The Fiber Society's Fall 2018 Technical Meeting and Conference

Advanced, Smart, and Sustainable Fibers, Materials, and Textiles

October 29–31, 2018

Conference Chair

Dr. Gang Sun
University of California-Davis

Venue

UC-Davis Conference and Welcome Center
Davis, California

Program

Sunday, October 28
1:00 PM–5:00 PM  The Fiber Society Governing Council Meeting, Everson Hall, Room 262
5:00 PM–7:00 PM  Hyatt Place Hotel Lobby (173 Old Davis Road Extension, Next to Conference Venue)
Monday, October 29

7:00  Registration, UCD Conference Center
7:30  Beverages and Light Breakfast

8:00  Welcoming Remarks and Announcements (Ballroom B/C)  
Gang Sun, Conference Chair  
Dr. Fadi Fathallah, Associate Vice Provost for Global Affairs, UC-Davis  
Jintu Fan, President, The Fiber Society

8:15  Plenary Lecture: Yi Cui, Stanford University  
Fibers Across Multiple-length Scales for Energy, Environment, and Electronics

8:50  Break

Morning Session

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<th>Ballroom B</th>
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| **Session: Fiber Manufacturing and Characterization**  
Chair: Takeshi Kikutani | **Session: Nanofibers and Nanofibrous Materials**  
Chair: Xiangwu Zhang |
| 9:00 Dynamic Cross-linking Spinning: A New Strategy for Spinning Hydrogel Fiber  
Meifang Zhu, Donghua University | Unidirectional Moisture Transport Nanofibrous Membranes  
Xianfeng Wang, Donghua University |
| 9:25 Electrospinning Pre-determined Nanofibrous Geometrics by Rapid Jet Deflection  
Jintu Fan, Cornell University/Hong Kong Polytechnic University | Electrospinning Polypyrrole for Novel Carbon Dioxide Sensors: A Study of Electrospinning Parameters for Highly Sensitive and Conductive Nanofibers  
Ashwariya Lahariya, Cornell University |
| 9:50 Artificial Spider Silk: Toward Sustainable and Resilient Future  
Jinlian Hu, Hong Kong Polytechnic University | Constructing Fiber-based Flexible Triboelectric Nanogenerator with Electrospinning Technique  
Zhaoling Li, Donghua University |

10:15  Break

| 10:30 Cotton Fibers Creep Behavior: Viscoelastic Modeling and Bundles Friction  
Dominique Adolphe, ENSISA (for Wafa Mahjoub) | Sub-nano Thick Silk Nanoribbons Exfoliated from Silkworm Silk  
Yaopeng Zhang, Donghua University |
| 10:55 Structure and Properties of PET Fibers Prepared with Infusion of Ethanol in Continuous Cold Drawing Process  
Dongwoo Go, Tokyo Institute of Technology | Enhanced Piezoelectric Response of Electropsun PVDF with ZnO Nanorods  
Minji Kim, Cornell University |
| 11:20 Image Processing Technique for Cotton Color Grading  
Nayab Khan, University of Agriculture | Flexible, Ion-conducting Ceramic Textiles  
Kun Fu, University of Delaware |
| 11:45 Cooling Performance of Integrated Thermoelectric Thermoregulatory Undergarment  
Jintu Fan, Cornell University/Hong Kong Polytechnic University (for Lun Lou) | 1D Nanomaterial Innovations from Biomass  
You-Lo Hsieh, University of California-Davis |

12:10  Lunch (Room A)
### Afternoon Session

#### Student Paper Competition

**Chair:** Meifang Zhu

- Xingchen Liu, University of California-Davis, *Amphiphilic and Semi-crystalline Soy Protein Microfibrils from Aqueous Colloids*
- Hongyan Wu, Donghua University, *Ultralight, Highly Resilient, and Flexible Electrospun Nanofibrous Aerogels for Effective Warmth Retention*

**Break**

#### Session: Fiber Manufacturing and Characterization

**Chair:** Jintu Fan

  - Yuki Miyao, Tokyo Institute of Technology
- *Preparation of Polyether-ester Elastomeric Fibers Through High-speed Melt Spinning and In-line Drawing Processes*
  - Takeshi Kikutani, Tokyo Institute of Technology

#### Session: Fiber-based Sensors and Intelligent Products

**Chair:** Dominique Adolphe

- *Conductive Bicomponent Fibers Containing Polyaniline Produced by Electrospinning*
  - Hang Liu, Washington State University
- *Smart Moisture Actuators Based on Fiber Materials*
  - Mufang Li, Wuhan University (for Dong Wang)
- *Cellulose Nanofibrils Exfoliated Graphene for Moisture-responsive Actuator*
  - Xuezhu Xu, University of California-Davis
- *Preparation of Conductive Fiber Assemblies and Their Applications on Flexible Sensors*
  - Weibing Zhong, Donghua University

#### Session: Nanofibers and Nanofibrous Materials

**Chair:** Francesco Fornasiero

- *Electrospun Nanofibrous Aerogels: Progress in Materials, Properties, and Applications*
  - Bin Ding, Donghua University
- *Novel Mineralized Nanofibers for Water Remediation: Discussion of Their Synthesis and Performance*
  - Ericka Ford, North Carolina State University

#### Session: Nanofibers and Nanofibrous Materials cont’d

#### Poster Session and Reception
Tuesday, October 30

7:45    Beverages and Light Breakfast

8:30    **Plenary Lecture (Ballroom B/C):** Jianyong Yu, Donghua University
        *Progress and Perspectives of Electrospun Nanofibrous Materials*

9:05    **Break**

**Morning Session**

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<td><strong>Session: Nanofibers and Nanofibrous Materials</strong>&lt;br&gt;Chair: Bin Ding</td>
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<td>9:25 Cotton Recycling for Making New Functional Fiber&lt;br&gt;Jonathan Chen, University of Texas-Austin</td>
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<td>9:50 Controlled Surface Modifications of Polyester Fibers&lt;br&gt;Radical Graft Polymerization&lt;br&gt;Gang Sun, University of California-Davis</td>
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<td>10:15 Graft Polymerization of DADMAC into Cotton for Ultra-deep Black Dyeing&lt;br&gt;Shahriar Salim, North Carolina State University</td>
<td>Low-thermal Conductivity Carbon Fibers from Electrospinning&lt;br&gt;Spero Gbewonyo, North Carolina Agricultural and Technical State University</td>
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**Break**

11:00 Open

11:25 Optimization of Process Parameters to Control Yarn Packing Density of Cotton Spun Yarn by Using Multiple Regression Models<br>Mohammad Ali Zeeshan, Balochistan University of Information Technology

11:50 Generation of Hydroxyl Radicals from H₂O₂ Under UVB Irradiation for Effective Bleaching of Cotton Fabrics<br>Peixin Tang, University of California-Davis

12:15 **Lunch (Room A)**

**Afternoon Session**

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<td>1:20 Heart Valve: Functionalization and Optimization of the Material&lt;br&gt;Frederic Heim, ENSISA (for Amna Amri)</td>
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<td>2:10</td>
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<td>Zirconium Hydroxide-based Sorptive and Reactive Textiles</td>
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<td>Arginine-NIPAAm Hybrid Hydrogel with Biocompatible and Antibacterial Properties</td>
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<td>Fiber Society Annual Business Meeting: Open to Fiber Society Members Only</td>
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<td>• Speaker: Alex Patist, Bolt Threads, Silk and Leather: Practical Biological</td>
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**Wednesday, October 31**

7:45 Beverages and Light Breakfast

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<td>Francesco Fornasiero, Lawrence</td>
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<td>Long-term Stability of Copolymer</td>
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<td>Aramid Fibers</td>
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<td>Amy Engelbrecht-Wiggans, National</td>
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Physiological and Physical Strain Associated with Wearing Chemical Protective Clothing in a Hot Environment
Guowen Song, Iowa State University

Nanocellulose Aerogel Coaxial Fibers for Thermal Insulation
Jian Zhou, University of California-Davis

Characterizing Thermal Protective and Thermophysiological Comfort Performance of Fabrics Used in Firefighters’ Clothing Using 2D Intermediate Tests
René Rossi, Empa

Carbon Nanotube/Polyvinyl Alcohol-coated Yarn: An Advanced Fiber for Multifunctional Textiles
Fujun Xu, Donghua University
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<td>Wettability and Adhesion of Thermoplastics in Glass Fiber-reinforced Model Composites</td>
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<td>Elizabeth Welsh</td>
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We Gratefully Acknowledge Our Contributors

Division of Textiles and Clothing, University of California-Davis: *Beverage Bottles*

UC Davis Confucious Institute, University of California-Davis: *Book Marks*

Wuhan Fiber Valley Science and Technology Development Company, Wuhan, China: *Bags*
Plenary Speakers
Fibers Across Multiple-length Scales for Energy, Environment, and Electronics

Yi Cui

Department of Materials Science and Engineering, Stanford University; Stanford Institute for Materials and Energy Sciences, SLAC National Accelerator Laboratory

yicui@stanford.edu

Fibers are a rich family of materials, with diameters from nanoscale, microscale to macroscale; compositions from polymer, metal, semiconductors to ceramics; morphology from solid, hollow, core-shell, branching to other complex shapes. Fibers enable transport carriers such as photons, electrons, and ions to long distance yet maintaining mechanical flexibility. Here I will present our research in the past decade on how to explore unique properties for fibers for application in energy, environment, and electronics.

First, fibers morphology can enable high-energy density batteries. The most important example is nanowire and hollow fibers of Si anodes, which overcome volume expansion problems during lithium insertion into Si and offer 10 times higher specific capacity than the existing carbon anodes. The materials design is a game changer for high-energy batteries.

Second, nanoporous polyethylene fibers can be woven into a textile. The porosity scatters the visible light yet allowing human body infrared radiation to pass through. This generates a new type of cooling textile. With the understanding to control the human body infrared radiation, it is possible to generate a textile with cooling, heating, bifunctional (heating and cooling by flipping the side), and cooling under the sign. The concepts here could have high impact to the textile industry.

Third, filters made of polymer nanofibers will be discussed to have superior effect to capture pollution particles such as PM2.5 from the air, while maintaining low air pressure drop. Conducting nanofibers can enable the effect of electroporation for water disinfection with high speed and low cost.

Lastly, metallic nanofiber networks offer exciting solution as transparent conducting electrodes. They have low sheet resistance and high optical transparency, and are mechanically flexible. They can be promising candidates for solar cells, touch screen, and display.
Progress and Perspectives of Electrospun Nanofibrous Materials

Jianyong Yu
Innovation Center for Textile Science and Technology, Donghua University, Shanghai, China
yujy@dhu.edu.cn

Electrospinning is a versatile and straightforward approach to fabricate nanofibers with controllable compositions and structures for a wide range of applications in broad implications ranging from thermal insulation, tissue engineering, and electrical devices to environmental remediation.[1] Whereas, the current electrospun nanofibrous materials gradually emerges some bottlenecks, involving the brittleness of one-dimensional (1D) ceramic nanofibers, the difficulties in thinning the fiber diameters and optimizing the torturous porous structures of two-dimensional (2D) nanofibrous membranes, and the anisotropic lamellar deposition property of three-dimensional (3D) nanofibrous materials, which have significantly limited their practical applications. Aiming at addressing the current challenges of nanofibrous materials, we carried out a systematic study towards the controllable fabrication and hierarchical structure regulation of 1D soft ceramic nanofibers (SiO₂, TiO₂, Al₂O₃, etc.), 2D ultra-fine nanonets with extremely small diameter (<20 nm) and tortuous nanofibrous membranes with tunable surface wettability, and 3D isotropically assembled nanofibrous aerogels for multifunctionalities in terms of high temperature heat insulation, filtration, waterproof and breathable fabrics, oil-water emulsions separation, sound absorption, etc.[2,3] Such progress might open up the possibility of moving beyond our current alternative to ensure sustainable lives for the future.

REFERENCES
Nature has created many natural protein polymeric materials suitable for use as fibers – like spider silk. Bolt Threads has developed a molecular biology platform to mimic and/or tune the properties and inherent environmental compatibility of these materials and manufacture them via large-scale fermentation. Our platform includes genetic-level control over the amino acid sequence, allowing us to fine-tune the polymer processing windows and functional material properties.

Today, Bolt Threads is capable of producing a recombinant spider silk protein at commercial scale and spin this material into filament and staple yarns. Bolt Threads filaments exhibit spider silk biomimicry and can be generated with high consistency and at large volumes. This first fiber demonstrates the baselined capability of Bolt Threads’ designer protein polymer pipeline. The mission of Process Development at Bolt Threads is to continue to optimize and deploy scalable, economically viable processes for the production of the natively-inspired bio-derived materials with sustainability, performance, and market advantages.

This session will describe our first commercial product, a recombinant spider silk protein which we will spin into filament and staple yarns, as well as our most recent launch of Mylo™ a mycelium leather.

A little more about Bolt Threads. Bolt Threads is a vertically integrated company driven by 100+ dynamic scientists, engineers, artists, and operations specialists. At Bolt, our endeavors include molecular biology and materials science R&D, polymer production, fiber manufacturing, product development, marketing, merchandising, and direct to consumer sales. Bolt Threads operates direct to consumer through Best Made Co. and partners with well-known brands such as Stella McCartney and Patagonia.

REFERENCES
www.boltthreads.com
Fiber Manufacturing and Characterization
Dynamic Cross-Linking Spinning: A New Strategy for Spinning Hydrogel Fiber

Guoyin Chen, Tao Chen, Kai, Hou, Meifang Zhu

State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, Donghua University, Shanghai China

zhumf@dhu.edu.cn

Recently, due to the reason of the particular spatiotemporal properties such as high length/diameter ratio, flexibility and knittability, hydrogel fibers have been gained large attentions in fields of biomimetic device, biomaterials, signal transmission media and so on. Several techniques have been established to fabricate hydrogel fibers, including wet spinning, electrospinning, extrusion, 3D printing and microfluidic. Among these, fabricating hydrogel fiber from monomer is easily functionalize and structure design, however, there still exist challenges to product hydrogel fibers in large scale with uniform structure by a convenient method. Here, we presented a novel dynamic-cross-linking spinning (DCS) method for directly fabrication of size-controllable chemical crosslinked PEGDA fibers continuously, and the influences of spinning parameters on structure, diameters, and swelling behavior of hydrogel fiber have been analyzed systematically. Furthermore, based on this methods, we developed a series of hydrogel fibers with different structures and functions, such as composite hydrogel fibers, hollow hydrogel fibers, core-sheath structured hydrogel fiber, temperature-sensitive hydrogels as shown in Figure 1, which have great potential applications in biosensing, energy storage, and photomedicine.

