series syringe pump is used to fill and control the pressure of the entire system. The pump is programmable with the ability to control rate and levels of pressurization/depressurization.

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The fabric to be dyed was wrapped around a dyeing beam, and the dye was placed in a vessel preheated to the desired dyeing temperature. After loading in a certain amount of fabric and dyes, the vessel was then sealed. A known amount of carbon dioxide was charged into the dyeing vessel with a carbon dioxide pump, and the vessel was pressurized to the desired pressure. Stirring in the dyeing vessel then start. Ethanol is added to the dyeing vessel as the co-solvent at the same time by a co-solvent pump. After one hour, a stop valve was slowly opened to release the carbon dioxide until the pressure of the dyeing vessel reached atmospheric pressure. After the dyeing, the fabric was removed, washed with 2gpl anionic soaping agent at 353 K for 5 minutes, and then rinsed in acetone at ambient temperature to conform dye fixing.

![Figure 1. Schematic of Supercritical Fluid Dyeing Apparatus](image)

**Impregnation and Dyeing from Supercritical Fluids**

In the process of impregnation, which is easily the converse of extraction, a substance is dissolved in the supercritical fluid; the solution flowed past solid substrate, and is a special case of this. A German patent was granted in 1994 for a process in which a dye, free of additives is dissolved in a supercritical Fluid ad the substrate to be dyed is suffused with this. A small production plant is now commercially available. The advantages of this process are:

1. That Contaminated waste water steams are not produced:
2. Dispersants are not required to solublise a disperse dye in water.
3. Solubility are controllable by pressure, allowing control of the dyeing intensity and color.
4. Diffusivities in the fluid are higher, making mass transfer in the fluid faster.
5. Take-up of carbon dioxide by the polymer fiber causes it to swell slightly giving faster diffusion within the polymer.
Advantages of Supercritical Carbon Dioxide Dyeing

That supercritical CO2 dyeing could offer many potential advantages over the conventional methods of dyeing. One of the most significant of these is the possibility of enhancing desirable fiber properties through the dyeing process, thereby creating an “improved fiber.” It has been shown that supercritical carbon dioxide is partially dissolved in polyester fiber producing a softener-like swelling effect. Supercritical CO2 has been shown to reduce the Tg of PET by 20 to 30 OC under typical SCF dyeing conditions. If the fiber properties are adversely affected as a result of morphological changes, the potential future commercial use of CO2 as a dyeing medium Supercritical sizing may tempt the industry to try a better -- cheaper, faster, smaller and cleaner -- method.

Furthermore, economic benefits include increased productivity and energy savings. Successfully commercializing supercritical fluid CO2 processing will improve the economics of dyeing and other textile chemical processes by eliminating water usage and wastewater discharges and increasing productivity by reducing processing times as well as required chemicals and auxiliaries and reducing energy consumption and air emissions. As a result, SC-CO2 processing will be more rapid, more economical, and more environmentally friendly.

Supercritical CO2 does penetrate and can have an effect on the fiber Structure. However, it was also evident, based on the total changes observed in the fiber Morphology and properties, that the medium used in the process, whether it be air, water or supercritical CO2, was not the critical parameter, rather, heat and tension were the critical parameters affecting the fibers during these dyeing experiments. Today it might be possible, choosing other exposure conditions, to significantly affect the fiber morphology during supercritical fluid dyeing.

Comparison between Conventional and Super Critical Carbon dioxide dyeing

Conventional dye- fiber reactions use water transport- medium, and result in:

1. Reactions with low reaction efficiency due to the competing hydrolysis hydroxyl ions water (hydrolyzed dye cannot react w/ fiber).
2. Environmental problems due to residual, untreated hydrolyzed dye present in effluent.
3. Contaminated and waste water requires water treatment and its purification is costly.
4. Drying is required.

Replacing water with supercritical fluids (SCF) as a transport medium can result in:

1. Eliminating toxic waste (no hydrolyzed by- product)
2. Lower costs for the entire dyeing process reduce operating, material, and energy costs.
3. Color fastness of fibers dyed in supercritical fluid will be similar to fibers dyed in water.
4. Color fastness of fibers dyed with disperse(non reactive) dye will be extremely low
5. Dye- fiber reaction (color fastness) will be directly proportional to the temperature increase.
6. The dye can separate easily from the carbon dioxide and hence both can be recycled.
7. Drying is not required.
8. The heat and mass transfer is easy; this makes the dyeing process potentially easy.
**Conclusion**

In the Textile industries their is relatively little practical experience in dealing with the high pressure used with super critical fluids, any SCF equipment is going to represent a relatively high capital investment.

At the present, SCF textile wet processing is completely limited to a batch process and is on a relatively small scale at that. In this technical environment the future success of SCF processing in the textile Industry will depend on developing a more extensive basic research understanding of SCF processes, expanding the focus of SCF preceding beyond coloration, the availability of a new generation of low cost pilot scale machinery and being able to add value to a product by using this technology.

**Acknowledgment**

First of all we would like to express profound gratitude to the management of the institute, Principal Dr. Ing.V.P.Singh, Advisor Prof. Dr. H.V.S.Murthy and Head of the department, and Prof. Dr. Prabhakar Bhat for giving encouragement and guidance to work on Supercritical Carbon Dioxide Dyeing.

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The Study of Improving the Tearing Strength of Coated Fabrics with Silicone

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The polyurethane (PU) has been used as coating materials on cotton fabrics. By measuring the results of different coating thickness on fabrics, the optimized way of coating and coating-amount were found and the principle about the tearing strength’s decrease of coated fabrics was studied also. The different proportion of mixing of the amino-silicone and the polyurethane has been adopted. Comparing to the water proof ness, moisture permeable and the tearing strength of these fabrics, the optimized proportion of silicone and PU was fixed and the phenomenon that the cotton fabric tearing strength will drop down after coating was effectively solved.
The removal of water-soluble effluents generated by textile industries is an important issue in wastewater management. Microbial treatment of environmental pollutants, including dyes, with white rot fungi has received wide attention as a potential alternative for conventional methods in wastewater treatment. Eight sulphonated azo dyes with similar molecular structures were selected and degraded by the white-rot fungus *Pleurotus ostreatus*. The decolorization effect was measured with a UV-spectrophotometer. Due to the different molecular structures of the dyes, the decolorization efficiency ranged from 70 percent to 90 percent after a 7-day treatment. Dyes with electron withdrawing group(s) on the benzene ring degraded quickly and had relatively high decolorization. Dyes with electron donating group(s) on the benzene ring had relatively lower decolorization. Dyes without functional groups on the benzene ring had relatively high decolorization, even though they were less affected in the first three days. The solubility of the dyes, as well as the difference in the solubility parameters of the molecular units that are connected to the azo bonds, affects the decolorization. The more similar the solubility of the compounds connected to the azo bonds, the better the decolorization.

Chemical instrumental analysis methods such as high performance liquid chromatography (HPLC) and capillary electrophoresis combined with electrospray ionization mass spectrometry (CE-ESI-MS) were used to identify the degraded products and to develop a relationship between the chemical structures of the dyes and fungal decolorization. The fragmentations of several sulphonated dyes were investigated and the mechanisms for these breakdowns were proposed. The fragmentation supplied important information which was used to analyze the structure of the compounds. Several major degradation products were isolated and identified by HPLC and CE-ESI-MS, and the corresponding possible degradation pathways were proposed.

In addition, in order to explain the degradation pathway, enzyme assays were performed. Characteristics such as enzyme activities and concentrations were determined and related to the effectiveness of decolorization.
Session 5 B
Radiative emission of electrically generated excited states and the dissociation of light-absorption generated excited states form a principle for polymer materials to be used in light-emitting and photovoltaic devices. The dispersion of carbon nanomaterials such as carbon nanotubes can significantly improve the light emission and photovoltaic response in polymer materials. However, it is very challenging to obtain the dual properties: light emission and photovoltaic response from a singlet polymer device because these two processes are mutually competing. We found that adjusting the dispersion of carbon nanotubes can lead to an effective control of the dual light-emission and photovoltaic response and consequently yield multifunctionalities from a singlet device of polymer carbon nanotube composite. This presentation discusses how to use nanomaterials to adjust the mutually competing properties: light emission and photovoltaic response in polymer materials.
In recent years the importance of the polymer light emitting diode (PLED) has grown immensely. They have proved very desirable in numerous applications due to very high efficiencies, low power consumption, and ease of fabrication. Typically, these devices have been constructed in a layered, thin film fashion consisting of electron and hole transport materials doped with a luminescent dye. By changing the dye it is possible to tune the emission of the PLED to particular wavelengths. However to produce a white light emitting PLED, simple blending of the various dyes with the host material results in drastically reduced performance due to energy transfer between the various dye molecules. To achieve maximum efficiency the dye molecules need to be separated by some distance. We propose here forming hole and electron transport materials and appropriate dye molecules into colloidal particles. Using a mini-emulsion method, it is possible to create individual colloidal particles that emit in the red, green, and blue regions of the visible spectrum. Single color and white light electroluminescent devices have been demonstrated upon appropriate mixing of the colloidal suspensions.