REFERENCES

ACKNOWLEDGMENT
This work is financially supported by National Natural Science Foundation of China (NO. 51733002); National Key Research and Development Program of China (2016YFA0201702/2016YFA0201700); The Fundamental Research Funds For The Central Universities (2232018A3-01); Program for Changjiang Scholars and Innovative Research Team in University (IRT 16R13); Science and Technology Commission of Shanghai Municipality (16JC1400700) and Innovation Program of Shanghai Municipal Education Commission (2017-01-07-00-03-E00055)
Electrospinning is widely used for fabricating nanofibrous materials for various applications including biosensors, optical polarizers, fuel cell gas diffusion layers, tissue scaffolds, filtration materials, as well as drug delivery systems. In many of these applications, it is desirable to produce nanofibrous materials of pre-determined fiber orientations, structures and geometries for enhanced performance in many applications. Nevertheless, it is inherently challenging to fabricate electrospun nanofibrous materials of controlled fiber orientation and structure as the electrospun polymer jet tends to whip randomly due to the bending instabilities resulted from the variability of adjacent ion interactions and viscoelastic properties. Historically, controlling electrospun fiber orientation has been attempted through electrical, magnetic and mechanical means with limited levels of success. Here we report on a novel technique of manipulating the polymer jet path to control the fiber orientation and fabricate nanofibrous materials of pre-determined geometries, by a set of intermediate electrodes with rapidly changing polarization via AC switching amplifiers. Star, triangle, hexagon and circular shaped nanofibrous geometries with controlled fiber orientations were spun with the new technique using a 10% polyethylene oxide (PEO) polymer solution.
Artificial Spider Silk: Toward Sustainable and Resilient Future

Jinlian Hu, Harun Venkatesan, Jianming Chen, Lin Gu, Yuangzhang Jiang

Institute of Textiles and Clothing, The Hong Kong Polytechnic University
jin-lian.hu@polyu.edu.hk

Spider dragline silk is well-known for their excellent strength, extensibility, toughness and biological properties which overwhelmingly exceed most of the engineered materials available today. Artificial Spider silk proteins have been spun using wet spinning, dry spinning and electrospinning methods. Wet spinning is found to be successful among other methods due to their versatility to adapt various spinning conditions required to produce superior fiber properties. However, the current systems of artificial spinning methods are lacking chemical and physical triggers involved in spider silk spinning process. In natural spinning of spider silk, self-assembly of spidroin oligomers form micelle like structures. These micelle-like structures channeling through spinning duct undergoes changes in chemical environment (pH & ion gradient) and shear forces subsequently accompanied by elongational flow that increases hydrophobic interaction and promote formation of β-sheets via liquid-liquid phase separation process. We understood that biomimicry is essential in artificial spider silk spinning to attain properties comparable to their natural counterparts. Our research team have opted chemical polymerization and recombinant routes to study artificial spider silk-like proteins and their respective fibers. Recombinant spider silk proteins are spun into fibers using two different methods. Firstly, we had utilized liquid crystalline theory to produce fiber by controlling micellar spider silk protein (eTuSp1) in bioinspired microfluidic device followed by nonsolvent induced phase separation process. Microfluidic channel is tapered in a way mimicking the spinning duct of spider to induce mechanical shear to the protein. Secondly, fiber formation is achieved through lock-trigger mechanism of engineered NT & CT terminal regions of spider dragline silk protein (MaSp1 & MaSp2) and spinning conditions were set to mimic pH gradient and ion-exchange process of spider silk glands. Such biomimetically spun fibers demonstrated humidity responsive behavior like their natural counterparts. However, recombinant technology is quite successful in producing artificial spider silk protein but their scalability remains a challenge. Scalability and controllable hierarchical structure can be obtained by synthetizing silk-like peptide polymers hybrids. Our bioinspired chemical approaches to produce artificial spider silk proteins are accomplished by introducing short chain polyalanine (PA) into multiblock polymers, amine-initiated ring opening polymerization of BLG-NCAs to form PBLG-PPG-PBLG peptide segments. Synthetic silk-like polypeptide fibers are wet spun and applied suitable post drawing techniques to improve their molecular orientation.

KEYWORDS: Artificial spider silk; biomimicry; recombinant technology; chemical polymerization.
Cotton Fiber Creep Behavior: Viscoelastic Modeling and Bundles Friction

Wafa Mahjoub¹, O. Harzallah¹, J-P. Gourlot², J-Y. Dréan¹

¹Laboratoire de Physique et Mécanique Textiles, Université de Haute-Alsace, France
²Centre de Coopération Internationale en Recherche Agronomique pour le Développement, Montpelier, France

wafa.mahjoub@uha.fr

INTRODUCTION
Fiber mechanical properties is one of the key properties of cotton and has important influences on yarn production and yarn quality. Various tests have been developed to characterize cotton fiber behavior. This study was carried out to investigate the effects of the tensile and creep tests on bundles. Analogical models with different fiber parameters were studied for predicting a general model valuable for every different cotton fiber variety.

APPROACH
The time-dependent behavior of cotton fibers may be studied by conducting tensile and creep experiments. The aim of the whole research is to discuss the basic concepts of viscoelastic behavior of cotton fibers as well as modeling the creep response. The inter-fiber friction is also studied as far as it is a very important component. Four cotton fiber varieties have been studied in both single and bundle. Prior to testing, all cotton samples were conditioned for at least 48 hours in standard conditions (HR% = 65±4%, T = 20±2°C).

RESULTS AND DISCUSSION
Cotton is viscoelastic. Viscoelasticity is the property of materials that exhibit both viscous and elastic characteristics when undergoing deformation. Cotton fiber response to creep test has been modeled to a dashpot (Part 1 in Fig. 1, representing the viscous element $\eta_1$) in series with a Kelvin Voigt model (Part 2 in Fig. 1, in which a spring represents the elastic element $E$ and a dashpot represents the viscous element $\eta_2$) are connected in parallel.

In this research, both single and bundles testing methods have been used. The four different varieties of cotton fibers studied are named C07, C42, C55 and C56. They have different physical properties (such as maturity, fineness, micronaire, length, tenacity, etc.). We conduct that the difference in the physical properties influences the mechanical one, determined from the creep tests, and are related to the inter-fiber friction (the friction component K). We conduct also that the inter-fiber friction is in the origin of the difference between the second part viscosity ($\eta_2$).

CONCLUSION
The relationship between fibers and yarn structure taking in account the inter-fiber friction is, in part, a perspective of our research. Now, analogical models for both single and bundle cotton fibers have made a big advance in understanding cotton’s viscoelastic behavior as well as the influence of the mechanical parameters.
Structure and Properties of PET Fibers Prepared with Infusion of Ethanol in Continuous Cold Drawing Process

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INTRODUCTION

Our earlier studies focused on the fiber formation behavior upon continuous drawing of as-spun amorphous poly(ethylene terephthalate) (PET) fibers through an ethanol bath at room temperature. As in the case of batch-type drawing in ethanol, multiple-necking behavior and infusion of ethanol were confirmed. Unstable state of process with the large fluctuation of drawing tension and the lack of uniformity of neck formation were observed. However, by installing a snub pin in the ethanol bath, the stability of the process as well as the efficiency of ethanol infusion were improved. In order to assess the potential of this unique process for the industrial application, multi-stage drawing was conducted in this study for the further improvement of mechanical properties.

EXPERIMENTAL

Low-oriented amorphous PET fibers were prepared through the melt spinning process at a take-up velocity of 400 m/min. In the continuous drawing process of the fiber in ethanol, a snub pin was installed near the inlet of an ethanol channel to improve process stability as shown schematically in Figure 1. In order to obtain a uniform fiber, the machine draw ratio was increased up to the natural draw ratio to prevent the remaining of undrawn part. For comparison, some samples were prepared through the drawing in air at room temperature or by applying the multi-stage drawing. The structure and mechanical properties of the resultant drawn fibers were evaluated by the X-ray analysis and the tensile test.

RESULTS AND DISCUSSION

DSC measurement of the drawn fibers confirmed the infusion of ethanol into the fiber through the cold drawing in the ethanol bath. Development of highly oriented crystalline structure caused by the ethanol induced crystallization was revealed from the appearance of crystalline reflections in 2D-WAXD. Stress-strain curves of the prepared drawn filaments are compared in Figure 2. The elongation at break of the ethanol-drawn fiber was greatly increased without lowering the tensile strength as compared with the air-drawn fiber. Further increase of tensile strength through the addition of second-stage drawing was also confirmed.

Figure 1. Schematic of the system for continuous cold drawing of PET fiber in ethanol channel equipped with a snub pin.

Figure 2. Tensile properties of PET fibers prepared through the single- and multi-stage cold drawing with and without ethanol bath.
Image Processing Technique for Cotton Color Grading

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Cotton color measurement is very important property which plays important role in determining the cotton grade. Although other physical properties like length, strength, fineness and maturity are also very important in determining the cotton color grade, in aesthetic property cotton color is very important. There are different methods used to measure the cotton color in cotton growing countries globally. Use of HVI (High Volume Instrument) is a common practice for cotton color measurement along with other physical properties. But, the final color grade mostly awarded on the basis of visual inspection. There is a strong disagreement between the visual color grading and instrumental color grading.

One of the biggest problems in this disagreement is the presence of trash particles on the surface of cotton sample. The instrumental measurement always consider the trash particles as color deterioration of cotton fiber but, actually these trash particles can be removed prior to yarn manufacturing process which results in better color grade. So, the visual inspection does not take into consideration the trash particles and give color grade quite different than instrument measurement.

In this research we tried to develop a programme using image analysis technique which will measure the cotton color close to the visual inspection. The basic principle of this technique is that it does not take into consideration certain trash particles present on the surface of cotton samples while giving color grade to the cotton sample. This technique is based on two important color factors lightness and chroma. The final grade assigned to the cotton is based on the cotton region and trash region is excluded from the surface and the color values of the trash region do not disturb the final color grade. The principle of this instrumental measurement is based on the visual grading system. The results obtained from this image analysis tool are satisfactory and the disagreement between the instrument and visual measurement can be minimized. The reduction of the disagreement between the instrument and visual color measurement is a great achievement in the cotton industry and the cotton color grading will be more precise.

Figure 1. Lightness and chroma values shown on the cotton sample.

Turkish cotton samples and also ceramic tiles used for the evaluation. AMS standard ceramic tiles (two boxes, n=10) one box is been provided with the xenon flash light standards and the other one is provided with the Incandescent light source standards and the HV standards are provided by the AMS. These standard ceramic tiles are well prepared possess a smooth surface for the evaluation. White, brown, yellow, grey and central,
these five colors for each set of box were provided with the standards readings. Figure 2 shows the standards and samples used for the measurement. The ceramic tiles were measured with the Hunter Lab.

*MiniScan Portable Color Spectrophotometer.* As discussed earlier, AMS provided with the HVI Rd and +b values as reference values the data obtained from the Hunter Lab MiniScan is compared with the provided data. For all the measurement five replications were made and average per sample was taken then average Hunter Lab values were compared with the HVI values. All the measurements were performed at the laboratory conditions (70 ± 2 °F and 54 ± 2% RH).

For the visual grading of cotton samples in a light cabinet AT color used and common, untrained observers were invited for the color measurement of cotton samples instead of professionally trained cotton classers. And these observers were asked to grade the cotton samples by taking the ceramic tiles as reference measurement. Five replications again were taken from each observer for five different days. Each observer was fully aware of the international cotton grading system, but they never performed the cotton grading as they were untrained people. This phenomenon was performed for set of standards (xenon, incandescent). To measure the non-contact method color measurement, Konica Minolta CA-210 was used for the telescope measurement in the Laboratory of Color and Appearance Measurement, Technical University of Liberec. An illuminant D65 is used at the 10 degree observer. As this is non-contact method, the distance from probe of the telescope and the sample is 7cm. A light cabinet as mentioned earlier again used to illuminate the sample and the readings of x, y and Lv were taken. We used some color space conversion calculations to convert these readings into the Rd and +b value and then the relationship between the visual grading and non-contact method was studied. So, two relationships are studied here in the first relationships it was studied that whether the HVI standard provided by the AMS (Memphis, Tennessee, USA) have relationship with the non-contact measurement method and then the visual inspection was compared with the instrumental color measurement taken in the laboratory by taking the AMS standards as reference standards. The spectral values of the cotton samples were taken from the Hunter Lab and these spectral values used for the conversion of Rd values in the color space conversion formulas. The new telescopic method is never used in the cotton color measurement, which can be called as non-contact method.

![Figure 2. Non-contact method used for testing.](image1)

![Figure 3. AMS standards ceramic tiles.](image2)
RESULTS AND DISCUSSION
For the AMS standard ceramic tiles the reference Rd values were obtained from the master HVI-1000 colorimeter in Memphis, Tennessee, USA. The HVI colorimeter uses two filters to measure the samples diffuse reflectance at the two visible spectrum regions and the value of Rd and +b obtained from the Uster algorithms. In the Laboratory of Color and Appearance measurement in the Technical University of Liberec, two sets of tile were measured again with the non-contact method. One set for incandescent and the second with the xenon light source. Then the value of Rd and +b were compared between the HVI-1000 and with the non-contact method.

Figure 4(a). AMS standard ceramic tiles (xenon). HVI Rd vs. non-contact method Rd.

Figure 4(b). AMS standard ceramic tiles (xenon). HVI +b vs. non-contact method +b.

In the table it is clearly visible that there is a strong relationship between the HVI measurement and with the non-contact method measurements. In both the parameters (Rd, +b) the values are very close to each other with R2 value of (0.99, 0.98) respectively.

CONCLUSIONS
The color standards provided by the AMS for the cotton color measurement confirmed through non-contact method and it was confirmed that the results shows strong relationship between the two methods. The measurement of Rd values of cotton samples with the two methods mentioned above also shows strong relationship between two methods but contradiction with the visual inspection. Disagreement between the visual inspection and the instrumental grading continues as presented before this research articles. This disagreement shows that the new method (telescopic measurement) for the cotton color measurement can be implemented with some more focus in this method.
In this research, a thermoregulatory undergarment system (TRUS) integrated with the thermoelectric energy conversion unit (TECU) was developed for personal cooling in building environment. Previous studies indicate that an expansion of neutral band for building by 4°F on both the hot and cold sides will result in 15% energy saving in the heating, ventilation, and air conditioning (HVAC) system. A thermoregulatory undergarment that controls microclimate temperature of individual clothing system enables lower energy cost of the HVAC system and also can realize individual thermal comfort according to personal requirement. Based on the calculation, to rise the indoor ambient temperature from 7°F to 79°F without compromising the thermal comfort, a personal cooling system should be able to provide 23 W cooling power with at least 0.35 coefficient of performance (COP) for energy saving.

To achieve this cooling power in a hot environment (79°F), a combination of TECU and a tree-like tubing structure was developed. The TECU is capable of creating cooled air flow which is then distributed to upper chest and upper back areas by the tree-like tubing structure. The preferred tubing structure and the optimum operation condition of the TECU were screened out through a flow test. Under the optimum conditions, the maximum cooling power of the TRUS is evaluated by the non-sweating thermal manikin and measured to be 17.29 W with a COP of 0.532. This result coincides with the theoretical cooling power based on the flow test but is still lower than the target 23 W. In the following human subject test, participants who wore the TRUS felt slightly cool in the hot environment, with a skin temperature drop by about 0.54°C. It proves that the TRUS has enough cooling capacity to maintain the thermal comfort of wearers at 79°F ambient temperature, meanwhile saving energy for the HVAC system.
A biodegradable bio-based copolyester, poly (3-hydroxybutyrate-co-3-hydroxyhexanoate) (PHBH) with the 3-hydroxyhexanoate composition of 5.4 mol%, was used in this study. The PHBH has low crystallization rate. In addition, the glass transition temperature of the PHBH is lower than the room temperature. Accordingly, in the ordinary melt spinning process, the PHBH remains in the rubbery state when it is cooled down in the spin-line and wound up on the take-up bobbin. On the other hand, we have shown that by applying the high-speed melt spinning process, the PHBH can crystallize in the spinning process through the mechanism of orientation-induced crystallization. Even though highly oriented and highly crystallized fibers were obtained, it was difficult to obtain fibers having sufficiently high mechanical properties. Therefore, with the aim of improving the mechanical properties of the PHBH fibers, an liquid isothermal bath (LIB) was installed into the high-speed melt spinning process as shown in Fig.1. When the spin-line passes through the LIB, temperature of the molten polymer can be controlled to the liquid temperature whereas the frictional stress of about 100 times higher than that in the air can be applied to the spin-line in the liquid.

Tensile strength of the high-speed spun PHBH fibers and the fibers prepared using a LIB are shown in Fig.2. In the process with the LIB, the liquid temperature, the depth of the liquid bath and the take-up velocity were varied. Tensile strength of the high-speed spun PHBH fibers increased with the increase of take-up velocity from 0.5 to 2 km/min, and then levelled-off. Obtained maximum tensile strength was only around 100 MPa. On the other hand, tensile strength of the fibers prepared with the LIB increased with the reduction of liquid temperature and with the increase of take-up velocity. At the take-up velocity of 0.5 km/min, effect of the liquid depth was not clear, whereas at the take-up velocity of 1 km/min, liquid temperature of 40 °C and liquid depth of 20 cm, the fibers with the tensile strength of 176 MPa could be obtained. The WAXD measurement revealed that along with the highly oriented α-form crystals, co-existence of the β-form crystals was confirmed for the PHBH fibers of high strength prepared using the LIB.
Preparation of Polyether-ester Elastomeric Fibers Through High-speed Melt Spinning and In-line Drawing Processes

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INTRODUCTION
Elastomeric polymers are normally composed of crystalline hard-segment (HS) and rubbery soft-segment (SS). With the reduction of the composition of the HS, elastomeric property may be improved whereas the crystallizability may be deteriorated. In this research, polyether-ester copolymers (TPE) composed of butylene terephthalate (BT) as the HS and poly(tetramethylene glycol) (PTMG) as the SS were subjected to the high-speed melt spinning process with the aim of utilizing the mechanism of orientation-induced crystallization for the enhancement of crystallization, and the effect of molecular structure on the structure and mechanical properties of the as-spun elastomeric fibers were investigated. In addition, with the aim of improving the mechanical properties, the direct spin-draw process was also applied for the preparation of the TPE elastomeric fibers.

EXPERIMENTAL
Three types of TPEs were used in this research. TPE-A has the lowest weight fraction of the HS, whereas the weight fraction of the HS for TPE-B and TPE-C were similar. On the other hand, chain length of the SS was similar for TPE-A and TPE-B, whereas the SS chain length for TPE-C was about a half in comparison with that for TPE-A and TPE-B. In the high-speed melt spinning, the attainable maximum take-up velocities for TPE-A, TPE-B and TPE-C were 9, 6, and 8 km/min, respectively. In the direct spin-draw process, fibers were prepared under the in-line drawing conditions with the first roll speed of 1 or 2 km/min, and the in-line draw ratio of up to 3.8x. The temperature of the rolls was kept at room temperature.

RESULTS AND DISCUSSION
In the high-speed melt spinning of the polymer with the lowest weight fraction of HS (TPE-A), sticking of the fibers on the take-up bobbin was observed at low take-up velocities because of its low crystallizability, whereas the highly crystallized and well-separated elastomeric fibers were obtained at high take-up velocities. On the other hand, when the results for the two types of TPEs with similar weight fraction of HS but different chain length of the SS were compared, it was found that the crystallizability judged from the neck-like deformation behavior in the spin-line of the polymer with longer SS (TPE-C) was slightly higher than TPE-B presumable because of the higher molar fraction of the HS. Contrary, tensile modulus for the as-spun fibers of TPE-C was significantly higher than that for TPE-B.

In the high-speed spinning of the elastomeric fibers, elastomeric contraction of the fibers occurred when the fibers taken-up on the bobbin were cut-off. This is because high spin-line stress caused by inertia and air-friction forces was applied to the elastomeric fibers of low tensile modulus before the winding. Similar or more enhanced contraction behavior was expected for the direct spin-draw process because only the elastomeric extension can be applied during the in-line drawing process between the first and second rolls. Surprisingly, elastic contraction of the fibers wound up on the second roll was much higher than the value predicted from the elastic contraction for the take-up velocities of 1 or 2 km/min and the draw ratio. This result suggested the continuation of structure development of elastomeric fibers between the two rolls in the spin-draw process. The fibers prepared in this process showed higher tensile strength in comparison with the high-speed spun fibers with the similar level of elongation at break. In addition, these fibers exhibited higher elastic recovery after applying 400% or more of elongation.

REFERENCE
Japan Patent 5999630
Fiber-based Sensors and Intelligent Products
Using intrinsically conductive polymer (ICP) to fabricate conductive fibers demonstrates great potential and low-cost merit for the development of smart textiles. The challenges of using ICPs, such as conjugated conductive polymers, are their low solubility and poor processability for fiber spinning. Polyaniline (PANI) is one of the most representative ICPs and has many advantageous properties, such as low cost of the aniline monomer, easy polymerization, biocompatibility, and environmental stability. Currently, PANI is mostly processed by in-situ polymerization onto fabrics or by blending with other polymers to spin fibers for conductive textiles. In this study, a simple barbed Y-connector as a “2-in-1” spinneret was applied for the electrospinning of heterogeneous nanofiber consisting conductive PANI and nonconductive Polyethylene Oxide (PEO) in a side-by-side fashion. PEO was selected because of its good mechanical properties and softness, as well as versatility in different applications. The PANI/PEO fiber mat was characterized for the morphology, mechanical properties and electrical resistance. The results showed decent mechanical ductility (200-600% elongation at break), low rigidity (<100MPa tensile modulus) and good electrical conductivity ($10^{-3}$-$10^{-5}$ s/cm) in the direction of fiber alignment. Constant changing tendencies were observed for these properties when the content of PEO, PANI and their weight ratio were varied. The electrical conductivity of the side-by-side nanofiber mat was much higher than that of PANI/PEO blended fiber mat. The results suggested that PEO served as a substrate for PANI to construct conducting path along the fiber length, and the use of the 2-in-1 spinneret provided a solution for electrospinning of non-spinnable materials.
Biomimetic actuators with rapid response speed, high sensitivity and selectivity to external stimulus have found potential applications in smart switches, artificial mussels and soft robots. The nano-scale structures of actuators enhance the exposed area to the stimulus, as well as enable the versatile controls of the actuation behaviors. The freestanding, flexible and porous water driven actuators with poly(vinyl alcohol-co-ethylene) (EVOH) nanofibers as substrate and super hydrophilic nano-scale cellulose materials (cellulose nanofibers, cellulose nanocrystals, bacterial cellulose) as active substance via uniform mixing or surface depositing were fabricated. The effects of the EVOH nanofiber substrate, the structures and concentrations of nano-scale cellulose materials, as well as the different environmental stimulus like humidity, temperature on the performance of actuators were studied. The water driven actuation mechanism was proposed from the macroscopic, molecular aspects and the analysis of Gibbs free energy and mechanical energy. The actuator could bend to an angle of 180° and recovered less than one second for more than 100 circles without compromising properties when the environmental moisture changed. Furthermore, the multidimensional deformation behaviors of the water stimulated actuators could also be well tuned by varying the orientations of the nano-scale materials. Additionally, the applications of the prepared actuator were demonstrated.
Cellulose Nanofibrils Exfoliated Graphene for Moisture-responsive Actuator

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Graphene has unique properties such as high transparency, density, electric and thermal conductivity, etc. Typical processes to exfoliate graphite into graphene, such as mechanical exfoliation, micromechanical cleavage, chemical vapor deposition, and liquid phase exfoliation, have limitations, i.e., extremely low yields (i.e., only 0.05 mg mL⁻¹), substantial residual exfoliating agents, such as surfactant (up to 35 wt.%), requiring exceedingly long time (sonication up to 400 h), and substantially decreased electrical conductivity (from 65 to 15 S cm⁻¹). Therefore, effective and efficient methods for the mass production of this remarkable material are still in need of development.

In this study, the uniquely amphiphilic surface characteristics of cellulose nanofibrils (CNFs) has been demonstrated as dual exfoliating agent and dispersant for processing graphite into aq. graphene suspension. This is based on similar concept of surfactant that the hydrophobic surfaces of CNFs adhere to graphene whereas the negatively charged hydrophilic carboxylates stabilize the graphene in aqueous dispersion and prevent aggregation.

Cellulose was extracted from rice straw by a three-step procedure. Cellulose nanofibrils (CNFs) were produced as 0.67 wt% aq. suspension by 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO)-mediated oxidation (5 mmol g⁻¹ NaClO/cellulose) followed by blending (37,000 rpm, 30 min) also previously reported. Aq. cellulose nanofibrils suspension was then mixed with graphite flakes and blended to produce aq. graphene suspensions. Graphene yield was optimized by varying blending speed and CNF:graphite feed ratio. Aqueous graphene dispersions with concentrations at up to 1 mg mL⁻¹ at an outstanding yield of 84.2 wt%. The graphene produced were 0.7 – 3 µm in lateral dimensions and 50% as less than triple layers (0.7-0.9 nm) and 47% multilayers (1.0-10.0 nm), leaving only 3% is un-exfoliated graphitic platelets with >10 layers. For the first time to our knowledge, graphene/CNF paper made from suspensions has unique moisture-triggered responsivity characteristics because the hygroscopic nature of CNF in the graphene/CNF thin films allowed it to act as actuators that response to moistures. The solution processing of graphene/CNF should open a wide range of potential applications.

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ACKNOWLEDGMENT
This work is supported by the California Rice Research Board (Project RU-9).
Preparation of Conductive Fiber Assemblies and Their Applications on Flexible Sensors

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Wearable strain and pressure sensors with ultrahigh sensitivity and flexibility have been tremendously concerned due to their abilities to mimic human somatosensory system and percept surrounding strain and stress as well as pressure distribution.\textsuperscript{[1-3]} Various materials and structures including polydimethylsiloxane (PDMS), polyurethane (PU), Ecoflex and rubber are designed to assemble wearable strain and pressure sensors with high performance. However, to achieve superior three-dimensional (3-D) conformability to the complex surface of the human body, fiber-based materials are one of the most suitable candidates for its flexibility, drapability and comfortability.

Herein, we proposed a novel continuously producible method for elastic fibrous composite yarn which is composed of polyolefin elastomer (POE) fiber yarn and three-dimensional interpenetrating silver wires. And the conductive yarn was applied in wearable strain sensors which achieve high gauge factor (GF) value as 13920, low detection limit as 0.065\%, the fast response time of 10 ms and 15 ms under stretched and released (seen in Fig. 1) Furthermore, promising applications in human motion capture, expressions diagnose, artificial vocal cords and human pulse monitoring are successfully operated, which indicates the non-negligible potential of demonstrated fibrous strain sensors.

After that, an ultrasensitive fiber based pressure sensor was fabricated via constructing surface patterns via replicating the texture of an available plain-weaved nylon textile. The influence of the geometry of the surface pattern and the density of the additive conductive component to the sensing performance was systematically investigated. As a result, the pressure sensor showed excellent sensing performance, including the high sensitivity of 19.4 kPa\textsuperscript{-1} below the pressure of 2.76 kPa, low detection limit of 1.7 Pa as well as excellent durability. The pressure sensors demonstrated the promising potential for wearable electronic applications, like detecting the spatial pressure distribution and monitoring the human muscle motions.

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ACKNOWLEDGMENT

The authors thank the Nature Science Foundation of Hubei Province (No. 2016CFA076) and the Fundamental Research Funds for the Central Universities (No. CUSF-DH-D-2018059) for their financial support.
Nanofibers and Nanofibrous Materials
Unidirectional Moisture Transport Nanofibrous Membranes

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Moisture transport properties of porous materials play a critical role in maintaining the comfort of body which are mainly controlled by surface chemistry, structure and morphology of substrate. We provided an overview of our recent works on fibrous membranes for unidirectional moisture transport.¹ We fabricated a dual layer surface treated nonwoven (NW)/electrospun nanofibrous membranes with excellent unidirectional liquid moisture transport characteristics. In the resultant membranes, surface functionalized NW facilitates the moisture transfer, additionally, nanofibrous layer rapidly pulls out moisture from NW layer which accelerates in the forward direction (i.e., nonwoven to nanofiber direction). Whereas, the water droplet spread rapidly on the nanostructured surfaces due to its extremely hydrophilic nature of nanofiber, therefore, liquid molecules don’t have enough force to move.² Use of color in textiles helps in developing aesthetically pleasing apparels, in the second work, we report an aesthetically pleasing and breathable dual-layer cellulose acetate (CA)/dyed CA nanofiber membrane with exceptional directional moisture transport performance.³ In our recent work, we report a scalable strategy to create trilayered fibrous membranes with progressive wettability by introducing a transfer layer, which could guide the water transport continuously and prevent the inner layer from being rewetted. The resulting trilayered fibrous membranes exhibited a high one-way transport index R (1021%) and a desired breakthrough pressure (16.1 cm H₂O) in the reverse direction, indicating a continuous directional water transport property. The successful synthesis of such fascinating materials would be valuable for the design of moisture management textiles with directional water transport property for personal drying applications.