References

Flexible Electrochromic Devices using Inkjet Printed Electrochromic Layer

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Recent research efforts in the fabrication of electrochromic devices (ECDs) are directed toward fabricating micro patterned and self-addressable ECDs for high resolution display devices [1]. In order to realize this high definition ECD, the patterning of electrochromic materials is considered as a key technology. Among various patterning methods, inkjet-printing (IJP) has been recently attracted as a direct non-contact patterning method of materials because it generates high resolution patterns with at least amount of materials in high speed [2]. In this work, we present our endeavor to fabricate ECDs using inkjet-printed PANI-silica colloidal nanocomposites for electrochromic layers as described in figure 1. 300-400 nm size PANI-silica colloidal nanocomposites have been synthesized in an aqueous medium [3]. The resulting PANI-silica colloidal nanocomposites were printed by commercial desktop inkjet printer on the ITO coated PET (ITO-PET) film. Then, ECDs were fabricated using this inkjet printed PANI electrochromic layer and poly (ethylene glycol) (PEG) –LiClO₄ based polymeric electrolyte.

Figure 1. Schematic procedure of electrochromic device fabrication using inkjet printed electrochromic layer.

300-400nm sized PANI-silica particles were inkjet printed on ITO-PET film and formed 800nm thick electrochromic layer. The resulting electrochromic device using this inkjet printed electrochromic layer and PEG-LiClO₄ based polymeric electrolyte shows color changes corresponding to its different redox states. It exhibits green of the emeraldine form at neutral state (0V) and changes to blue at +0.7V as it is oxidized to nigraniline state. Then, it shows pale yellow at 0.8V as it is reduced to leucoemeraldine state. In addition, transmittance spectra of this ECD in the same potential range showed about 50% transmittance contrast ratio at 780nm (Δ% T780nm).

References

A novel method for constructing a white-light organic light-emitting device using a vinylcarbazole/oxiziazole functionalized methylmethacrylate (MMA) co-polymer system doped with fluorescent dyes was investigated. As a first step to achieving a colloidal light-emitting device with broad spectral emission, green emitting particles were used to fabricate devices. Colloidal particles consisting of the functionalized MMA co-polymer and fluorescent dye were synthesized by emulsion polymerization. Devices were constructed by spin-casting of colloidal particles into a close-packed monolayer. An organic capping layer was applied to fill imperfections in the monolayer. The composition and thickness of the capping layer was optimized for best device performance. Devices are characterized by their current-voltage curves and their luminance. Typical colloidal devices delivered a peak brightness of 4.5 cd/m², a luminous efficiency $\eta_L$ of $2.7 \times 10^{-3}$ cd/A and a luminous power $\eta_P$ of $3.0 \times 10^{-4}$ lm/W.

References

**Effect of the thermal history on the crystallization behavior of bacterial polyesters**

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**Introduction**: Poly(3-hydroxybutyrate) and its copolymers are produced by a wide variety of microorganisms as intracellular carbon and energy storage materials. It is well known that the melt processing these polymers into fibers and films is very hard because of their low Tg and low crystallization rate. Further the spherulitic crystallization gradually occurs after cooling resulting in the brittle character. Because of these problems, fine multi-filaments have not been prepared. The purpose of this study is to investigate the effect of the thermal history on the crystallization behavior of bacterial polyesters and to find the optimum melt spinning condition to prepare the fine multi-filaments.

**Experimental**: A homo poly(3-hydroxybutyrate) (PHB) and four poly(3-hydroxybutyrate-co-3-hydroxyhexanoate)s (PHBH) with various 3-hexanoate contents were used. Sample codes and various molecular characteristics are given in the table 1. The thermal properties were evaluated with DSC under N2 atmosphere. Melt spinning of PHBH were carried out with a laboratory scale screw extruder. Polymer melt extruded to the air at 15°C was taken on a bobbin placed 3 m below the spinneret. As spun fibers were then cold drawn at 80°C with a drawing machine. Fiber structure was determined by using a WAXD.

**Results and discussion**: PHBH showed a lower Tm than homo PHB due to 3-hydroxyhexanoate component as a contaminant for the crystallization and crystallized very slowly when they were cooled from 200°C, which is higher than Tm of homo PHB. However, when PHBH were cooled from a temperature lower than Tm of PHB (c.a. 180°C), the crystallization was significantly enhanced and the crystallization temperatures were increased as shown in Fig.1. These results indicate that some sort of higher order structure still remains in the PHBH melts at the temperature range between Tms of PHBH and homo PHB. Unfortunately, either the WAXD or the polarizing optical microscope did not detect any higher order structures at this temperature range.

When these PHBH were melt spun at 160°C, the fibers sufficiently crystallized and did not stick together. However, the cold drawing was only possible for high Mw PHBHs, PHBH-3H and PHBH-8H. WAXD study revealed that the high Mw PHBH as spun fibers show a oriented pattern, although low Mw PHBH are randomly oriented. These results gave two requirements for the melt spinning of PHBH. 1; Optimum melt spinning temperature has to be chosen so that the crystallization is promoted in the spinline, and 2; sufficiently high Mw polymer should be used so that the high tension is applied to the spinline and the fiber is consisted of the oriented crystal. Applying these conditions, we have obtained the fine multi-filament of PHBH-3H and –8H.

**Table 1 PHB and PHBHs used in this study.**

<table>
<thead>
<tr>
<th>Sample code</th>
<th>3HH content (mol%)</th>
<th>Mw (x 10^5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PHB</td>
<td>0</td>
<td>4.2</td>
</tr>
<tr>
<td>PHBH-3H</td>
<td>3.4</td>
<td>4.7</td>
</tr>
<tr>
<td>PHBH-3L</td>
<td>3.2</td>
<td>1.6</td>
</tr>
<tr>
<td>PHBH-8H</td>
<td>8.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>

**Fig.1 Crystallization temperatures of PHBHs cooled from various temperatures**
Polymer Brushes for Creating Fiber Surfaces of Controlled Structure and Technological Relevance

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A polymer brush is an ultra-thin layer composed of individual polymer chains, all attached by their ends to the underlying surface (substrate). Polymer chains can be selected from a wide range of chemical structures to provide the desired surface chemistry and physics. Any material can be used as a substrate, as long as the polymer chains can be attached to its surface in some way. Attachment of the chain to the surface can be achieved by a covalent bond between an active site on the substrate and a reactive group on the chain end, by formation of an ionic bond between a charged group on the substrate and an oppositely charged group at the chain end, or by formation of an adsorptive interaction between the surface and a segment on the chain end that is attracted to the surface of the substrate.

By means of polymer brushes, surfaces can be created that have the specific chemical and physical properties needed for specific applications. Polymer brushes that change surface topography patterns or change chemically in response to a control signal can be useful for biotechnology applications. Polymer brushes that trap or release designated species can be useful for homeland security applications. In applications of the future, fibers will be the preferred substrates for polymer brushes, because fibers have the flexibility and high surface area that are lacking in flat, monolithic substrates.

The major barrier to the achievement of versatile surfaces for specific applications up to now has been the prior absence of a clear understanding of the formation process and how it can be controlled. Recent research in our laboratory has resulted in a detailed understanding of this formation process.1-6 We have established that this formation process (1) does not agree well with the considerable amount of theory published well before experiments were done and (2) has aspects that are surprising and counter-intuitive. The importance of these findings is that they can be exploited to construct novel and useful polymer brushes of complex and controlled structure. In addition, these useful brushes can be constructed on fibers of all kinds, leading to versatile fibrous assemblies to solve current technological problems.

References
Posters
Functionalized Nanofibers for Defense Applications

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Electrospinning process is a fairly well established experimental method to produce fibers at the nano-scale range that enable them to find applications in filters, protective fabric liners, face masks, etc. More recently, adding functionality to nanofibers has gained increased attention from the research community. The objective of this research is to fabricate a novel nanocomposite material having higher efficiency in detoxifying chemical warfare agents, organophosphorous compounds and other toxic substances. Nanocrystalline metal oxides have been used to achieve these properties. Electrospinning of nanoparticle-polymer solution was carried out using a single step process. Results show that phase separation may occur enabling deposition of nanoparticles on the surface resulting greater adsorption and catalytic reactivity. Beside that, several other functional nanoparticles have been also used to fabricate nanofibers that will find applications as fire retardant materials, cellular scaffolds and sunscreen nanomaterials. Nanoclays (hydrotalcite and hectorite), chitosan and titanium dioxide have been added respectively to the nanofibers to achieve those functionalities. Nanofibers were characterized by using scanning electron microscopy (SEM), transmission electron microscopy (TEM) and x-ray diffraction (XRD) techniques. Nanofibers in the range of 50 to 250 nm have been reported here and the adsorption studies show their potential as self detoxifying nanofiber webs. The work will have enormous technological and social impact as it contributes to national defense and homeland security.

Figure 1: a) SEM Image of Functionalized Nanofibers b) TEM Image of a Single Nanofiber and c) TEM Image of a Metal Oxide Embedded Nanofiber

References


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Relationship between Cotton Breeds and the Comfort of Knitted Fabrics: A New Understanding

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One of the inherent characteristics that makes cotton “king” among fibers and enhances consumers’ appeal is its comfort characteristics. Comfort here does not refer to the psychological comfort or feel but to the physiological comfort such as the water vapor permeability (WVP) often commonly referred to as breathability. The rate at which water vapor moves through a fabric plays an important role in determining the comfort as it influences the human perception on the next-to-skin feel and the cool/warmth feeling. When a fabric allows the transport of water vapor at a faster rate, that fabric is said to be a breathable fabric. In other words, the faster a fabric breaths, the better is the comfort. This property has direct implications on the end-use applications, consumer appeal and sales value of the fabric. More importantly, in the case of cotton, its inherent characteristic is its comfort on which it is pre-sold to customers.

There have been a number of reports on the breathability characteristics of fabrics. However, there is no report on the relationship between different cotton varieties and the moisture vapor transport through knitted fabrics made from them. To the best knowledge of the authors, this paper reports for the first time the relationship between the fiber breeding origins, which determine the fiber variety and the breathability of knitted fabrics made from them. This study will have far reaching significance on the breeding of cotton for superior characteristics for targeted and end-use applications. This research effort opens-up a new line of thinking on the genetic understanding of fiber characteristics to tailor important end-use applications.

As the main objective of this study was to understand the influence of cotton varieties with varying breeding backgrounds on the breathability (WVP) of knitted fabrics made from them, it was thought logical to evaluate the WVP in griege form. Any further treatment to the knitted fabrics was thought to interfere with the study on the understanding of varietal influence on WVP values. Thirty one untreated single jersey knitted fabrics were knitted from yarns spun from different cottons which include the most popular US cotton varieties grown in Mississippi, Georgia and Texas during the crop years 2001, 2002 and 2003 respectively. The processing of cotton to knitted fabrics was carried out in The Cotton Quality Research Unit, ARS Laboratory, USDA, Clemson, SC. Although number of test methods are available at present for assessing the moisture vapor transport of the fabrics but in this work we used the British Standard evaporative dish method BS: 7209:1990, which tests the fabric at 20°C and 65% humidity.

It is evident from the study that the variety DP555 BG/RR has the highest WVP rates and hence gives better comfort to the wearer compared to other varieties. Incidentally, DP555 BG/RR has been the leading cotton variety planted in the US in recent past few years. The basic understanding of the genetic breed-breathability relationship enables to target germplasms for superior properties in cotton fibers. This will lead to the development of high performance fabrics made from cotton fabrics.

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1. Introduction
The challenges in encapsulation of phase Change Materials (PCM) are to obtain the capsules with high core content and wall integrity. However, it has been observed that increasing the core content leads to poor formation of wall, and therefore, inferior stability of microcapsules under use. In this study an attempt has been made to prepare thermally stable microcapsules with high core content of PCM. Stable wall of microcapsules were prepared from melamine-formaldehyde using appropriate prepolymer composition. Using these capsules, a fabric with a heat storage capacity of 100 J/g was successfully prepared and evaluated for thermoregulation.

2. Experimental
The melamine-formaldehyde pre-polymer was prepared using a modified procedure [1]. The PCM emulsion was prepared at high shear rate using sodium luryl sulfate as an emulsifier and poly(vinyl alcohol) as protective colloid at 40 °C. The encapsulation was carried out by lowering the system pH while increasing the temperature to 70 °C. The capsules were separated washed, dried, and cured at 100 °C for 1.5 h.

A thermo-regulated fabric was prepared by coating the fabric with a mixture of microcapsules, binder based on vinyl acetate and acrylonitrile co-polymer, thickener RAN 5000, the reaction catalyst diammonium hydrogen phosphate and distilled water. The coated fabric was dried and cured at 120 °C for 20 minutes.

3. Results and Discussion
3.1. Surface morphology and size distribution
Figure 1 shows that perfectly round and stable microcapsules under optical and scanning electron microscope. The size distribution of the capsules is shown in Figure 2. All capsules were formed in a narrow size range with an average size of about 3.5 microns. Microcapsules were found to be stable to heat up to 120 °C and solvent wash. Higher stability of the microcapsules is ascribed to the use of proper prepolymer composition and the curing of capsules at 100 °C for one and half hour after drying, which led to proper condensation of wall material.

Fig. 1. Optical and SEM micrograph of the capsules
3.2. *Thermo regulated fabric*

The encapsulation process was optimized to obtain microcapsules having very high core content of up to 70% with heat storage capacity of >160 J/g. These microcapsules were applied using a pigment binder over cotton/PET fabric to study their thermoregulation.

Figure 3 shows the DSC thermogram of the coated fabric. The fabric had a very high heat storage capacity of 105 J/g, which was stored and released in a narrow range of temperature change. Figure 4 shows the thermoregulation effect of fabric under controlled temperature environment.

The modified fabric could effectively moderate the abrupt change in environment temperature for a much longer time compared to the control fabric for both the cooling and heating cycles

4. Conclusions

Thermal storage fabric with a very high heat capacity of 100 J/g could be successfully made using stable microcapsules with heat capacity of 160 J/g prepared using a modified encapsulation process.

**References:** [1] Patent pending
Pesticide exposure through dermal absorption in the work environment and household contamination via soiled garments presents a serious health hazard for agricultural workers and their families. Currently available protective clothing systems act primarily as impermeable barriers, not only to harmful chemicals but also to water vapor and airflow which makes them often uncomfortable in field conditions. Comfort, cost, and availability prompt many workers to choose nonspecialized garments constructed from cotton or polyester blends which provide a low level of chemical protection. This research focuses on a class of active textiles with decontamination properties and their potential application for use in protective garments. A model system for studying chemical protection by mechanisms of absorption, barrier and decontamination for this type of porous material will be presented.

The types of surface treatments include; organic, imide 4% DMDMH (1,3 dimethylol 5,5-dimethly hydantoin), and amine 4% MTMIO (3-methylol 2,2,5,5-tetramethyl imidozalidin 4-one) N-Halamines as well as and 2% DMDMH/2% MTMIO mixture (G. Sun, UC Davis) and inorganic, metal oxides, TiO$_2$ and MgO (Nanoscale Inc., Manhattan, KS). Textiles include 35% cotton, 65% polyester plain weave (Testfabrics Inc., West Pittiston, PA) and surface modified polyurethane microporous membranes (K. Tan, Cornell). Aldicarb, 2-methyl-2-(methylthio) propionaldehyde O-(methylcarbamoyl)-oxime (Chem Service Inc., West Chester, PA) an oxime cabamate is the challenge toxin. After exposure to the challenge solution, and its oxidized products, aldicarb sulfoxide and aldicarb sulfone were extracted from the textiles using acetonitrile and analyzed by high performance liquid chromatography (Aligent 1100 series HPLC with method conditions: 15 °C, C18 column, 50 acetonitrile / 50 water buffer pH 3, 220/4 detector, flow rate 1 ml/min). An internal standard (4-bromo 3,5 dimethylphenyl N-methyl carbamate, BDMC) was used along with external standards. This research addresses the effectiveness of self decontaminating textiles (N-halamine treated cotton) as active surfaces which reduce dermal pesticide exposure while maintaining comfort. We expect that protection will increase with decrease in pore size and surface area/sites available for oxidation. Qian and Sun (2005) demonstrated the ability of imide and amine N-halamine treated textiles to oxidize pesticides and perform antimicrobial functions while submerged in solution. This study incorporates standard surface contamination and a diffusion cell with a model skin membrane to model the target environment.

\[
\text{C}_7\text{H}_{14}\text{N}_2\text{O}_2\text{S (aldicarb)} \rightarrow \text{C}_7\text{H}_{14}\text{N}_2\text{O}_3\text{S (aldicarb sulfoxide)} \rightarrow \text{C}_7\text{H}_{14}\text{N}_2\text{O}_4\text{S (aldicarb sulfone)}
\]
Preliminary results indicate that both the N-halamines and metal oxides can oxidize, aldicarb on the textile surface. This study will identify the maximum load per unit area and time profile for the oxidation reaction and determine whether this type of surface treatment provides a significant degree of additional protection from pesticide exposure. All of the test conditions 4% DMDMH, 4% MTMIO, blank fabric and evaporation of pesticide in air showed the formation of aldicarb sulfoxide. Using a two tailed T-test with $\alpha/2=.05$ there is a significant difference between the 4% MTMIO, 4% DMDMH and the control fabric for the formation of aldicarb sulfone. Quantities of aldicarb sulfoxide and remaining aldicarb were comparable for treated fabrics and the control. The greater effectiveness of the DMDMH treated fabric to oxidize aldicarb is consistent with Qian and Sun’s findings due to the greater bond strength of the amide halamine in comparison to the imide.