REFERENCES

Sensitive and inexpensive CO₂ sensors comparable to commercial CO₂ sensors in performance are being made using conducting polymer-Polypyrrole (PPy). PPy nanofibers 100-400 nm in diameter showing conductivity in the range of 3.66*10⁻³ S/m-7.04*10⁻³ S/m were produced using electrospinning process. Electrospinning involves applying high voltage electric force to a polymer jet which results in formation on nano to sub-micron range ultrafine fibers. Stable, doped polymer dispersion of PPy and Polyethylene Oxide (PEO) as a carrier were used for electrospinning. Eight electrospinning parameters were studied to optimize the collection of highly conductive and sensitive PPy nanofibers; with high surface area suitable for CO₂ sensitivity. PPy being a conducting polymer holds potential to be used in gas sensing and electronic applications in nanofiber form. PPy structure allows for delocalization of electrons along the pi-orbitals of the polymer chain giving conducting properties. CO₂ molecules can interact by forming weak bonds with these pi-orbitals. This gives a change in resistivity on exposure of PPy to CO₂ gas. SEM, TEM, Four-probe conductivity measurement and elemental analysis were used to characterize the nanofibers. Laboratory scale set-up is being used to measure sensitivity to CO₂.
Constructing Fiber-based Flexible Triboelectric Nanogenerator with Electrospinning Techniques

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With the rapid advancement of modern technology, wearable electronic devices become more and more indispensable to daily life. However, powering them in a stable and sustainable manner remains a challenge and highly desired. Human biomechanical motions, such as walking, arm swinging, typewriting, finger motion, and even breathing, available in large quantities, are clean and renewable energy sources with great potential. The bottleneck in the development of wearable electronics could be broke if these energy sources could be effectively harnessed. Reliance on the coupling effect of contact electrification and electrostatic induction, triboelectric nanogenerators (TENG) have been proven to be a fundamentally new green energy technology for ambient mechanical energy harvesting, featured as being simple, reliable, cost effective as well as high efficiency. Here, we developed a lightweight, flexible and sustainable power source by fabricating a nanofibrous membrane constructed wearable TENG, which is capable of converting human biomechanical energy into electricity for next-generation wearables. And the electrospun nanofibrous membranes were tailored to enhance the triboelectric polarity, mechanical strength as well as surface hydrophobicity, which will eventually improve the device output performance, robustness and capability of operation even with high environmental humidity. Via harvesting the biomechanical energy from body motions, the wearable TENG was demonstrated to sustainably power a commercial thermal meter, a calculator, an electronic watch, and light up about 560 LEDs. Given a collection of compelling features of being flexible, breathable, environmentally friendly and cost-effective, the fabricated TENG can be extensively applied not only to self-powered wearable electronics but also possibly to power generation at a large scale.
Sub-nano Thick Silk Nanoribbons Exfoliated from Silkworm Silk

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Native silk nanoribbons (SNRs) with a height of around 0.4 nm were exfoliated from natural silkworm silk (DS) by partially dissolving DS in alkaline solution. In the process of dissolving, the solvent does not destroy the nanofibril structure completely, but influenced the dimensions of SNRs. Transmission electron microscope, atomic force microscope, synchrotron radiation small angle X-ray scattering, and molecular dynamics simulations were applied to confirm the single molecular layer nanoribbon. It was found that SNRs containing single β-sheet layer and amorphous structure are the basic building blocks of the DS with hierarchical structures. The novel partially dissolution method for fabricating SNRs may pave a new route for creating stronger and tougher silk based materials and further ensure the functionality and durability of materials in various applications, such as filtration, tissue engineering, biosensing and optics.

ACKNOWLEDGMENT
This work is supported by the National Natural Science Foundation of China (21674018), and the National Key Research and Development Program of China (2018YFC1105802, 2016YFA0201702).
Enhanced Piezoelectric Response of Electrospun PVDF with ZnO Nanorods

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A novel breathable piezoelectric membrane has been developed by adding zinc oxide (ZnO) to electrospun polyvinylidene fluoride (PVDF) nanofibers. Notable improvements in the piezoelectric response of PVDF membrane was achieved without compromising breathability, conformability, or safety of the material. Three ZnO nanorod addition method was investigated: fillers in electrospinning solution, hydrothermal growth on the fiber surface, and electrospraying on the PVDF membrane surface. PVDF is one of the most frequently used piezoelectric polymers due to its high piezoelectric coefficient values. However, its piezoelectric response requires further improvements for the use of high-performance sensors or energy harvesters. Previous studies have demonstrated piezoelectric ceramic and polymer composites with a remarkably improved piezoelectric constant. However, such composites often lack breathability, and some contain piezoelectric ceramics with heavy metal which limits its use in wearable applications. The said limitations can be alleviated by electrospinning piezoelectric polymers into porous membranes, and by selecting non-toxic piezoelectric ceramics. Unlike many piezoelectric ceramics containing heavy metal, ZnO is a non-toxic material which has been widely used in many fields of applications including cosmetics. The fabrication process is simple and economical due to no additional poling process needed for PVDF membranes after electrospinning in a high electric field.
Flexible, Ion-conducting Ceramic Textiles

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Fibrous ceramic fabrics are commercially available products in different technical fields. The fabrication technologies of fibrous ceramic fabrics are mainly categorized into direct spinning process and indirect template process. Here, we employed the template process to create the proof-of-concept lithium ion conductive fibrous ceramic fabrics, which generates unique structure with fine 3D scale distribution of continues lithium ion conductive phase, high surface area/volume ratio, low gravimetric density, multi-level porosity, certain strength and flexibility, for solid-state safe, high energy Li-metal batteries. The architectural advantages may allow the fibrous ceramic fabrics to be integrated to build components of batteries, such as flexible composite polymer electrolyte and rigid electrode skeleton. The fibrous garnet textile shows pronounced new characteristic features to tolerate certain flexural strength, geometrical tailoring and organic solvent erosion, which are distinct from the rigid appearance of typical sintered ceramic body. The chemical analysis confirmed cubic phase of the fibrous garnet fabric and homogenous distribution of the constituent elements. Because of the simplicity, rapidity and cost saving characteristics of template method, applicable transition from laboratory scale fabrication procedure to industry scale manufacturing is potentially achievable.
Designing and fabricating nanofibers from cellulose, the nature’s most abundant and renewable polymer, has been met with the challenges of its dissolution, fiber spinning and the resulting change from the native Cellulose I crystalline structure, among others. Native cellulose has already been processed into many useful forms of fibers, pulp, paper and composites. To further advance cellulose materials science and technology, this paper highlights diverse top-down and bottom-up approaches to generate ultra-fine cellulose fibers with cellulose I and cellulose II crystalline structures. Cellulose derivatives offer ready dissolution and efficient nanofiber engineering via electrospinning into varied nano-scale dimensions and geometries, meso-porous and sheath-core hybrid structures as well as surface active fibrous materials and nanocomposites. Highly crystalline Iβ nanocelluloses can be efficiently derived in controlled geometries and surface chemistries. Ways to assemble nanocellulose into highly crystalline nanofibers and functional materials such as fibrous membranes, amphiphilic aerogels and super-absorbent will be presented and analyzed in their similarities as well as distinctive differences, in particular their crystalline structures, thermal behavior and chemical functionalities as novel functional materials.

ACKNOWLEDGMENT
Funding from USDA-NIFA, California Rice Research Board, Chevron and AgTech Innovation Center is appreciated.

REFERENCE
Electrospun Nanofibrous Aerogels: Progress in Materials, Properties, and Applications

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Electrospun nanofibers, as the forefront of advanced fibrous materials, hold extraordinary potential applications ranging from environmental, energy to biology owing to their integrated advantages of fine diameter, extremely high aspect ratio, and ease of scalable synthesis from various materials. Despite their outstanding potential, the major problem associated with electrospun nanofibers is their anisotropic lamellar deposition character, which leads to the bottlenecks in further improving the thickness and porosity of current electrospun nanofibrous materials. Alternatively, three-dimensional nanofibrous aerogels (NFAs) with both high porosity and excellent compressive resilient might open up the possibility of solving the above problem and expand the applications of electrospun nanofibers; however, creating such NFAs has proven extremely difficult. Herein, we demonstrate a novel strategy to create fibrous, isotropically-bonded elastic reconstructed (FIBER) NFAs with a hierarchical cellular structure and superelasticity by combining electrospun nanofibers and the fibrous freeze-shaping technique. Our approach causes the intrinsically lamellar deposited electrospun nanofibers to assemble into elastic bulk aerogels with tunable densities and desirable shapes. The resulting FIBER NFAs exhibited densities of > 0.12 mg cm\(^{-3}\), rapid recovery from deformation, slight plastic deformation with 14.5% after 1000th cyclic compression at a large strain of 60%, efficient energy absorption, and multifunctionality in terms of the combination of sound absorption, warmth retention and oil/water emulsion separation. Furthermore, the corresponding nanofibrous hydrogels with ultrahigh water content (99.8 wt%) were also prepared and exhibited robust elastic-responsive sensitivity. Additionally, the newly developed ceramic nanofibrous aerogels possess low thermal conductivity (~0.025W m\(^{-1}\) K\(^{-1}\)) and intriguing temperature-invariant superelasticity to 1100°C. The successfully synthesis of such fascinating FIBER NFAs provide a new insight into the design and development of multifunctional NFAs for various applications.

Figure 1. (a) Schematic illustration of the fabrication of FIBRE NFAs; (b) a 20 cm\(^3\) FIBER NFA (\(\rho = 0.12\) mg cm\(^{-3}\)) stands on the tip of feathers; (c) FIBRE NFAs with diverse shapes; (d) the water retention capacity of the nanofibrous hydrogels (NFH): a sample of squashed and dried NFH of ~10 mg could hold ~5 g of water; (e) ceramic nanofibrous aerogels heated by a butane blowtorch without any damage.
Novel Mineralized Nanofibers for Water Remediation: Discussion of Their Synthesis and Performance

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Semiconductor metal carbonates and metal oxides are considered by many for studies of environmental remediation. Rather than use these photocatalysts and adsorbents as loose nanoparticles for environmental effectiveness, we propose the nucleation and growth of these minerals from the surface of nanofibers. Further, nanofiber mineralization offers scalable approaches to functionalizing fibrous materials for water filtration. In this work, we will discuss the formation of mineral coatings by double displacement precipitation reactions and sol-gel synthesis. Further, we describe structure and property relationships that will affect their functionality.

Metal carbonates of the lanthanum rare earth metals had shown impressive adsorption capacities against phosphate ions (more than 90 mg/g of material in 24 hours, see Figure 1). The alkali metal halide of precursor metal carbonates affected the morphology and adsorptive capacities of lanthanum carbonates at high and low pH over time. Time-dependent studies had shown phosphate ion adsorption over 200 mg/g. Lastly, we explored the role of nanofiber surface chemistry on the formation of titanium oxide (TiO₂) mineralized nanofibers. Changes in surface chemistries of poly(vinyl alcohol) nanofibers were incurred by electrospinning at voltages greater than 15 kV. The more hydrophilic surface resulted in wrinkled, crosslinked PVA- as sol-gel synthesis persisted over 3-5 cycles of dipping in TiO₂ precursors, see Figure 1-. This low-temperature processing of TiO₂ has generally resulted in adsorption opposed to photocatalytic degradation.

Figure 1. Morphologies of lanthanum carbonate and TiO₂ mineralized nanofibers.

ACKNOWLEDGMENT

Water Resources Research Institute – North Carolina Sea Grant (WRRI-NCSG) provided support for this graduate research. Work from project 14-177 NC with The Nonwovens Institute was featured in this study. The authors recognize the NC State Water-Nano GRIP (Game-Changing Research Incentive Program) for their support and consideration of manganese oxide nanotechnologies for water purification. Support was received from the National Science Foundation Environmental Chemical Sciences Program (award CHE–1407180) and NCSU Research Innovation and Seed Funding program. A portion of this work was conducted at Smithsonian Institution. We thank Bruce Stewart, Cara Santelli, Dominique Caput, and Owen Duckworth for their contributions at various stages of the overall biogeochemical study. We appreciate Evan Kane at Wake County Department of Environmental Services for providing us with well water samples.
Short Nanofibers-based Hierarchical Structures for Lipase Immobilisation

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Short nanofibers are fibers with diameters in the sub-micron scale and with finite length (up to 1mm). A major advantage of short – compared to electrospun (long) – nanofibers is their suspendability in liquid media. Such advantage enables a user to use the fibers in suspension or to prepare constructs of pre-determined density, such as paper-like membranes or thin coatings. It is here proposed that short nanofibers may be useful as reusable substrates for enzyme immobilisation.

This investigation is focused on lipase immobilisation onto the surface of short chitosan nanofibers (SNFs) native or modified with other functional groups. The objective of the study was to understand how the functionality of surface modified SNFs affected lipase immobilisation and to identify optimal reaction conditions for immobilisation. SNFs were functionalized via established chemistry approaches by using two pathways, glutaraldehyde and EDC-NHS. The so-functionalized SNFs were then characterized by testing different fiber diameters or preparing constructs with different fiber-per-unit-volume density (paper, loose fibers in suspension), as the shear-dispersion approach offers a wide choice in the assembly of SNFs. The immobilisation of the enzyme on nanofibers was demonstrated, with high-efficiency protein binding and reusability. Lipase immobilised on loose fibers retained over 90% of its specific activity when reusing it up to 10 times, in comparison to free lipase (which cannot be reused), whereas lipase bound on paper samples, retained around 60% of its original specific activity (Figure 1). Furthermore, lipase immobilised on short chitosan nanofibers (in both forms) displayed significant thermal stability at high temperatures. Aside from thermal stability, the main advantage identified for lipase immobilised on SNFs is its easier separation, recovery and re-use.

The nature of short nanofibers is such that they can be treated as if they were particles, and fibers of different type, geometry, functionality can be mixed in suspension with great ease. This unique set of properties will allow to build multi-functional coatings or constructs, otherwise not accessible. This work also demonstrates that short chitosan nanofibers are a viable support for the immobilisation of enzymes (lipase acting as a model molecule), opening the way to novel constructs with high-application and tuning potential.

Figure 1. The process to prepare short nanofibers constructs, electron microscopy image of a construct (Scale bar: 1μm) and enzyme activity after re-use of a slurry of short nanofibers functionalized with lipase.

ACKNOWLEDGMENT This research was supported in part under the Australian Research Council’s Industrial Transformation Research Hub funding scheme (project number IH140100018). The present work was carried out with the support of the Deakin Advanced Characterisation Facility.
Flexible and Hierarchical Structured MnO$_2$ Nanoparticle-decorated SiO$_2$ Nanofibrous Membranes with High Catalytic Performance

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Construction of nanostructured MnO$_2$ functionalized ceramic nanofibrous membranes with good flexibility, a hierarchical mesoporous structure, high catalytic performance and excellent durability is of great significance to the remediation of dyeing wastewater; however, creating such materials has proven to be extremely challenging. Herein, we demonstrated an intriguing approach for preparing flexible, hierarchical porous, and nanostructured MnO$_2$ immobilized SiO$_2$ nanofibrous membranes (MnO$_2$@SiO$_2$ NFM) through the combination of electrospinning and hydrothermal synthesis. The morphologies of MnO$_2$@SiO$_2$ nanofibrous membranes could be regulated via changing the concentration of reactants, which would affect the specific surface area, porous structure, and mechanical properties of membranes. The resultant MnO$_2$@SiO$_2$ NFM exhibited prominent catalytic performance towards methylene blue (MB) after forming Fenton-like reagent by cooperating with hydrogen peroxide, including high degradation degree of 95%, fast degradation rate within 40 min, good reusability in 5 cycles. Furthermore, the membranes with good flexibility could directly be taken out from solution and be used for next cycle without any post-treatment. The successful fabricating of such membranes with extraordinary catalytic performance could provide a platform for the preparation of high-performance catalysts for the remediation of dyestuffs pollution or the pre-treatment of discharging wastewater.