Thermoresponsive shape memory (SMP) fibers were prepared by melt spinning from a polyester polyol based polyurethane shape memory polymer (SMP) and were subjected to different post-spinning operations to modify their structure. The effect of drawing and heat-setting operations on the shape memory behavior, mechanical properties, and structure of the fibers was studied. It was found that the shape recovery of the thermoresponsive shape memory (SMP) fiber improved with the post spinning operations. In contrast to the as-spun fibers, which were found to show low stress built up on straining to temporary shape and incomplete recovery to the permanent shape, the drawn and heat-set fibers showed significantly higher stresses and complete recovery. The as-spun fiber underwent stress hardening, whereas, the drawn fiber showed stress softening with cycling. The fibers drawn at a DR of 3.0 and heat-set at 100 °C gave stress values that were about 10 times higher than the as-spun fibers at the same strain and showed complete recovery on repeated cycling. This improvement was explained on the transformation brought about in the morphology of the permanent shape of the SMP fibers from randomly oriented weakly linked regions of hard- and soft- segments to the better-segregated, oriented and strongly H-bonded regions of hard-segments. The tensile strength of the thermoresponsive shape memory fiber increased by about 300% from as-spun to the fiber drawn at DR-3.0. There was a decrease in tensile strength of about 20-25% after heat-setting of the drawn fiber. The sonic modulus and the birefringence of the SMP fiber also increased exponentially on drawing due to increase in orientation of the soft and the hard segments but decreased with heat-setting due to molecular rearrangements at the heat-setting temperature. Significant changes in shrinkage % and stress relaxation were also observed in the thermoresponsive shape memory (SMP) fiber after the post spinning operations.
INTRODUCTION

In order to replace a human diseased heart valve, surgeons usually implant valve prosthesis by open heart surgery. Despite good experience and results of heart valve replacement procedure, some patients at high risk can not support such invasive surgery.

We describe a concept of percutaneous aortic valve stent specially designed for being assembled with a textile valve.

The stent has to avoid traumatic anchorage, support blood pressure, and ensure the heart valve function without impairing mitral valve and coronary ostia.

The goal of this work is to test the in vitro behaviour and performance of stent prototypes mounted in the aortic root that reproduces a native porcine root.

MATERIALS AND METHODS

Endoprosthesis manufacturing

Stent
The stent is obtained by a manual interlacing of one steel and one polyester filament. A tubular part ensures axial positioning and the 3 radial arches are configured to expand into the 3 sinuses of the aortic root to avoid translation motions.

The steel gives rigidity and elasticity to support pressure while the polyester interlacing allows compressibility by rotation freedom and no translation between cells segment. Two prototypes with slightly different geometry are tested.

The stent and valve assembling
The textile heart valve is circumferentially positioned around the stent and is fixed on the stent by only 3 knots at the commissure.
**Endoprosthesis testing**

The endoprostheses are delivered in a silicon mould of an ideal aortic root to be closed to heart anatomy.

The in vitro testing is done in term of stent behaviour observations, static and dynamic regurgitation (leakage versus pressure on closed valve and leakage during closing).

We compare performances of a reference (textile valve moulded in a rubber tube with no paravalvular leakage) with the 2 prototypes.

### RESULTS

**Static regurgitation**

The first prototype has no basal annulus reinforcement and shows a dramatically high static regurgitation under pressure (due to important paravalvular leakage).

The second prototype has an expedient static regurgitation, little greater than the reference.

**Dynamic regurgitation**

We observe the endoprosthesis has a correct open/close cycle with a regurgitation close to the one of the reference valve, but at a pressure about only 60mmHg between distal and proximal part of the endoprosthesis. However, it’s not yet possible to reach pressure up to 80mmHg (hemodynamical conditions) due to valve leakage.

**Stent behaviour**

Under static high pressure (140mmHg), we notice the endoprosthesis are well anchored in the root, slightly deformed elastically.

Dynamic testing shows a logical small to and fro motion due to sudden drop and rise on pressure of blood.

### CONCLUSION

Our textile Heartvalve Endoprosthesis shows promising results. Under hemodynamical conditions, the stent supports pressure and stay at the implantation site but are not yet able to maintain strongly enough the expand configuration of the valve annulus which implicitly creates paravalvular leakage.

By controlling manufacturing parameters, relevant modifications of the stent design, and appropriate choice of stent material, we should rapidly reduce paravalvular leakage and obtain expedient performances for this percutaneous device.
In recent years the need of health textile led to the introduction of a number of new fibers around the world. Bamboo charcoal fiber, a multifunctional fiber is one of them.

This article introduces the processing technique of bamboo charcoal and bamboo charcoal fiber. Bamboo charcoal, produced by the destructive distillation of bamboo. With the nano technology micronization, the bamboo charcoal becomes to powder and is dispersed evenly into spinning liquid. The bamboo charcoal fiber is created by forcing the mixture through the tiny holes of spinneret to form continuous filaments, then the filaments are cutted into staple fiber according the final application.

This paper makes a study on physical property of bamboo charcoal fiber, such as examining the surface configuration under the SEM, calculating the strength loss with the interfusion of charcoal powder, etc. The experimental data prove the knitted fabric made of such a yarn or fiber shows good smell adsorbability, water absorbability and mildewproof.
Synthesis of Reactive Dye Resist Agents for Cellulose
Based on Maleic Anhydride Copolymers

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1. Introduction

Recently, the customers want a cotton products which are various, sporty colors and natural patterns. The method which makes these products was doubling yarn, cross dyeing, specific printing, space dyeing and mélange yarn, etc. But, these methods are difficult to express various colors and patterns. Also management on color trend is hard and an occasion to be made by hand happens often. Especially, can’t be a small quantity batch production, because it is impossible to make with a small quantity to use mélange yarn. On the other hand, if the cotton products treat partially with dye resist agent, it is possible a continuous process and a small quantity batch production. The Reactive dye resist agent for cellulose must have characteristics which it remove dyeing site of cellulose by reaction with hydroxyl group of cellulose, it make inactive dye by reaction with dye or it prevent access of dye into cellulose. Maleic anhydride is high reactive cyclic compound, and can remove easily dyeing site because reaction between hydroxyl group of cellulose and anhydride group to form an ester. Also high molecular weight of polymer anhydride prevent access of dye because occur reaction between hydroxyl group and anhydride group in surface of cellulose. To study the feasibility of using maleic anhydride copolymer as a reactive dye resist agent for cellulose. It found that the effect of the molecular weight of maleic anhydride copolymers on the dye resist effect of cellulose.

2. Experimental

Scoured and bleached cotton print cloth (30s, 1×1 rib, knit) was used. Maleic anhydride, MAI, (Aldrich) and \(\alpha-\alpha’\)-azobisisobutyronitrile, AIBN, (Aldrich) was recrystallized from chloroform and methanol, respectively. Vinyl acetate, VAc, (Aldrich) was distilled under reduced pressure. Toluene and diethyl ether were used as received. Wetting agent, Triton X-100 (Aldrich) were used as received. The reactive dye used Cibacron Yellow HR and Cibacron Red HB(Ciba Special Chemicals, Switzerland).

Copolymerization of MAI and VAc was carried out by adding MAI and VAc monomers and AIBN initiator to toluene, purging the reaction mixture with nitrogen and stirring at 70 \(\degree\)C for 4 hours. The total monomer concentration in toluene was 10 wt%. The molecular weight of copolymers controlled by different monomer feed ratios. The copolymerization product was dissolved in warm toluene, precipitated in diethyl ether, filtered and dried in vacuum[1].

The molecular weight of poly(MAn-co-VAc) was measured on a Waters 2414 gel permeation chromatograph equipped with Styragel HR2, HR4 and HR5 columns (Waters). The measurement was carried out using THF as an eluent at 30 \(\degree\)C, and the relative molecular weights compared with PS standards were determined.

The cotton fabric was padded with a solution containing 10, 20, 30 or 40% poly(MAn-co-VAc), 4.8% sodium hypophosphite catalysis and 0.2% Triton X-100 to a pick-up of 100%, pre-dried at 85 for 3 min., then cured at 150, 160, 170 or 180 \(\degree\)C for 3 min. The cotton fabric was dyed with 5% o.w.f. dye(Cibacron Yellow HR and Cibacron Red HB as reactive dye), 70 g/L sodium sulfate, 100:1 liquor ratio, at 60 \(\degree\)C for 90 minute and added to 20 g/L sodium carbonate 30 minute after start of dye. The dye resist effect on dyed cotton fabrics was estimated K/S value by spectrophotometer(Macbeth Color-Eye3100, U.S.A).
3. Results and Discussion

When the copolymer is added to water to prepare the padding bath, the copolymer does not dissolve initially but slowly dissolves as the anhydride units hydrolyze to form acid groups (scheme 1). But, the anhydride group is again formed on heat treatment of the hydrolyzed poly(MAn-co-VAc) 180° for 3 minute in the presence of catalyst. Also reformed cyclic anhydride react with hydroxyl group of cellulose to form an ester link (scheme 2) [1]. Scheme 3 was showed that it compare between low molecular weight and high molecular weight of dye resist agent on the dye resist effect. The dye can prevent to penetrate into cotton fabrics because high molecular weight of dye resist agent occur reaction on the surface of cotton fabrics. Therefore, They are more effective than low molecular weight of dye resist agent.

Table 1 was showed molecular weight and polydispersity of poly(MAn-co-VAc)s which were prepared with different monomer feed ratio. Molecular weights in the range 3,300 to 16,300 and polydispersity in the range 1.51 to 1.65 were obtained. Figure 1 to 3 were showed the effect of molecular weight on the K/S value of cotton dyed after treatment with poly(MAn-co-VAc) respectively. The K/S value in the maximum absorption wavelength (λ_max) was decreased when molecular weight of poly(MAn-co-VAc) was increased. Figure 4 was showed that the effective of curing time on the K/S value of cotton dyed after treatment with poly(MAn-co-VAc)(Mw 13,400) as dye resist agent. The K/S value on the λ_max was decrease when the curing time was increased. But the cotton fabrics were occurred yellowing at above 2 minutes 15 seconds of curing time and 170° of curing temperature. Therefore, the curing time must be within 2 minutes 15 seconds when the cotton fabrics treated with poly(MAn-co-VAc) at 170°C. Figure 5 was showed the effect of concentration of copolymer on the K/S value of cotton dyed after treatment with poly(MAn-co-VAc) (Mw 13,400) at 170°C for 2' 15". The K/S value on the λ_max was decrease when the concentration of poly(MAn-co-VAc) aqueous solution was increased in the studied range. The estimated whiteness from K/S value was approximate 95% when the cotton fabrics treated with 40 wt% poly(MAn-co-VAc) aqueous solution at 170°C for 2 minutes 15 seconds.

4. Conclusion

The dye resist effect of treated cotton fabric with poly(MAn-co-VAc) was approximate 95% about K/S value on maximum absorption wavelength (λ_max), and the dye resist effect was increased when molecular weight of poly(MAn-co-VAc) was increased. The optimum conditions were 40 wt% poly(MAn-co-VAc) aqueous solution at 170°C for 2 minutes 15 seconds when the cotton fabrics treated with poly(MAn-co-VAc) in the studied range.

5. References

Scheme 1. Hydrolysis of poly(MAn-co-VAc) when dissolved in water and ring formation on heating.

Scheme 2. Reaction of cyclic anhydride with cellulose in the presence of catalyst.

Scheme 3. Schematic of the effect of molecular weight on dye resist effect (left: high molecular weight, right: low molecular weight).

Table 1. GPC molecular weight data of poly(MAn-co-VAc).

<table>
<thead>
<tr>
<th>Monomer feed mole ratio</th>
<th>GPC molecular weight</th>
<th></th>
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<tr>
<td>MAn / (MAn + VAc)</td>
<td>M_n</td>
<td>M_w</td>
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<td>0.1</td>
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<td>0.9</td>
<td>2,200</td>
<td>3,300</td>
</tr>
</tbody>
</table>
Fig. 1. K/S value of cotton dyed after treatment with poly(MAn-co-VAc) (dye: Cibacron Yellow HR, dyeing conditions: 5% o.w.f dye, 70g/ℓ Na₂SO₄, 60 °C×30min, then with 20g/ℓ Na₂CO₃ added and dyeing continued at 60 °C for 90 min, liquor ratio 20:1).

Fig. 2. K/S value of dyed cotton to poly(MAn-co-VAc) (dye: Cibacron Red HB, dyeing conditions: same as above).

Fig. 3. The effect of $M_w$ on K/S data of cotton dyed after treatment with poly(MAn-co-VAc) (• Cibacron Yellow HR, wavelength: 440 nm, ▲ Cibacron Red HB, wavelength: 520 nm).
Fig. 4. The effect of curing time on the K/S value of cotton dyed with Cibacron Red HB after treatment with 20 wt% poly(MAn-co-VAc) of Mw 13,400 at 170 °C (for dyeing conditions see Figure 1, K/S value untreated cotton dyed : 22.5).

Fig. 5. The effect of concentration of copolymer on the K/S value of cotton dyed with Cibacron Red HB after treatment with poly(MAn-co-VAc) of Mw 13,400 at 170 °C for 2' 15" (for dyeing conditions see Figure 1, K/S value untreated cotton dyed : 22.5).
Effect of Thickness Variation on the Chemo-Physical Properties of Electrospun PLGA Nanoweb

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Electrospinning is a powerful and straightforward technique for the mass fabrication of polymer nanofibers. When the electrical force on the surface of a suspended drop of polymer solution overcomes the surface tension, a charged jet is ejected toward the collector. As shown in Fig. 1, most of electrospun nanofibers are typically obtained in the form of web. The mathematical simulation model of web building has been made.

The velocity of drum-type collector will affect the orientation of electrospun nanofibers. Furthermore, it will also affect the thickness of electrospun nanoweb if the collector moves transversely as well as rotationally. Fig. 2 shows a schematic diagram of the partial traces of electrospun nanoweb.

In the special applications of electrospun nanoweb such as artificial blood vessel and filter, control of thickness variation is required. To understand the correlations between the velocity of drum-type collector and the thickness variation of electrospun nanoweb, we fabricated electrospun PLGA nanofibers whose diameter distribution was uniform. Also, we investigated the effect of the velocity of collector on the morphology and mechanical properties of electrospun PLGA nanoweb. Finally, we calculated the thickness of electrospun nanoweb as well as variational parameters by a simple accumulation model and compared it with experimental results.

Key words: nanofiber, electrospinning, PLGA, collector velocity

< Figure 1. SEM image of electrospun PLGA nanofibers >
<Figure 2. a schematic diagram of the traces of electrospun nanoweb>
Electrostatic characteristics of the washer-dryer combination under various drying conditions

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This paper suggests the optimal conditions for the laundry to prevent from the electrostatic charge by the evaluation of the electrostatic characteristics during the drying process. Cotton, nylon, and polyester fabrics were used as test specimens, employing the standard washing cycle. The electrostatic characteristics of laundry were measured after the automatic drying process.

The results showed that the moisture regain decreased and the electrostatic charge increased with the drying time. For cotton fabrics, it was exhibited that electrostatic charge was under 1000V for drying time, and hardly generated electrostatic charge due to high moisture regain. On the other hand, electrostatic charge produced on nylon fabrics increased rapidly with drying time, it amounted up to approximately 8000V after 120 min. For polyester fabrics, electrostatic charge amounted to 2000-4000V.

It was observed that the specimen fabrics were already dried up before the standard drying cycle suggested for the consumer was finished. Consequently, the excessive drying caused a generation of electrostatic charge due to the removal of the trace of moisture and the excessive friction. Especially, the softener played an important role to prevent from a generation of electrostatic charge, whose insertion was more effective in the drying than in the rinsing process.