Figure 1. (a) SEM image of MnO$_2$@SiO$_2$ NFM; (b) optical image showing the flexibility of MnO$_2$@SiO$_2$ NFM; (c) the degradation performance of various MnO$_2$@SiO$_2$ NFM towards MB at time intervals.
Designing Nanofiber Structures for Chemical and Biological Protection, Wound Dressings, and Laser Ultrasound Transducers by Centrifugal Spinning

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Nanofibers are an important class of material that is useful in a variety of applications, including filtration, tissue engineering, protective clothing, composites, battery separators, energy storage, etc. So far, electrospinning is the most studied method for producing nanofibers. A literature search using the Web of Science™ database shows that Year 2017 alone had publication of over 3000 articles in the electrospinning of nanofibers. Among these publications, over 50% focused on the investigation of the electrospinning process and the characterization of the resultant nanofibers, and the others mainly address the innovative use of electrospun nanofibers for various applications. However, the wide-spread commercial use of electrospinning is limited due to its low production rate, poor safety, and high cost. Most other nanofiber production methods, such as phase separation, template synthesis, and self-assembly, are complex and can only be used to make nanofibers from limited types of materials. This presentation introduces a simple, yet versatile technique for producing nanofibers of various materials including polymers, carbons, ceramics, metals, and composites. Centrifugal spinning eliminates the limitations encountered by current nanofiber production methods and can produce nanofibers at high speed and low cost. Centrifugally-spun nanofibers can be used in various applications, including filtration, tissue engineering, protective clothing, composites, battery separators, energy storage, etc. The applications of centrifugally-spun nanofibers in chemical and biological protection, wound dressings, and laser ultrasound transducers will be discussed in this presentation.
Development of Electrospun Composite Nanofibers for Proteins Separation

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In this study, composite electrospun nanofibers were developed for separation of a protein from a protein mixture. This was achieved by adding a percentage of poly(methyl vinyl ether-alt-maleic anhydride)(PMA) to polyvinyl alcohol (PVA), which resulted in composite nanofibers with combined properties of appropriate adsorbent groups (from PMA) and high mechanical properties (from PVA). These nanofibers have negative charge on their surface (from PMA) which can attract molecules with positive charges. The effects of various parameters such as buffer pH, PMA content, and protein concentration on the adsorption performance of the membranes were investigated. Lysozyme (LYZ) was used as the model protein and a high adsorption capacity of 476 mg/g was obtained at pH 6 owing to the electrostatic attraction between the negatively charged nanofibers and positively charged proteins. Such the high capacity was due to the combined effects of high specific surface area, highly porous structure, and abundant functional carboxyl groups of the nanofibers. By tailoring the pH, the negatively charged nanofibers selectively adsorbed positively charged proteins from a protein mixture. Also, the results showed that the membranes can be regenerated/reused with no significant effect on the capture capacity. The obtain results can provide a promising method for the purification of proteins in small analytic devices.

Figure 1. Left: Electrospun PVA/PMA nanofibers for proteins separation; Right: SDS-PAGE analysis for purification of LYZ solutions and LYZ/BSA mixture (Buffer pH=6).

ACKNOWLEDGMENT

The authors are grateful for the support provided by the USDA National Institute of Food and Agriculture, Hatch project NYS-3297402 and Hatch multistate project NC-1194 NYC-3297860.
Nanofiber scaffolds for tissue engineering became highly interdisciplinary research field which expands from fluid dynamic to stem cell research and finally to regenerative medicine technologies. The original hypothesis that nanofibers owing to their nanoscopic cross-sectional dimensions can mimic the structure of natural extracellular matrix for spatial guidance of cell proliferation has transformed into the understanding that this guidance is realized through multiple signaling via mechanical and biochemical cues in addition to spatial alignment. The current hypothesis suggests that live stem cells in biomimetic extracellular environment possess all mechanisms required for proper transformation and proliferation. However, these mechanisms should be turned on by proper signaling through mechanical and biochemical processes in the extracellular matrix. These signaling mechanisms are yet to be discovered amid substantial progress is continuously developing in the field.

In this work we systematically vary mechanical characteristics of polycaprolactone nanofibers while retain an unchanged fiber alignment, diameter and chemical composition. The nanofibers in a broad range of mechanical characteristics were fabricated using two nanofiber spinning methods: electro-spinning and touch-spinning. This allowed to extend the range of Young’s modulus of the nanofibers from 10 to 230 MPa and a fraction of crystallinity from 30 to 80% for the prepared samples of nanofiber scaffolds (Figure 1). The results are consistent with our hypothesis about different mechanisms of the fiber formation on the last stage of fiber “cold drawing” when the touch-spun fibers undergo a greater drawing ratio and thus approach a greater polymer coil stretching, alignment and crystallization than electro-spun nanofibers. In addition to the improved mechanical properties of the fibers, the touch-spun fibers demonstrate improved stability in the aqueous cell culture environment. The latter helps to avoid misalignment and entanglement of the nanofibers after a 2 week stem cell differentiation period. Our experiments demonstrated that the fibers with enhanced mechanical properties (touch-spun fibers) outperformed softer (electro-spun fibers) for scaffolding of neuron cell differentiation. It turned out that enhanced mechanical properties and stability of the fibers in the cell culture environment are beneficial for the preferential development of neural cells vs. non-neural cells.
Electrospun Polyacrylonitrile Nanofibers: An Antifungal Material

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INTRODUCTION
In recent years, integration of electrospun nanofibers in commercial products has been growing exponentially. Electrospun nanofibers have been found in a number of applications such as air/water filtration, performance apparel, drug delivery, scaffold for tissue engineering, and etc. The electrospun nanofibers are already exposed to environment through corresponding commercial products in the form of nanofibrous mat and inevitably in contact with microorganisms including fungi. However, little is known about biological interaction between electrospun nanofibrous mat and fungal cells. In this study, the interactions between electrospun polyacrylonitrile (ESPAN) nanofibrous mat and the baker’s yeast, Saccharomyces cerevisiae, a model genetic microorganism and a type of fungus that is central to the fermentation industry, was investigated.

RESULT
Culture progression of *S. cerevisiae* SK1 cells grown with ESPAN nanofibrous mat and control substrates (blank, PAN film, PAN microfibrous mat) using OD₆₀₀ measurement over the course of 8 hours was monitored. SK1 cells demonstrated a standard long growth pattern against time under the circumstances of enough nutrient in all the control cultures. Compared to those control cultures, the cell culture with ESPAN nanofibrous mat, however, showed significantly lower growth capacity from the very beginning, indicating immediate inhibition of *S. cerevisiae* growth upon contact with ESPAN nanofibrous mat. A cell number drop was even observed at the end of the 8-hour growth period.

![Graph](image)

Figure 1. OD₆₀₀ variation of *S. cerevisiae* cultures with different PAN substrates within 8-hour growth period.

CONCLUSION
We have discovered for the first time that compared to PAN film and PAN microfibrous mat, ESPAN nanofibrous mats adversely affect the growth, morphology, and viability of yeast cells without any aid from antifungal agents. The antifungal functionality of ESPAN nanofibrous mat provides exciting promise as a next-generation material that control fungal growth through contact.
Low-thermal Conductivity Carbon Fibers from Electrospinning

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INTRODUCTION
Polyacrylonitrile (PAN)-based carbon fibers are normally high-thermal conductivity material depending on their carbonization temperature and corresponding graphitic structure inside. However, there are a few instances particularly in aerospace industry where high-thermal conductivity of carbon is not desirable. Low-thermal conductivity carbon fiber is the material of choice as part of fuel tank composite material as well as ablative thermal protection material for rocket and reentry vehicles. In this research, low-thermal conductivity carbon material from electrospinning was investigated. The intrinsic product from electrospinning is a non-woven nanofibrous mat with micrometer scale inter-fiber pores. The idea is to introduce submicrometer and nanometer porous structure in individual electrospun carbon nanofiber (ECNF) by integrating PAN with poly(methyl methacrylate) (PMMA) and SiO₂ nanoparticles (SNPs) and couple it with micrometer scale inter-fiber porous structure of ECNF mat.

RESULTS
Table I. Average pore width, pore volume, BET surface area and corresponding thermal conductivity of the ECNF nanofibrous mat carbonized at 900°C.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Average Pore Width (nm)</th>
<th>Pore Volume (cm³/g)</th>
<th>BET Surface Area (m²/g)</th>
<th>Thermal Conductivity (W/m-K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon film</td>
<td>15.09</td>
<td>0.02</td>
<td>4.43</td>
<td>7.64 ± 0.33</td>
</tr>
<tr>
<td>ECNFs</td>
<td>16.11</td>
<td>0.03</td>
<td>10.65</td>
<td>2.48 ± 0.08</td>
</tr>
<tr>
<td>ECNFs from PAN/PMMA (70/30)</td>
<td>16.11</td>
<td>0.13</td>
<td>31.81</td>
<td>1.49 ± 0.14</td>
</tr>
<tr>
<td>ECNFs from PAN/PMMA (50/50)</td>
<td>20.66</td>
<td>0.16</td>
<td>33.90</td>
<td>1.28 ± 0.08</td>
</tr>
<tr>
<td>ECNFs with 5 wt.% SNPs</td>
<td>18.78</td>
<td>0.07</td>
<td>15.61</td>
<td>1.70 ± 0.19</td>
</tr>
<tr>
<td>ECNFs with 10 wt.% SNPs</td>
<td>18.88</td>
<td>0.13</td>
<td>39.18</td>
<td>1.25 ± 0.05</td>
</tr>
<tr>
<td>ECNFs with 5 wt.% SNPs from PAN/PMMA (70/30)</td>
<td>15.63</td>
<td>0.17</td>
<td>45.15</td>
<td>1.15 ± 0.08</td>
</tr>
<tr>
<td>ECNFs with 5 wt.% SNPs from PAN/PMMA (50/50)</td>
<td>18.58</td>
<td>0.18</td>
<td>49.02</td>
<td>0.87 ± 0.04</td>
</tr>
<tr>
<td>ECNFs with 10 wt.% SNPs from PAN/PMMA (70/30)</td>
<td>17.57</td>
<td>0.18</td>
<td>54.21</td>
<td>0.38 ± 0.02</td>
</tr>
<tr>
<td>ECNFs with 10 wt.% SNPs from electrospinning PAN/PMMA (50/50)</td>
<td>20.09</td>
<td>0.19</td>
<td>55.14</td>
<td>0.15 ± 0.02</td>
</tr>
</tbody>
</table>

CONCLUSION
A low-thermal conductivity carbon nanofibrous material was successfully prepared by integration of PMMA and SNPs into PAN spinning solution followed by consecutive electrospinning, stabilization and carbonization. The low-thermal conductivity of ECNF mat is attributed to low-carbonization temperature, homogeneous distribution of low-thermal conductivity SNPs in ECNFs, and more importantly a simultaneous micro-, submicro- and nano-porous structure, which is a result from micrometer scale inter-fiber pores from electrospinning, submicrometer scale pores from PAN/PMMA phase separation in electrospun nanofibers, and nanometer scale pores from SNP relocation during volume-shrinking carbonization. The porous carbon structures from microscale down to nanoscale in ECNF nanofibrous mats enabled an overall thermal conductivity reduction from 7.64 to 0.15 W/mK, a 98% reduction, with respect to the non-porous carbon film counterpart. This research demonstrated a novel way to reach low-thermal conductivity carbon fibrous materials for thermal insulation.
Flexible MOF-based Nanofibrous Membranes for Efficient CO₂ Capture with Long-term, Ultra-stable Recyclability

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In the global transition to a sustainable low-carbon economy, CO₂ capture and storage technology plays a critical role for deep emission reduction. Metal–Organic Frameworks (MOFs) are crystalline materials with ultra-high porosity, tunable pore size, and rich functionalities, holding the promise for CO₂ capture. However, the intrinsic fragility and depressed processability of MOF crystal particles make the fabrication of flexible MOF nanofibrous membranes (NFMs) rather challenging. Here, we report an effective strategy for the versatile fabrication of self-supported and flexible HKUST-1 NFM with ultra-high HKUST-1 load rate (up to 82 wt %), stable and uniform HKUST-1 growth through the combination of the electrospinning, multistep seeded growth, and activation process. Significantly, the HKUST-1 NFM exhibits prominent CO₂ adsorption capacity of ~4 mmol g⁻¹, good selective capture for CO₂ over N₂, and remarkable recyclability. The CO₂ capacity retains 95% of the initial value after 100 adsorption–desorption cycle tests, indicating that the resultant HKUST-1 NFM has long-term and ultra-stable recyclability, as well as significant practical value. Thus, this scalable and low-cost production pathway is able to convert MOF particles into self-supported and flexible NFMs and thereby they are better applied to efficient post-combustion CO₂ capture.

In the present work, we demonstrate an effective strategy for the versatile fabrication of self-supported and flexible HKUST-1 NFM with ultra-high HKUST-1 load rate (up to 82 wt %), stable and uniform HKUST-1 growth through the combination of the electrospinning, multistep seeded growth, and activation process. To the best of our knowledge, there are no reports on flexible HKUST-1 NFM for efficient CO₂ capture with long-term cycling stability up to now. This study aims at developing self-supported and flexible MOF NFM-based sorbents with ultra-high MOF load rate, stable and uniform MOF growth, which exhibits high specific surface area, suitable and tunable pore size, prominent CO₂ capacity, good CO₂/N₂ selectivity, as well as long-term and ultra-stable recyclability. Thus, our work has revealed, for the first time, the resultant HKUST-1 NFM can be potentially applied to the efficient post-combustion CO₂ capture with prominent CO₂ capacity and remarkable cycling stability.

As schematically shown in Figure 1, we designed the HKUST-1 NFM.

Figure 1. Schematic illustration of electrospun PAN/HK NFM used as skeleton to produce self-supported and flexible HKUST-1 NFM with ultra-high HKUST-1 load rate, stable and uniform HKUST-1 growth.
Moisture and Oily Molecules Stable Nanofibrous Electret Membranes for Effectively Capturing PM$_{2.5}$

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The fabrication of electret air filter with robust charge stability towards moisture and oily molecules is becoming increasingly essential due to the severe haze pollution, which is featured with high relative humidity (80-90%) and contains oily components (30-40%); however, this remains an ongoing challenge. Herein, the nanofibrous electret membrane enable to enduringly capture PM$_{2.5}$ containing moisture and oily components with high efficiency and low air resistance is successfully designed and fabricated. Benefiting from the high electrical resistivity of polyvinyl butyral (PVB) and high dielectric constant of silicon nitride nanoparticles (Si$_3$N$_4$ NPs), the surface potentials of PVB/Si$_3$N$_4$ nanofibrous membranes were increased from 1610 to 2010 V when the content of charge enhancer (Si$_3$N$_4$) increased from 0 to 1 wt%. More importantly, the charge dissipating mechanism under water and oil condition was revealed. Significantly, by further introducing hydro-oleophobic fluorinated polyurethane (FPU), the stable surface potentials of PVB/Si$_3$N$_4$-FPU membranes were elevated by 62.8% under the condition of high relative humidity (85%) and oily component content (48.4%), compared with PVB/Si$_3$N$_4$ membranes. The resultant membranes achieved a high filtration efficiency of 99.950%, low air resistance of 55.0 Pa, and robust durability, which could provide a promising method for the design of electret air filtration materials used in haze environment.

Figure 1. The PM$_{2.5}$ capture performance of PVB/Si$_3$N$_4$-FPU nanofibrous membranes for removing PM$_{2.5}$ from 500 to 35 μg cm$^{-3}$ during 30 cycles.
Intumescent Flame Retardant Bilayer Coatings of Casein and Ammonium Polyphosphate on Cotton Fabric

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ABSTRACT
The objective of present study was to investigate the effect of bilayer coating of different concentrations of ammonium polyphosphate (APP) and casein on thermo-oxidative properties, flame retardant behavior as well as thermal conductivity of char residues of cotton fabrics. Intumescent flame retardant coating, composed of cationic casein and anionic ammonium polyphosphate has been constructed on cotton fabric by bilayer coating. The flame retardant behavior was found to increase predominantly with increase of APP concentration than the casein concentration due to greater release of phosphoric acid and ammonia from APP. Whereas, the higher concentration of casein was found more advantageous to form the distinct layer of intumescent char formation, where cotton fabric samples coated with 15% casein and 7.5% APP contents produced maximum char of 44%.

INTRODUCTION
Cotton fiber is ideally used in apparels, draperies, tents, pillowcases, towels, bed sheets, etc because of its soft, comfortable, and breathable features. However, cotton fiber also poses many challenges due to its flammable and hydrophilic nature where heat and humidity combine to form fire conducive environments. Once cotton fabric is ignited, the flame spreads fast and is difficult to extinguish. The limiting oxygen index of cotton is only about 19 % and the combustion temperature is 360–425 °C.

The present paper described a simple strategy to build intumescent coatings on cotton fabric surfaces using environment friendly resources. The bilayer assemblies of positively charged casein were coupled with negatively charged APP, and deposited on cotton fabrics by traditional coating methods. The casein–APP pair can represent an intumescent-like system, in which casein can act as both carbon source and foaming agent, whereas APP is able to generate phosphoric acid at high temperatures.

MATERIALS AND METHOD
Materials
The plain woven 100 % cotton bleached fabric having 145 g/m² aerial density (Licolor, a.s. Czech Republic), bovine milk casein (Sigma-Aldrich, Czech Republic), Ammonium polyphosphate, APP (Aako Netherland), Sodium hydroxide and hydrochloric acid (Lach-Ner, Czech Republic) were used in this study.

Prepartion and application of casein and APP solutions on cotton fabric
Casein (5, 10, 15 wt. %) and APP (2.5, 5.0 and 7.5 wt %) suspensions of different concentrations were prepared in distilled water and then applied on cotton fabric through conventional coating method to deposit bilayer coating on the surface of cotton fabric. In this way, the total of nine samples was prepared (see Table I). The uptake (add on percentage) was also calculated.

Table I. Details of coated samples.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Casein Conc. (%)</th>
<th>APP Conc. (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAS5+APP2.5</td>
<td>5</td>
<td>2.5</td>
</tr>
<tr>
<td>CAS5+APP5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>CAS5+APP7.5</td>
<td>5</td>
<td>7.5</td>
</tr>
<tr>
<td>CAS10+APP2.5</td>
<td>10</td>
<td>2.5</td>
</tr>
<tr>
<td>CAS10+APP5</td>
<td>10</td>
<td>5</td>
</tr>
<tr>
<td>CAS10+APP7.5</td>
<td>10</td>
<td>7.5</td>
</tr>
<tr>
<td>CAS15+APP2.5</td>
<td>15</td>
<td>2.5</td>
</tr>
<tr>
<td>CAS15+APP5</td>
<td>15</td>
<td>5</td>
</tr>
<tr>
<td>CAS15+APP7.5</td>
<td>15</td>
<td>7.5</td>
</tr>
</tbody>
</table>
CHARACTERIZATION AND TESTING
Bilayer coated cotton fabrics were characterized for FTIR, SEM, phosphorous element contents analysis (ICP-AES), thermo-oxidative stability (TGA) and flame retardancy (horizontal flame tester) as well as the thermal conductivity of char residues (TCi), according to standard test methods.

RESULTS AND DISCUSSION
Data collected from characterization and testing of developed samples were analyzed and discussed to study and investigate reasons for the behavior of different functional properties.

Thermo-oxidative stability of casein-APP coated cotton fabrics
The thermal degradation of all samples consisted of two steps, 200–400 °C and 400–600 °C. The T_{ onset} values of cotton fabrics were found to shift towards the lower temperatures when coated with bilayer of casein and APP solutions. Figure 1 represent the weight loss with increase in temperature.

![Figure 1. TGA curves for different bilayer assemblies coated on fabrics.](image)

Flammability properties of casein-APP coated fabrics
The combustion behavior of the uncoated and the coated cotton fabrics was assessed by horizontal flame test to describe the real fire scene. The total burning time (s), burning rate (mm/s) and residue (%) were used to estimate the resistance for flame propagation (see Table II).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Total Burning Time (sec)</th>
<th>Flame Stoppage Time (sec)</th>
<th>Afterglow Time (sec)</th>
<th>Char residue (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>No treatment</td>
<td>36</td>
<td>-</td>
<td>15</td>
<td>3.1</td>
</tr>
<tr>
<td>CAS5+APP2.5</td>
<td>-</td>
<td>8</td>
<td>-</td>
<td>26.8</td>
</tr>
<tr>
<td>CAS5+APP5</td>
<td>-</td>
<td>7</td>
<td>-</td>
<td>32.5</td>
</tr>
<tr>
<td>CAS5+APP7.5</td>
<td>-</td>
<td>5</td>
<td>-</td>
<td>34.3</td>
</tr>
<tr>
<td>CAS10+APP2.5</td>
<td>-</td>
<td>6</td>
<td>-</td>
<td>28.9</td>
</tr>
<tr>
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<td>-</td>
<td>5</td>
<td>-</td>
<td>35.4</td>
</tr>
<tr>
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<td>-</td>
<td>3</td>
<td>-</td>
<td>37.2</td>
</tr>
<tr>
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<td>5</td>
<td>-</td>
<td>32.8</td>
</tr>
<tr>
<td>CAS15+APP5</td>
<td>-</td>
<td>4</td>
<td>-</td>
<td>40.1</td>
</tr>
<tr>
<td>CAS15+APP7.5</td>
<td>-</td>
<td>2</td>
<td>-</td>
<td>43.9</td>
</tr>
</tbody>
</table>

Table II. Burn characteristics of different bilayer assemblies coated on fabric.

![Figure 2. Thermal effusivity of char resides.](image)

![Figure 3. Thermal conductivity of char residues.](image)
In order to validate the formation of voluminous/intumescent char on surface of coated fabrics, the thermal conductivity of the residual chars were measured. From Figure 2 and Figure 3, it can be seen that thermal conductivity and thermal effusivity values of residual chars reduced with increase in APP or casein concentrations in bilayer assembly. Later, the flame retardant mechanism of bilayer assemblies of casein/APP contents was investigated by observation of the surface structure of burnt samples on scanning electron microscopy (see Figure 4). These results indicated formation of additional char residue by catalytic dehydration of casein in the presence of APP, and thus potential use of casein as carbon source in intumescent systems.

(a) CAS5+APP2.5  (b) CAS5+APP5  (c) CAS5+APP7.5
(d) CAS10+APP2.5  (e) CAS10+APP5  (f) CAS10+APP7.5
(g) CAS15+APP2.5  (h) CAS15+APP5  (i) CAS15+APP7.5

Figure 4. SEM images of char residues of different bilayer assemblies.

CONCLUSION
In present work, the environment friendly intumescent system of casein and APP was applied as bilayers on cotton fabrics by simple and industrially scalable technique. The effects of different concentrations of casein and APP in bilayer assembly were studied for thermo-oxidative stability and flame resistance behavior of cotton fabrics. From TGA analysis, higher concentration of APP depicted stronger sensitization of the cellulose decomposition as compared to casein. However, the higher concentration of casein was found to produce higher char residue irrespective of the APP concentration. From horizontal flame tests, cotton fabrics coated with 15% casein and 7.5% APP contents showed maximum char formation of 44% as compared to 3% char for uncoated cotton fabrics. The images of burnt samples confirmed the formation of distinct intumescence layer of char residue at higher concentrations of casein and APP, and it was also validated from the measurements of thermal conductivity of char residues. The SEM images of residues of burnt samples showed the formation of thin protective char layer on the surface of fibers at lower casein content. Interestingly, globular micrometric structures at localized spaces were observed for the lower APP contents, and more expanded globular micrometric structure at enlarged spaces were observed for higher APP contents. In this way, the present work realized that the casein could perform number of functions such as catalyzed dehydration, accelerated charring and intumescent char formation in APP based intumescent systems.

ACKNOWLEDGMENT
The financial support for conducting the present research work was provided from the Student Grant Competition Technical University of Liberec no. 21241 granted by Ministry of Education, Youth and Sports of Czech Republic.
Dyeing and Finishing
Cotton Recycling for Making New Functional Fiber

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INTRODUCTION
For today’s textile and fashion industry, sustainability has been more vital than ever. Life cycle assessment (LCA) for textile and apparel products out of a whole manufacturing chain needs to be implemented more seriously in terms of carbon footprint and environment impact. A big challenge is textile/apparel recycling. This results from two major barriers. One is that currently there is no mandate for textile waste management, allowing textile recycle continuously to be a voluntary practice. The other is unavailability of a municipal textile waste management system because of complex textile contents, making textile recycling economically infeasible. Consequently, fewer industrial end users can use recycled textiles to manufacture new fiber, fabric, apparel, and other consumer or industrial products.

This research addresses an approach for recycling cotton textile and apparel by use of recycled cotton to produce new functional rayon fiber for renewable textile and apparel manufacture. As the purest natural cellulose in nature, cotton fiber is the king of natural fibers widely used in today’s apparel and fashion manufacture. Because cotton is a high input and high value commodity crop, promotion of cotton recycling and utilization of recycled cotton for sustainable fashion and other material product development would be highly helpful for lowering cotton carbon footprint and enhancing sustainability of cotton production and uses.

METHOD
Recycled blue jean fabric was shredded into strip pieces and was immersed into a chlorine-based bleach for leaching. After rinsing and drying, the bleached blue jean fabric was ground into powder for alkaline treatment. The formed cotton fiber slurry was filtered and thoroughly washed with distilled water until reaching a neutral pH. The obtained cotton cellulose powder was dissolved in ionic liquid solvent 1-butyl-3-methylimidazolium chloride [BMIM]Cl using a mixer with controlled temperature, pressure, and shearing rate. Regenerated cellulose nano-fiber (RCNF) nonwoven was fabricated using an electrospinning method, followed by a room-temperature drying.

RESULTS AND CONCLUSION
RCNF from recycled blue jeans is a soft and porous membrane material ideal for medical and personal care applications in many ways as a biobased and biocompatible biomaterial. RCNF cytotoxicity was tested using an HL-1 cardiac muscle cell procedure. The assay result indicated that the recycled-cotton RCNF performed a 18% increase of the cell viability with reference to the cell-only sample, while tested control sample (wood-pulp RCNF) experienced a 22.7% reduction of cell viability.

RCNF is also a good cellulose-surface support material for enzyme immobilization that can functionalize the cellulose-based nonwoven web for bioactivity or protein modification. By embedding carboxylated carbon nanotubes (CNT-COOH) and graphene oxide (GO), the recycled cotton RCNF surface was activated to react with the nucleophilicamino functionality at the side chains of amino acids in lysozyme. As a result, the cotton cellulose RCNF was modified to form a polysaccharide-protein structure by covalently binding lysozyme. Bioassay for lysozyme activity was conducted using EnzChek Lysozyme Assay Kit. The test exhibited that the amount of attached lysozyme and bioactivity were significantly detected. It was also observed that the amount of immobilized lysozyme and bioactivity was proportional to CNT-COOH or GO loading rates onto RCNF.
Functional chemical modifications on poly(ethylene terephthalate) (PET) fibers could be successfully implemented and controlled by managing mutual interactions and affinities between different components in a radical graft polymerization system. The interactions and affinities of different components in the reaction system could be estimated by using Hansen Solubility Parameters (HSP), and proper management of the interactions (HSP distances ($R_s$), Scheme 1) by selecting monomers (M), initiators (I), an additional solvent (S), and a crosslinker (CL) with PET could provide reliable control to grafting yields of hydrophilic and hydrophobic monomers. The latest results proved the practicality of using the HSP in controlling radical graft functional monomers on surfaces of PET fibers. Six different monomers with different hydrophilic properties in different solvent and initiator systems were examined, and results confirmed that interactions of initiator-PET, initiator-solvent, monomer-PET, monomer-solvent and monomer-initiator play important roles in determining the grafting reaction efficiency. Results revealed that both hydrophobic and hydrophilic monomers could be effectively grafted onto surfaces of PET fibers as long as proper interactions are controlled based on the HSP analysis. The results have significant impacts on production of functionalized synthetic fibers through either fiber extrusion or fabric finishing processes.
Dyeing cotton to achieve ultra-deep black shades using reactive dyes has been a major ecological concern for textile industries, due to the disposal of high amounts of electrolytes and alkali as effluent from the dye house resulting environmental pollution. To reduce environmental pollution and make the process sustainable, cotton was cationized by graft polymerization using a quaternary ammonium monomer, diallyl-dimethyl-ammonium chloride (DADMAC) into cotton using a thermal initiator, potassium persulfate (KPS) and a crosslinking agent, N,N’-methylenebis(acrylamide) (MBA). Graft polymerization of DADMAC into cotton generates a large number of positively charged dye sites into the fiber, resulting almost 100% dye exhaustion, which paves the way for recycling water from the dye bath and ultra-deep black dyed cotton with good color fastness properties due to the ionic bonding between the dye and the cationized fiber. Moreover, cationization of cotton eliminates the need for electrolytes and/or alkali in the dyebath which significantly reduces environmental pollution. Additionally, DADMAC is not susceptible to alkaline hydrolysis and generation of epoxy moieties like other known quaternary ammonium salts, such as 3-chloro-2-hydroxypropyltrimethylammonium chloride (CHPTAC). In the present study, mercerized cotton treated with DADMAC and CHPTAC were dyed using a commercial reactive black dye (Avitera Black SE) with and without alkali at 10:1 liquor ratio and compared to untreated cotton dyed using traditional dyeing method with salt (anhydrous Na₂SO₄) using the same reactive dye, alkali and liquor ratio. It was shown that DADMAC-treated cotton achieved better color yield than untreated and CHPTAC treated cotton when dyed with alkali. If no alkali was used, the color yield of DADMAC treated cotton was comparable to untreated cotton while the color yield of CHPTAC treated cotton was significantly lower. Dye exhaustion for DADMAC and CHPTAC treated cotton were more than 95% when no alkali was used and 99% with alkali. Moreover, color fastness properties of DADMAC were comparable to CHPTAC treated cotton and untreated cotton dyed using conventional dyeing method.
Optimization of Process Parameters to Control Yarn Packing Density of Cotton Spun Yarn by Using Multiple Regression Models