It was also shown that the electrostatic charge could be decreased by drying the fabrics of one kind. On the other hand, the electrostatic charge increased remarkably when drying the different kinds of fibers at the same time. Therefore we suggest that the laundry be classified according to the kinds of fiber, and then be washed and dried before excessive drying to reduce electrostatic charge. And further, a proper use of softener is effective to reduce electrostatic charge.
Effect of spinning conditions on the nonwoven properties of electrospun polyurethanes

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Electrospun web for clothes has multiple functions simultaneously such as bacterial resistance, breathability, water & wind proof, thermal insulation. On account of it's distinction from coated polyurethane films for clothes, it is expected that the electrospun polyurethane web can cover a wide area of functional clothing. Therefore spandex (from H. co.) and Pellethane® elastomer (from DCh. co.) were electrospun under the various spinning conditions to fabricate polyurethane webs for clothing in this study. Effects of electrospinning conditions such as concentration of polymer solution, electric potential and distance from spinning tip to collector on the characteristics of electrospun nonwovens were investigated and optional condition was determined. N,N-dimethyleacetamide (DMAC) for spandex and N,N-dimethyleformamide (DMF) for Pellethane® was used to make polymer solution. In low concentration, the fiber was not formed from the polymer solution. And beads and droplets began to be created after a specific concentration. On the other side, the electrospinning at higher concentration was impossible as too much high viscosity. The thicker the electrospun web was, the less electric potential was created on the collector as covering of fiber web. Making nonwoven of enough thickness needed adjustment of voltage by rate of spinning progress.
The redefinitions of the energy policy of the US have multiple fronts. One objective of the DOE is to enforce the development of new lightweight materials with identical or better properties than existing materials. The goal is to reduce the weight of vehicles by 33% (in comparison to 2002) by 2010. One of the promising alternatives under consideration is injection molded parts made of polymers reinforced with large aspect ratio particles (i.e. long fibers or nanoparticles). However, these types of parts have not been successfully manufactured because of the unknown molecular behavior of the materials during processing. In this research we are trying to extend the Doi’s theory for rod-like systems to simulate the rheological behavior of these composites.

A numerical code is being written to simulate the flow of fiber suspensions in injection molding flow geometries. The preliminary results of the simulation for shear flow shows that the model can reproduce the experimental data under stress growth conditions. The code has been validated in shear and extensional flow which are used to determine material parameters.
Convergent synthesis of poly(methyl methacrylate) (PMMA) based electroluminescent colloids

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The development of small-molecule based, organic light emitting diodes (OLEDs) has received a great deal of attention in recent years as versatile alternatives to inorganic based LED systems. Here we report the convergent synthesis of all-inclusive poly(methyl methacrylate) (PMMA) based electroluminescent colloids containing a hole-transport material, an electron-transport material, and an electroluminescent dye through emulsion polymerization techniques. This multi-component colloid was synthesized from methyl methacrylate (MMA, base polymer), MMA-carbazole (MMAK, hole-transport molecule), tert-butyloxiadiazole methacrylate (TbuMMAO, electron-transporting molecule), and an electroluminescent dye. The ability to synthesize multi-component, monodisperse EL colloids of varying sizes (50-500 nm) will have great impact in the fields of LED based display materials.

Some of the advantages of these systems over their inorganic competitors are a consequence of the thin, light, and flexible properties of the organic layers that make up OLEDs. These OLED/PLED characteristics result in number of performance enhancements relative to the crystalline layers in inorganic light emitting diodes (LED) or liquid crystalline displays (LCD), for example: (1) the conductive and emissive layers of an OLED can be multi-layered resulting in a brighter device; (2) OLEDs do not require backlighting and consume much less power than LCDs; (3) OLEDs can be produced through simple spin-coating or ink-jet printing techniques and can be made into large, thin sheets; and (5) OLEDs have very large fields of view relative to LCD systems since they produce their own light. Nonetheless, there are currently a number challenges to the widespread implementation of OLED systems, such as their limited lifetime performance, sensitivity to the environment, and the restricted range of EL molecules available for OLED/PLED construction. Solutions to these challenges are being pursued actively both in the academic and commercial areas to gain a deeper fundamental understanding of the role that chemical architecture has on the solid state physics of the devices and the resulting performance of the devices as a light source.

Propargyl poly(acrylate) photonic bandgap composites

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In the field of optical materials, crystalline materials composed of monodisperse colloidal particle assemblies (both inorganic and organic) to control various light propagations has been well documented both in the solid state and in less stable solution based crystalline colloidal arrays (CCAs). In these systems, a series of forces ranging from electrostatics to physical constraints promote these uniform colloidal particles into highly crystalline ordered arrays. These crystalline arrays optical properties can be effectively manipulated from simple changes in the size of the particles to the distance between the particles (interparticle differences), all changes which are predictable and explicable using Bragg theory. There are several systems in the literature that eloquently demonstrate the versatility of CCAs in the creation of both hydrogel and solid films wherein the optics of the inherent material can be modulated through a series of external stimuli. To date; however, there is limited knowledge of different chemical routes to the modification of CCA composite materials after they have assembled into a film. Often times these polymeric CCAs are composed of either poly(styrene) or poly(methyl methacrylate) particles wherein the inherent optical properties of the materials becomes difficult to modify post-film preparation. Although it has been demonstrated possible to introduce various components into the interstitial regions of the CCA composites after film formation, procedures that chemically modify the core polymeric colloids within the CCA film are scarce. Herein, we introduce a chemical route to the functionalization of propargyl poly(acrylate) colloids embedded within an aqueous CCA hydrogel matrix using a “Click” chemistry approach. “Click” transformations, also known as Huisgen 1,3 dipolar cycloadditions, are copper (I) mediated cycloadditions between azides and either alkynes or nitriles which can be conducted under a variety of conditions and have been utilized extensively by Sharpless et. al. for difficult biological transformations in aqueous environments. In our system, monodisperse propargyl poly(acrylate) (PA) colloids were prepared using a standard emulsion polymerization technique.

Scheme 1. “Clicking” azidoanthracene groups onto monodisperse propargyl poly(acrylate) colloidal particles
A study was conducted to produce polypropylene fibers and nonwovens with the commercial samples of nanoclay additives. Experiments were carried out to produce spunbond fabrics using a 35MFR PP, supplied by Exxon-Mobil Company, with addition of nanoclay using a Reicofil spunbonding machine. Concentrates of nanocaly provided by Techmer PM, Clinton, TN, were intimately blended with PP and extruded. Spun fiber samples from the collector screen and spun bond samples after calendering were collected for analysis. All the fabric and fiber samples produced in the experiments were analyzed for physical properties and structure under standard laboratory conditions.

Most of the samples had fiber diameters in the range 18 to 25micron. The DSC data showed some change in crystallinity and melting behavior although no definite trend in crystallinity was observed. The samples with these additives showed some shift in peak melting temperature and change in the shape of the melting peak, indicating differences in crystallinity or crystal size distribution. There was a substantial increase in tensile strength and modulus when nanoclay additives were added. This increase in strength is not accompanied by a decrease in breaking elongation, which is very unique. This is a promising situation as we expect good tensile properties in the spunbond fabric. The nanoclay additives increase the tensile strength of the spunbond fabrics both in MD and CD. Further it is noticed that there is no substantial increase in strength by raising the level of additive from 1% to 2%. The increase in tensile strength and modulus of the webs is 10 to 20%, both in machine and cross directions. Similarly, bending length or stiffness of the fabric increased by 10 to 25%. Also, a substantial increase in burst strength was observed on addition of small amount of nanoclay.
Thermal Bonding of Polypropylene Films and Fibers

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and
R. A. Campbell, Sunoco Polymers, Pittsburgh, PA

Thermal bonding behavior of different grades of polypropylene was studied in this research. Initial bonding studies were done on different grades of polypropylene films of different morphology. Effect of film structure, properties and bonding conditions on the bonding efficiency was studied. A range of bond temperatures covering poor, optimum and over bonding was studied to understand the changes taking place. Bonded film strength was analyzed using tensile testing. Changes taking place to polymer in bond point and original surface were analyzed using SEM. Effect of different material and process variables on bonding are studied. Bonding studies on films were extended to fibers. Polypropylene fibers manufactured with different cross-section, denier, polymer melt-flow rate and different processing conditions were bonded with different heat sealers.

Higher bond strength was observed in the vicinity of melting temperature and the strength reduced with the increase in bonding temperature. Lower pressure and shorter time were found to be appropriate for bonding. Films with lower orientation formed better bond. Optimum bond strength observed and the optimum bonding temperature were different for different polymers. Fiber bond strength results were similar to results observed in case of films with respect to bonding temperatures studied.
Structure and Properties of Latex Coated Nonwovens

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Coated Nonwovens are used in a variety of applications, and are a significant part of the coated fabrics industry. U.S. Coated fabrics industry is poised for a growth rate of 3% annually through 2009. There is limited research in the area of coated nonwovens to understand the process and structure-property correlations, with the aim of enhancing the properties of these fabrics.