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Ring spinning frame is most commonly and widely used technique for the production of yarn. This yarn subsequently utilized for the production of fabrics. Recently the research studies are more emphasis to create the living comfort condition. The yarn package density is vital role to determine the feel, comfort, bulkiness, moisture management and dyeing characteristics of yarn. The yarn packaging density is largely influenced by the yarn manufacturing techniques and raw material used. The effects of spinning process parameters on packing density could be helpful to predetermine the yarn comfort behavior prior to the fabric stage. The mechanical properties of the staple yarn are not only depending on the fiber characteristics but also on the yarn structure i.e. the arrangement of the individual fiber on the cross section of the yarn. The fiber distribution on the cross section of the yarn and packing density of yarn has been investigated. The study provide the how the spinning process parameters affect the internal structure of ring spun yarn. The aim of this study is to ameliorate the package density of the to achieve dynamometric and comfort properties of yarn to attract wide variety of customers. Yarn packing density represents inter-fiber distance and friction, hence controlling the mechanical and comfort properties of the yarn; the main object of this study is to control the packing density of yarn during spinning process.

In this research study the 100 percent cotton used as a raw material and spun on ring spinning machine. The 135 ring yarns samples have been produced from three different types of yarn counts include (Ne 8, 16 and 24) with 9 different samples of each count were produced. Variables within a count were TPI (twist per inch) and spindle speed. Yarn packing density was calculated using the formula and by using the multiple regression models for optimization of process parameters which effect the yarn package density. The results indicate that the increase in yarn count, TPI and spindle speed increase the yarn packing density.

**KEYWORDS:** Count, IPI, packing density, process parameters, regression.
Citric acid (CA)-crosslinked cotton fabrics may show yellowish appearance which restricted the practical application of CA in formaldehyde-free anti-wrinkle finishing process. In this presentation, H₂O₂ bleaching systems under alkaline condition and UV irradiation were used, optimized and compared on CA-crosslinked cotton fabrics to recover the whiteness of the fabrics. It was found that both systems can provide good bleaching properties, whereas the bleaching under the alkaline condition showed damages to the anti-wrinkle property of the fabrics through hydrolysis reaction. Nevertheless, the UV irradiation of H₂O₂ under a neutral pH can minimize the crosslinking hydrolysis, and a longer wavelength of UV such as 312 nm performed better than using regular 254 nm UV light by showing less tensile strength loss of the fabrics. The reaction mechanism and potential product of the oxidation reaction on CA-crosslinked cotton were proposed and demonstrated through UV-vis spectrophotometer and Fourier transform infrared spectroscopy. Whiteness, wrinkle recovery angle and tensile strength of the fabrics were evaluated, and the results support the mechanism. More importantly, the UVB/H₂O₂ bleaching system was also applied to be a promising bleaching treatment for raw cotton fabrics with less energy and resources consumption.
Biomedical Applications
Heart Valve: Functionalization and Optimization of the Material

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INTRODUCTION
The recent solution to replace failing cardiac valves is called transcatheter aortic valve replacement (TAVR). Today, some of the implanted valves are made of biological tissues, which their durability is limited. As an alternative approach, valves made of fibrous constructions such as polyethylene terephthalate (PET) have recently proven durability over a 6 months period in sheep models. Exaggerated fibrotic tissue formation remains, however, a critical issue. Thanks to its hydrophilic and inert properties, poly(ethylene glycol) (PEG) is used to add a steric barrier to a surface, which helps minimizing protein binding and hence, cell attachment. This work investigates how to limit fibrosis through surface functionalization of PET with PEG.

MATERIALS AND METHODS
PET valves were treated using atmospheric pressure plasma (AlmaPlus, AlmaPlasma) operated at 18kV. The treatments were conducted in jet mode (4L/min of helium flow) in order to create carboxylic acid (-COOH) groups on the surface of the material. These carboxylic groups were activated with EDC/NHS, then covalently grafted with PEG-NH2 (Mw =1000g/mol). Each step was assessed using dynamic contact angle and X-ray photoelectron spectroscopy analyses.

The as-modified prostheses were implanted on juvenile sheeps for several months. After explantation, they are characterized to assess the fibrosis state of the valves. Histology, quantification of the calcium deposits. X-ray Fluorescence/Diffraction, Differential Scanning Calorimetry, Scanning Electron Microscopy and Energy-dispersive X-ray spectroscopy were conducted.

RESULTS AND DISCUSSION
The efficiency of plasma modification was evidenced by an increase of the characteristic carboxyl bands at 289 eV in high resolution C1s XPS spectra compared to the untreated PET: from 16% to 25%. In addition, the grafting of PEG-NH2 was confirmed by the presence of N1sXPS feature (3.2%) and by dynamic contact angle tests (droplet absorption time decreased from 9.3s to 1.6s).

The explanted valves exhibited less fibrosis but a higher rigidity that probably led to the animals’ death. This could be explained by the formation of deposits between the fibers (Figure1), which could be correlated to calcium nucleation (TableI) and led to limitation in fiber mobility and a less flexibility during the heart contractions.

![Figure 1. Explanted valve showing calcium deposit between fibers.](image)

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
<th>Atomic %</th>
<th>Error %</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>20.10</td>
<td>33.02</td>
<td>11.24</td>
</tr>
<tr>
<td>N</td>
<td>5.75</td>
<td>8.11</td>
<td>22.49</td>
</tr>
<tr>
<td>O</td>
<td>30.16</td>
<td>37.20</td>
<td>11.21</td>
</tr>
<tr>
<td>Na</td>
<td>1.85</td>
<td>1.59</td>
<td>14.05</td>
</tr>
<tr>
<td>Mg</td>
<td>0.58</td>
<td>0.47</td>
<td>18.08</td>
</tr>
<tr>
<td>P</td>
<td>11.72</td>
<td>7.47</td>
<td>3.65</td>
</tr>
<tr>
<td>Ca K</td>
<td>22.08</td>
<td>10.87</td>
<td>2.39</td>
</tr>
</tbody>
</table>

Table 1. Chemical composition of the deposits.

CONCLUSION
According to these results, the substrate functionalization limits fibrosis, but compromises textile flexibility.
Bioinspired Design of Open Fiber-based Microfluidics

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Insects have developed unique fiber-based mouthparts allowing them to exploit diverse foods, such as floral nectar, fruit, sap, blood, and sweat. The most attractive engineering feature of these fibers is that the natural fibers are self-cleaning. Natural fiber-based feeding devices with open channels are attractive for a variety of engineering applications. Currently, microchannels in fluidic devices are limited by rectangular and V-shapes. Sharp edges impose restrictions on wetting properties of these channels limiting their applicability to transport a wide range of fluids. These designs significantly narrow the range of polymeric materials appropriate for microfluidic applications. A search for an alternative design of open fluidic channels has been initiated in response to progress in development of fiber-spinning technology; this technology offers new opportunities to directly melt-extrude inexpensive open channel microfluidics. In these lecture, experimental and theoretical studies of wetting of the complexly-shaped natural and man-made fibers will be discussed.

ACKNOWLEDGMENT

This work was supported in part by National Science Foundation through the SC EPSCoR/IDeA Program under NSF Award no. OIA-1655740.
Smart Textiles for Medical Healthcare and Rehabilitation

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In this twenty-first centuries, the human race has witnessed a significant advancement in the field of healthcare and rehabilitations. With the advancement of science and technology, today the care providers are employing state-of-art products, able to streamline processes and offer personalized care with more efficiency. Textiles are also playing a vital role in these revolutions, offering a next-generation cutting edge technologies for several medical related products. This presentation will discuss on the recent research and developments in the field of compression textiles for healthcare and rehabilitation. In this research, the potential of memory textiles will be introduced. This novel function of memory effect allows programming and storing the temporary shape in the structure of textile, and also enabling it to retrieve the original shape reversibly with an external stimulus such as heat. The paper addresses the fundamentals of memory behavior and the related mechanism using a viscoelastic model consists of spring and dashpot elements. As an example of the potential application, the memory control in textile can be used for smart medical compression treatment to allow pressure control with massaging effect for an efficient treatment of compression therapy used in chronic venous disorders. It is anticipated that the research into memory control in textiles will grow in multiple dimensions as a result of their promising potential in many stress controlling applications such as pressure garments, bandages, massage devices, 3D mattress, sensors/actuators, etc.

REFERENCES

ACKNOWLEDGMENT
The author acknowledges the financial support received from the Department of Science & Technology (DST), India (Project Codes: MI01567; RP03454G).
Medical Textiles: How PET Fibers Topography Affects Wettability

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Textile material has been successfully used over the last decades in various medical applications like suture yarn, vascular graft, stent graft and hernia mesh. More recently, it has been shown that textile material could be considered to replace heart valves. In that latest application in particular, the foreign body reaction (FBR), once the textile is implanted *in vivo*, becomes a critical issue. Actually, FBR consists in fibrotic tissue ingrowth, which tends to stiffen the initially porous material, impeding the valve leaflets movement. However, the phenomenon is limited if the textile surface is hydrophilic. This latter property depends on the polymer used but also on the textile topography. The goal of the present work was to investigate how the yarn structure, topography as well as the textile construction can affect the wettability of a textile surface.

**METHODS**

In order to study the effect of the yarn structure on the wettability properties, 3 PET yarns characterized by same 90 microns equivalent diameter were considered: (1) monofilament, (2) multifilament, (3) staple yarn. A yarn surface was obtained by winding each yarn around an oblong support in a way to get contiguous spires and limit gaps between threads. A specific testing apparatus was developed to measure the contact angle of a drop of water placed under pressure on the tested surface. The goal was to reproduce the physiological blood pressure conditions. In a second approach, woven constructions were obtained from the same yarns in order to investigate the effect of the yarns arrangement on the wettability. At last, the roughness of the yarns integrated in the woven surface was modified and contact angle was measured.

**RESULTS**

Results bring out differences among the yarns which were used. In particular it could be pointed out that differences depend on the pressure applied to the drop (Figure 1). Considering mono and multifilament, it appears that multifilament is slightly more hydrophobic than monofilament when no pressure is applied. Conversely, under pressure, multifilament yarn becomes more hydrophilic as can be seen in the figure (Figure 2). Actually, under pressure the multifilament structure undergoes local rearrangement. Similar observations could be made at fabric level.

**CONCLUSION**

These preliminary results confirm that it is possible to adjust the wettability of a fabric surface by setting the topography at yarn and fabric level. Fibrous constructions can thus be optimized in order to limit fibrotic tissue in growth.

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**Figure 1:** Drop of water on multifilament before and after pressure is applied.

**Figure 2:** Contact angle as a function of pressure and filament type.
Construction of N-halamines/Metal Oxides Hybrid Nanoparticles and Application in Antibacterial Textiles

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Microbe related issues and biofilm formation have been great challenges in healthcare, medical devices, and household products. A series of N-halamine precursors including cyclic N-halamine siloxane precursors and vinyl-based N-halamine precursor monomers were synthesized, followed by covalently bonded onto TiO$_2$ and ZnO via sol-gel process, ultrasonic-refluxing method, atom transfer radical polymerization, and miniemulsion polymerization. The synthesized nanoparticles can be applied onto different substrates for producing multifunctionalized materials. SEM, TEM, XPS, FTIR, and TGA-DTG were used to characterize the prepared nanoparticles and materials. Antibacterial efficacies were evaluated against *E. coli* O157:H7 (Gram-negative bacteria) and *S. aureus* (Gram-positive bacteria). Biofilm-controlling ability of the prepared materials was investigated against selected model microbe *S. epidermidis* (Gram-positive), *E. coli* (Gram-negative), and *C. albicans* (Fungi) using cotton fabric as biofilm formation substrate. The biocompatibility of the prepared samples was assessed by *In vitro* cell cytocompatibility test. In addition, photocatalytic capability and UV light stability were also investigated after exposing to UV light for variable time. The storage stability and regenerable stability were also gauged. The results show that these N-halamine antimicrobial coated samples have great biocidal and biofilm-controlling efficacy, cytocompatibility, UV stability, and regenerability. The obtained data strongly encourage the application of these composites in textiles, wounds bandages, and healthcare facilities.
Classification of Compression Bandages

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INTRODUCTION
Compression bandages are well known and widely used for the treatment of venous disorders and lymphoedema. There are today classified two main categories of bandage which differ from each other by the mean of the Static Stiffness Index (SSI). SSI is the pressure difference observe on the leg, at point B1 between lying and standing position. This evaluation can only made on people and is quite inconsistent.

Compression bandages can also be classified by their maximum elongation (short / long stretch) or by their mechanical behaviors (elastic / non elastic). These three different classifications remain unclear for the medical staff.

METHOD
6 different bandages (Figure 1), representative of the market, have been tested against norm NF-S 97-115 (Préciser le type de dynamometer et les conditions de test).

RESULTS
Both Rosidal ® K and Somos® are plain weave fabrics, made of 100% cotton. Therefore, as measured on Figure 1, they both exhibit a non-elastic behavior. Biflex ® bandages are all fabrics with both leno weave and elastic covered yarn in warp direction. As measured they all have elastic behaviors. Results also show that stiffness is a lot higher for non-elastic bandages that for elastic bandages. The higher the stiffness is the more the pressure loss after 1hour will be.