Two different, one needlepunched and another spunbond, nonwoven fabrics have been coated with a latex material using the Mathis laboratory padding/coating-drying machine. Effect of processing conditions and formulations on the structure and properties of coated nonwovens are being evaluated. Results from this ongoing research will be presented.
1. Introduction

Back-propagation neural networks are flexible, nonlinear modeling tools that are an extension was tested. BNN models were built using Levenberg-Marquardt algorithm, with 30 hidden nodes, and log sigmoid non-linear transfer functions. Thickness at zero pressure, To (mm) and Thickness at maximum pressure Tm (mm) were predicted. Observations from 268 fabrics were us of traditional statistical techniques. The hypothesis that Back-propagation neural network models can be built in a similar fashion as a statistical model ed to train, test, and validate the BNN. Inputs were measured using ASTM standards. Model selection was performed using the, Bayesian information criteria (BIC), and the method of stopped training. The models with lowest mean square (mse) error were those with 6 inputs and 30 hidden nodes. Average prediction error in the validation set by stopped training for To (mm) was 7.45%, and Tm 7.59%, finally BIC (2.15) was consistent over number of trials on testing set.

2. Experimental

268 cotton fabrics were used for this study. Data for modeling was divided into 3 sets namely Training set(168), testing set (47) and Validation set(53). (Shown in Fig.1)

3. Results and discussion

A BNN - Levenberg marquardt algorithm
With 6:30:2 – Network Architecture was used for predictive modeling of compression properties of woven fabrics.

<table>
<thead>
<tr>
<th>Model Type</th>
<th>To(mm)</th>
<th>Tm(mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BNN model(6:30:2)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Training(168,62%)</td>
<td>6.292 %</td>
<td>5.971 %</td>
</tr>
<tr>
<td>Testing (47, 18%)</td>
<td>8.562 %</td>
<td>7.001 %</td>
</tr>
<tr>
<td>Validation(53,20%)</td>
<td>7.4165 %</td>
<td>7.593 %</td>
</tr>
<tr>
<td>Statistical Modeling</td>
<td>14.498%</td>
<td>14.68 %</td>
</tr>
</tbody>
</table>

Table 1 gives the Absolute Prediction error percentage which is calculated for both BNN models and Statistical model.
4. Validating network results with experimental results

The acceptability of Network outputs is judged by comparison to standard experimental values. The results of correlation from BNN models and measured values using KES- FB3 system was utilized for validation. The R² value for To(mm) was 0.7664 (shown in Fig.2) and for Tm(mm) was 0.7335 (shown in Fig.3)

![Figure 2: Correlation graph for To(mm)](image)

![Figure 3: Correlation graph for Tm(mm)](image)

5. Generalization results

Figure 4 shows the response of testing set network to Bayesian regularisation. This eliminates the guesswork required in determining the optimum network size. When using Bayesian algorithm, it is important to let the algorithm run until the effective number of parameters has converged. The training stopped with the message "Maximum MU reached." This is typical, and is a good indication that the algorithm has truly converged. One can also tell that the algorithm has converged if the sum squared error (SSE) and sum squared weights (SSW) are relatively constant over several iterations.

![Figure 4: Bayesian Information](image)
Table 2: Bayesian Information criteria for selecting number of hidden neurons

<table>
<thead>
<tr>
<th>Trial Nos</th>
<th>SSE*</th>
<th>SSW*</th>
<th>NOEP*</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.431084</td>
<td>0.0304381</td>
<td>2.15/272</td>
</tr>
<tr>
<td>2</td>
<td>0.431084</td>
<td>0.0304381</td>
<td>2.15/272</td>
</tr>
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<td>3</td>
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</tr>
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<td>2.15/272</td>
</tr>
<tr>
<td>5</td>
<td>0.431084</td>
<td>0.0304381</td>
<td>2.15/272</td>
</tr>
</tbody>
</table>

*SSE= Sum squared error  
*SSW=Sum squared weight  
*NOEP= Number of effective parameters

6. Conclusion

The neural network models are compared with multiple linear regression model. As it was anticipated, the neural network models provided better prediction capabilities because they generally offer the ability to model more complex non-linearity’s and interactions better than linear models. In this study, Bayesian regularisation with Levenberg-marquardt training algorithm is used to gauge the generalisation capacity of the network. This method is also utilized to overcome the problem of determining optimum number of neurons in hidden layer. The results obtained after simulations proved the efficiency of this methodology.

References


Acknowledgment

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Extrusion of Immiscible Polymer Blends for Fabrication of Highly Porous Fibers with Continuous and Size Adjustable Porous Structures

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Porous polymer fibers can be fabricated using different methods including blend mono-filament spinning, hollow fiber spinning, multi-component fiber spinning (e.g. with islands-and-sea morphology), and solution spinning. However, with the current technology, it is difficult to achieve a continuous porous structure across the entire fiber cross-section and control the pore size. This greatly limits potential applications of such fibers in many emerging biochemical and biomedical applications.

We report here a filament extrusion process of immiscible polymer blends for fabrication of highly porous fibers with continuous and size adjustable porous structures. The two polymers used in the blend were polycaprolactone (PCL), a biocompatible and biodegradable polymer, and polyethylene oxide (PEO), a biocompatible and water soluble polymer. We have previously used the same blend in compression molding of 3-D porous polymer scaffolds for tissue engineering applications and found that the resulting scaffold can greatly promote attachment and growth of seeded cells. The two polymers have similar melting temperatures and desired rheological properties for forming a co-continuous polymer blend. To produce highly porous PCL fibers with continuous porous structures, the two polymers at a mixing ratio of 30%-PCL and 70%-PEO were first blended in a batch mixer, then co-extruded, and finally selectively dissolved in water and dried. The batch-mixed PCL-PEO blend was found to have a co-continuous, ultra-fine blend morphology with characteristic size of phases below 1 μm. It is noted that such porous structures are too fine for tissue engineering applications since the pore size is considerably smaller than that of a typical cell. Filament extrusion was performed on a Dynisco capillary rheometer with a capillary orifice of 0.25 mm in diameter.

The PCL fiber after dissolution of the PEO phase was fractured to observe its cross-section structure. After submerging the fiber in liquid nitrogen for a short period, the fiber can be broken in a brittle fracture mode to preserve the porous structure. Figure 1 shows porous fiber morphologies obtained under different process conditions. It was observed that the residence time of the blend at its melting state inside the rheometer played a major role in influencing the pore size of the porous fiber. As the residence time increased, the pore size became larger, as shown in Figure 1. With residence times above 30 minutes, characteristic pore sizes around 10 μm or above were obtained. This enlargement of pores is believed to be caused by surface tension driven flow due to the interfacial tension of the two phases. The time scale for pore size evolution is dependent on the surface tensions and the viscosities of the two polymers. As the processing temperature increases, the size changing process will speed up because of the reduced viscosity. Such reasoning agrees well with the experimental finding. An alternative approach for increasing the pore size is a post annealing process after filament extrusion. The post annealing time was found to have a similar effect on the pore size as the in situ residence time.

The highly porous PCL fiber with a continuous and size-adjustable porous structure fabricated using the present method is anticipated to find various applications in the emerging biomedical industry, particularly for tissue engineering. Examples of tissue engineering applications could be injectable scaffolds for repair of retina defects and axon scaffolds for neuron regeneration.
References


Figure 1: Continuous porous structures of highly porous PCL filaments prepared by co-extruding a PCL-PEO blend and subsequently dissolving the PEO phase. Two levels of residence time of the polymer blend inside the extruder are compared: a) no resident time, and b) 30 minutes of resident time.
The range and intensity of characteristic emission of Light Emitting Polymers (LEP) in solution vary widely from their solid bulk phase. The increase in emission intensity in solution is ascribed to lack of crystalline imperfections where an exciton can be quenched. In a solution the LEP molecules disentangle and are spread apart, thus reducing inter and intra chain interactions. This phenomena leads to narrowing of band width and thus increase in energy gap of the LEP semiconductor. The result is a significant blue shift in emission in a LEP solution than from its bulk solid phase.

Polymerization induced phase separation (PIPS) has been used to disperse phases of a specific LEP into a polymer matrix with appropriate optical properties. The morphology of phase separation of the LEP can be controlled by varying the kinetics of the polymerization reaction of the transparent matrix polymer. Polymer beads obtained in this process have varying emission properties ranging from 400nm peak (Indigo) to 500nm peak (Green) depending on the morphology of phase separation. The spherical beads also provide a resonating cavity effect for light amplification.

Using similar principle fibers are made with dispersed LEP in nano phases. Design and fabrication of a device is proposed, that generates photoluminescence where the emission frequency can be changed by elongation of the fiber strands. As the fibers are loaded under tension, internal stresses would deform the LEP nano crystals resulting in alteration of the emission frequency. Optical and mechanical properties of the fiber can be chosen in a way to cause a resonant cavity effect thereby amplifying the emitted light.
The Use of Bacterial Cellulose as a Novel Non-Woven Fiber Matrix for Ceramic and Metal Deposition

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Certain bacterial species possess the unique ability to extrude pure cellulose nanofibrils into a non-woven matrix suitable for a variety of applications. Due to the great surface area of the fibers and the abundance of hydroxyl groups in the cellulose, the polymer network is highly hydrogen bonded and assumes a hydrogel structure in its native state. The unique structure and chemistry of bacterial cellulose provide an ideal template for the deposition of metals and ceramics. The chemical structure of the cellulose induces high localized concentrations of cations to produce specific nucleation initiation sites, and an environment suitable for targeted catalysis. The physical structure provides spatial constraints while the high surface area of the nanofibrils is accessible for nucleation and catalysis. Several types of catalytic groups can be incorporated into the polymer backbone to enable targeted deposition of metals such as palladium, platinum, and silver. Since 99.8% of its mass is water, bacterial cellulose easily permits serial infusions of aqueous solute precursors and reactants to form ceramics such as calcium-deficient hydroxyapatite and calcium carbonate. Bacterial culture conditions can be altered to modulate the size, shape, porosity, and thickness of the cellulose matrix used for biopatterned deposition. The size and composition of metal and ceramic particles formed within the cellulose matrix were determined by Scanning and Transmission Electron Microscopy (SEM/TEM), Fourier Transform Infrared Spectroscopy (FTIR), Laser-Induced Breakdown Spectroscopy (LIBS), and X-Ray Diffraction.
A new technology that would eliminate the disadvantages of the current PVA ultrafiltration (UF) recovery process: flash evaporation (FE). The FE process for recovery and concentration has been used in a variety of other industries but has never been demonstrated for size recovery in the textile industry. In this technology, the excess water in the dilute PVA desize stream is flash evaporated off in a controlled pressure manner and recycled back into the plant, giving a true closed-loop character to the overall desizing-size recovery/concentration-reuse sequence of processes and aiding the overall economics. The concentrated PVA stream exiting the FE process will also contain the low molecular weight “tail” of the PVA distribution that is currently lost in the UF process, and thus will have more favorable material balance economics than the existing technology. This study is concerned with the FE process, economics of FE and the properties of recovered PVA.
Acid Leuco Dyeing of Unmodified Polypropylene Fiber

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Since the commercialization of isotactic polypropylene, this fiber is being used in many industrial applications as well as in carpets and apparel due to its high degree of crystallinity, good handle, high modulus, strength and a high enough melting point for normal use. Due to its hydrophobic nature, most of the production of polypropylene (PP) fiber is colored by means of mass pigmentation. This route of coloration gives excellent fastness properties during end use; however, it restricts the producer in fulfilling the changing fashion demands of the market. An alternative way of coloring PP fiber exists in which the fiber can be made dyeable by means of post modification, creating active sites for dye association or adding hydrophilic comonomers, but this route has adverse effects on the mechanical properties and costs of the fiber. The development of a truly aqueous process for dyeing PP in its generic, unmodified form is thus of significant commercial importance vis-à-vis the rising demand for this inexpensive fiber.

The laboratory based process for dyeing unmodified PP, spun yarn fabrics supplied by TestFabrics, Inc. has been developed and some good vat dye candidates have been identified. The screening of good vat dye candidates for polypropylene fiber is based on the solubility parameter approach. The correlation between dye exhaustion and the solubility parameter for PLA fiber and disperse dyes has been reported by Karst et al. and has been shown that the closer the solubility parameter of the dye to the fiber results into better exhaustion of the dye inside the fiber. Solubility parameter is the square root of cohesive energy density. In the case of disperse dyeing on polyester, the dye molecule diffuses into the opened structure of the fiber at high temperature and is physically entrapped into the fiber after cooling. The similar phenomena can be expected in the case of acid leuco dye diffusion in the solid state structure of polypropylene fiber. This approach of dye diffusion involves the solubility of the dye molecule inside the hydrophobic fiber at elevated temperatures, and therefore the dyes which have solubility parameters closer to that of the PP fiber will likely to give more color yield upon high temperature dyeing. The analogous disperse dye form being utilized for polypropylene coloration is the acid leuco (non-ionic) form of vat dyes. The acid leuco forms of vat dyes with low solubility parameters have shown good affinity and substantivity for unmodified polypropylene fiber, presumably following a solid solution, hole filling dyeing mechanism.
Effect of Molecular Weight and Polymer-Solvent Concentration on Electrostatic Processing of Chitosan

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Electrospinning is a process by which sub-micron sized polymer fibers can be produced using an electrostatically driven jet of polymer solution. The fibers are collected as a non-woven mat and offer a high surface area to volume ratio. Chitosan is a non toxic and biodegradable biopolymer derived from naturally occurring chitin. Chitosan has excellent metal binding and anti-microbial properties which could be beneficial in air and water filtration applications. Our current research focuses on electrospinning of chitosan. As chitosan naturally has a high molecular weight, it exhibits high solution viscosity even at low concentration which complicates the electrospinning process. Also, the choice of solvents for chitosan is limited as it only dissolves in aqueous acid solutions and fluorinated hydrocarbons. From our preliminary results we have found that electrospinning of chitosan is strongly dependant on its solubility in aqueous acids. The solubility of chitosan in aqueous acids is limited by its high molecular weight and also the strength of the acid solution. The goal of the current work is to determine the conditions under which chitosan solutions can be successfully electrospun and to determine processing-structure relationships. Important variables are molecular weight, solution pH, solution concentration, and solution conductivity. Acid hydrolysis is used to reduce the molecular weight of chitosan. Results of our ongoing study will be presented wherein we are working to find suitable polymer-solvent concentration, polymer molecular weight, and optimum electrospinning parameters for fabrication of defect-free chitosan nanofibers.
Mechanical Properties of Fibers Using Nano-Tensile Testing*

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Due to the recent evolution of nano-structured polymeric, metallic, and biological multifunctional materials at rapid pace, there is an immediate need in determining mechanical properties using small volume of materials. Determination of modulus, stress state corresponding to yield and/or failure and associated hardening or softening behavior is most reliably obtained using a direct tension test due to the simplicity in describing the state of stress and strain over a gage length. In the present research, a nano-tensile testing system is being developed for determining the tensile properties of fibers and related materials. The determination of fiber properties via tensile methods clearly requires specific consideration related to type of grips and specimen mounting procedures. Important questions regarding specimen gripping without compromising sample and alignment as applied to fibers having diameter in the range of 5 to 100 microns has been explored to date. The testing system that is being used has exceptional resolution related to the measurement of axial load (nano-Newton) and displacement (nanometers) by using a nano-indentor head in an inverted configuration. The global displacement of these fibers is being measured at micron resolution, with axial force resolution at 50 nano-Newton, exceeding the performance capabilities of any standard load cell.

This study presents initial results of tensile testing with polypropylene, glass, copper, and carbon fibers. A review of currently existing international (ISO) and national (ASTM) standards for carbon fiber testing revealed a need for improved templates for mounting fiber specimens. Details of a novel design of new fixtures and template for easy mounting of fiber type specimens along with experimental setup related to specimen mounting and alignment aspects will be presented. The present testing system is currently being used to test new materials (electro-spun polymeric fibers with single wall carbon nanotubes) being synthesized at the Center for Nano-phase Materials Science (CNMS) of ORNL. In addition to obtaining stress-strain behavior, using the phase difference between applied sinusoidal load and corresponding displacement, storage and loss modulus as a function of global strain is being measured. This could provide new insight into damage evolution and phase transition during deformation behavior of materials subjected to stress at constant temperature.

*This abstract is from a technical paper that will be presented at Materials Science and Technology (MS&T 2006) Conference during October 15-19, 2006 in Cincinatti, OH. Please refer to the paper for additional details.
Polymeric fibers have been made in various cross-sections in order to enhance their properties and performance. The desired properties may range from stiffness, luster, and dirt hiding capabilities in the case of carpets and textiles, to attraction and retention of dust in filtering applications, to increase in surface area and permeability for hollow fiber membranes.

Modern spinneret capillary manufacturing technology such as EDM (Electrical Discharge Machining) allows quick and inexpensive fabrication of elaborate capillaries shapes, thus giving us an ability to test hundreds capillary designs. As a result testing of over 400 capillary designs and studying polymer flow effects in capillaries we have developed technology to produce practically any fiber shape. In addition a number of various product prototypes were fabricated with the spun shaped fibers that verified fiber shape efficacies and improved product performance.

Combining fiber shape technology with multi-component fiber spinning offers even greater opportunities for new products with unique properties.

Lately new discoveries made in the field of nano-technology and bio-genesis pose a new challenge of handling and orienting substances on the scale of $10^{-9}$ to $10^{-6}$ m. The shaped fibers with their micron sized geometric features and variety of materials present a flexible and inexpensive solution.

This presentation gives dozens of examples of fiber shapes and discusses their potential applications.