<table>
<thead>
<tr>
<th>Bandage / Manufacturer</th>
<th>Elastic / non elastic (El / N El)</th>
<th>Maximum elongation (in %)</th>
<th>Stiffness (in N/m/mm)</th>
<th>Strength under the application elongation (in cN/cm)</th>
<th>Strength under the application elongation after 1 h (in %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rosidal ® K / L&amp;R</td>
<td>N El / SS</td>
<td>87,0</td>
<td>22,89</td>
<td>109,5</td>
<td>-52,4</td>
</tr>
<tr>
<td>Somos® / BSN</td>
<td>N El / SS</td>
<td>75,6</td>
<td>11,68</td>
<td>111,7</td>
<td>-49,0</td>
</tr>
<tr>
<td>Biflex ® 16 / Thuasne</td>
<td>El / LS</td>
<td>130,3</td>
<td>1,54</td>
<td>57,0</td>
<td>-7,9</td>
</tr>
<tr>
<td>Biflex ® 17 / Thuasne</td>
<td>El / LS</td>
<td>140,0</td>
<td>1,61</td>
<td>96,7</td>
<td>-14,8</td>
</tr>
<tr>
<td>Biflex ® 16 – SS / Thuasne</td>
<td>N El / LS</td>
<td>88,2</td>
<td>1,53</td>
<td>52,6</td>
<td>-11,2</td>
</tr>
<tr>
<td>Biflex ® 17 – SS / Thuasne</td>
<td>N El / LS</td>
<td>91,4</td>
<td>2,13</td>
<td>101,3</td>
<td>-10,6</td>
</tr>
</tbody>
</table>

Figure 1. Compression bandages tested: description and main characteristics.

DISCUSSION
Maximum elongation and elastic / non elastic behavior are two different parameters. It is indeed possible to have short stretch bandages with elastic behavior. Non elastic compression bandages have a higher stiffness than elastic bandages. The consequence is that the pressure loss (which is linked to the strength of the bandage when applied) is quickly decreasing over time. To be able to keep the pressure as constant as possible it will need frequent reapplication of the medical staff.

CONCLUSION
Testing method describe in norm NF S97-115 in a quick and reliable to characterize compression bandages either through maximum elongation or through elastic / non-elastic behavior.
Advanced Smart Medical Compression Stocking
Augmented with Memory Textiles

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Compression stockings/bandages have been using for the conservative treatment of chronic venous disorders such as venous ulcers and varicose veins over many decades. Gravity is the main reason for occurring these symptoms in the lower extremity of the human body and needs an adequate level of treatment. It has been a great challenge for both health practitioners and manufacturers to maintain/control the desired level of interfacial pressure. Different class of stockings are needed for various levels of compression and it depends on various leg attributes. Pressure loss over time is a major issue in the stockings due to stress-relaxation in the textile fibers and time-dependent system behaviors. There is an imperious need of any scientific approach for solving the major practical issues. In line with this, an unprecedented approach has been made for designing the smart compression stocking by addressing major key issues and augmented with memory textile structural engineering. A novel phenomenon of stress-memory behavior has been discovered in a thermal responsive semi-crystalline memory polymer. Where, stress in the material can be programmed, stored, and retrieved reversibly upon a cyclic external heat stimulus. The memory polymer was synthesized, optimized, and processed into a continuous strand of filaments and integrated into textile single jersey ground structure by inlay method to keep them as a main load bearing element. The freely standing load bearing element helps to control the internal stress/pressure upon external stimulus. Six different knit structures were employed and tested to get an optimized structure. For the first time, it is shown that, the internal stress/pressure and massage effect in the stockings can be modulated by structural modifications and other physical parameters. The effect of massage function on blood flow velocity in the popliteal vein on lower limb was objectively measured by Doppler ultrasound scanning. This study also sheds the insight of stocking structural modification for pressure control and provides the benchmark for achieving the efficient compression. This advanced stress-memory polymeric filaments based multifunctional compression stocking provides static pressure, massage effect, and easy size fitting in a more controlled manner for smart compression therapy.

REFERENCES

ACKNOWLEDGMENT
The authors would like to thank the funding support from General Research Fund for the project “Fundamental Study of Stress Memory and its Application in Smart Compression Stockings using Memory Polymer Filaments” (Project #: 15204416), and Fang Brothers Research Student Scholarship from the HK Polytechnic University, Hong Kong.
Highly Sensitive and Selective Biosensors Based on Fiber Organic Electrochemical Transistors

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Organic electrochemical transistors attract great attention for biological sensing owning to amplified function and biological compatibility. However, the planar structure don't satisfied the portable and wearable requirements. Fiber organic electrochemical transistors are easily integrate into fabrics and show excellent flexibility. In addition, it shows higher specific-volume ratio to increase sensitivity. In this study, the carbon material and conducting polymer composites have been prepared, and the fiber electrochemical transistors based on the composites have been assembled and their electrical property has been characterized, the devices fabricated with different gate electrode have been compared, it shows the platinum gate electrode supply good stability than gold or fiber electrode. The biosensor based on fiber electrochemical transistors has been investigated. Such novel fiber electrochemical sensor exhibits superiority as high current response, low detection limit and good reproducibility, the biosensor can also be selectively detected in the presence of interferences. Moreover, the device presents the accurate analysis in real samples, which provides a promising potential utility in the healthcare and biological application.

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ACKNOWLEDGMENT
This work was financially supported by the National Natural Science Foundation of China (51503157, 51473129).
Biobased Fibers and Texile Chemicals
Artificial Intelligence on Fashion and Textile: The Past and the Future

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Today, utilizing image or pictures for decision making has been popular and crucial in whatever our daily life and work place while fundamental research on artificial intelligence (AI) techniques has been a hot topic brought by the rapid technological advancement of computing technology in a recent decade. This talk will present about the overview of development and application of AI in different fashion and textile sectors, namely, fashion design, textile manufacture, apparel manufacture, fashion marketing and retailing. Challenges, opportunities and research trend of implementing AI in fashion and textile industry, in particular, two projects about how AI can be utilized in fashion retailing and textile manufacturing will be discussed.

AI AND FASHION RETAILING
Current fashion image searching technology used on online platforms is based on the whole fashion image to search the exact or other similar images. However if a customer is interested in some particular fashion attributes of a fashion image and wants to search other fashion items with these attributes, the current searching technology cannot meet the needs of the customer. This greatly limits the potential development and applications for offering more customized shopping experience. From artificial intelligence (AI) research perspective, this limitation of the current image searching technology is caused by the absence of available fashion image dataset constructed with both fashion professional knowledge and fulfils the requirement of deep learning, i.e. the current technology is unable to train a machine to accurately understand and recognize the fashion attributes of each fashion image. Fostering the application of AI in the fashion industry, a “FashionAI Dataset” is co-developed by Institute of Textiles and Clothing, The Hong Kong Polytechnic University and Alibaba Group to solve two fundamental problems of the deep learning algorithm: “apparel key points detection” and “fine-grained attribute recognition.”

AI AND TEXTILE MANUFACTURING
Fashion retailers do not expect to find holes, broken yarns, slubs, etc. on apparel, as substandard merchandise greatly influences the brand’s reputation and business success. Customer purchased the fashion merchandise with defects will cause a bad experience to the fashion brand. Along the fashion value chain, various checkpoints are established to evaluate the quality of textile materials in which fabric inspection is a vital step to ensure the quality of fabric and identify defects before making up a quality apparel product. Although there are a few automatic fabric inspection systems on the market, they can only be used to inspect fabric with a simple weave and colour, such as greige fabric and white colour. As apparel is always made from fabrics with different weaving structures and colours, these automatic fabric inspection systems definitely cannot fulfill the current industry needs, and thus almost all textile mills and apparel manufacturing enterprises still rely on manual visual inspection methods. However, labours are subject to fatigue and boredom, and thus inspection results are often unreliable and inconsistent. On the basis of AI technology, an intelligent fabric defect detection system named “WiseEye” which is integrated with the weaving machine can automatically locate and measure defects on most types of woven fabrics with different weaving structures and solid colours on a real-time basis.
A non-acid based finishing agent, neopentyl glycol diglycidyl ether (NPGDGE), was used to modify cotton fabrics. Direct characterization of the modified cotton fabric was conducted by nuclear magnetic resonance (NMR) without grinding the fabric into powder. NaOH and MgBr2 were compared in catalyzing the reaction between this di-functional epoxide and the hydroxyl groups of cellulose. Possible reaction routes were discussed. Scanning electron microscopy (SEM) images showed that while the MgBr2-catalyzed reaction resulted in self-polymerization of NPGDGE, the NaOH-catalyzed reaction did not. Fourier transform infrared spectroscopy (FTIR) showed that at high NaOH concentration cellulose restructures from allomorph I to II. Solution-state and solid-state 13C Nuclear Magnetic Resonance (NMR) studies verified that NPGDGE was incorporated into cotton fabrics and that higher NaOH concentration increased the efficiency of NPGDGE grafting. The cotton fabrics did not lose tensile strength after treatment with NPGDGE.

Figure 1. Packing of cotton fabric in a rotor for SSNMR study.

REFERENCE
Zirconium Hydroxide-based Sorptive and Reactive Textiles

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ABSTRACT
Protective textiles rely heavily on sorption to protect against chemical warfare agents (CWAs). Textiles with carbon-based sorptive substrates are currently used, but have a high thermal burden for the wearer. New materials are needed that offer the same or better protection with a lower thermal burden. Zirconium hydroxide is a good candidate for incorporation into protective textiles, as it is both sorbent and catalytic against multiple CWAs. This work evaluated zirconium hydroxide-based sorptive substrates against HD, G-series, and VX simulants. Protective properties were evaluated for substrates both in powder form and once incorporated into a textile. Zirconium hydroxide powders of varying sizes were laminated onto textiles at different loading densities. Transport, permeation, sorption, and catalytic properties were evaluated for the zirconium-hydroxide based sorptive textiles and compared to a carbon-based sorptive textile. Sorptive performance of zirconium hydroxide-based powders and textiles were comparable to the carbon-based sorptive. Additionally, catalytic activity by zirconium hydroxide was observed against multiple CWAs in both dry and humidified environments. Transport properties varied to an extent with zirconium hydroxide particle size, but was largely influenced by choice of textile. Overall, zirconium hydroxide-based sorptive textiles were comparable to currently used carbon-based sorptive textiles, with the addition of catalytic activity. Zirconium hydroxide shows great promise in the use of protective textiles against CWAs.

ACKNOWLEDGMENT
We would like to thank the Defense Threat Reduction Agency (DTRA) for funding the work described in the abstract.
Arginine-NIPAAm Hybrid Hydrogel with Biocompatible and Antibacterial Properties for Wound Dressing Application

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Despite the zwitterionic characteristic and biocompatible property of arginine based hydrogels, the brittle behavior and non-transparency still remain as a significant problem for wound dressing. In order to improve the mentioned problems and endow more properties for wound healing, a multi-functional hybrid hydrogel P(M-Arg/NIPAAm) with temperature response, anti-protein adsorption and antibacterial properties was prepared. NIPAAm improves transparency and mechanical property as well as acts as a temperature-response drug release system. The hydrogel was carried out by free radical copolymerization of methacrylate arginine (M-Arg) and N-isopropyl acrylamide (NIPAAm) monomers using N, N’-methylene bisacrylamide as a crosslinker, and ammonium persulfate/N, N, N’, N’-tetramethylethylenediamine as the redox initiator. To endow the antimicrobial property, chlorhexidine diacetate (CHX) was preloaded into the hydrogel and polyhexamethylene guanidine phosphate (PHMG) was grafted on the hydrogel surface, respectively, which make the hydrogel useful as a favorable antibacterial dressing. The antimicrobial property of two series of hydrogels was evaluated and compared. The successful synthesis of M-Arg, PHMG and hydrogels was proved by 13C NMR, 1H NMR and FTIR spectroscopy. The hydrogel morphology characterized by scanning electron microscopy confirmed that the homogeneous porous and interconnected structures of the hydrogels (Figure 1a). The swelling (Figure 1b), protein adsorption property (Figure 1c), in vitro release of CHX, antimicrobial assessment, cell viability as well as in vivo wound healing in a mouse model were studied (Figure 1d). The hybrid hydrogel has a combination effect of biocompatibility, environmentally responsive transformation behavior, biodegradability, anti-protein adsorption and antimicrobial properties. The in vivo results showed the nontoxicity and antimicrobial P(M-Arg/NIPAAm) hydrogel accelerated the full-thickness wound healing process and had the potential application in wound dressing.

Figure 1. (a) SEM images of the hydrogels before degradation: (A) PNIPAAm; (B) P(M-Arg/NIPAAm)]; (C) P(M-Arg/NIPAAm); (D) P(M-Arg/NIPAAm); (E) P(M-Arg); (F) P(M-Arg/NIPAAm) flat hydrogel. (b) Swelling ratio of the PNIPAAm, P(M-Arg/NIPAAm) and P(M-Arg) hydrogels in PBS suffer (pH 7.4, 0.1 M). (c) The amounts of HSA and HPF adsorption per unit area with prepared hydrogels. (d) Evaluation of the wound area closure.

ACKNOWLEDGMENT

The research is supported by the National Key Research and Development Program of China (Project no. 2017YFB0309001) and the Fundamental Research Funds for the Central Universities and sponsored by the Shanghai Pujiang Program 14PJ1400300.
Influence of Various Forms of Polypropylene Matrix (Fiber, Powder, and Film States) on the Flexural Strength of Carbon-Polypropylene Unidirectional Composites

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An investigation into the flexural properties of unidirectional composites (UDC) produced from blending of carbon fiber and polypropylene (PP) matrix in varying forms of fiber, powder and film are reported. Dr. Ernst Fehrer (DREF) hybrid yarns were manufactured from PP fibers and carbon tows, and were consolidated to yield UDC-D. PP powder was layered on carbon tows by electrostatic spray coating and were consolidated to form UDC-P. A third laminate, UDC-F was manufactured by the consolidation of PP films stacked between carbon tows. The fiber volume fraction was ~ 50 % in the UDCs. Better mechanical properties of UDC-P were observed in 3 point bending and short beam tests. Analysis by micro-computed tomography, and scanning electron microscopy indicate better impregnation of PP powder in UDC-P despite higher viscosity than PP fiber.
Advanced Fibers and Textiles
Effect of Long Chain-branched Polypropylene for Melt-blown Nonwoven Fibers

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In the melt-blowing process, the molten polymer extruded from a nozzle is elongated by high-velocity and high-temperature air flow. This is a well-known industrial process to manufacture nonwoven fabrics consisting of randomly orientated fine fibers with the diameter of less than 3μm. Nonwoven fabrics having average fiber diameter of 2 μm or less is widely used for filter applications. Recently, requirement for filtering with higher accuracy and lower pressure loss has been increasing. For this purpose, nonwoven fabrics having finer diameter fibers with more uniform diameter distribution is necessary. Even though various attempts such as the reduction of nozzle diameter and the increase of process air speed were made, it is still difficult to manufacture nonwoven fabrics with the fiber diameter of 1 μm or less. In our previous research, we pointed out that the instability started to occur in elongating polymer melt for producing ultrafine fibers. Eventually, the fiber diameter distribution contained a certain amount of thick fibers. In this study, with the aim of stabilizing the melt blowing process for producing nonwoven fabrics with fine diameter fibers, effect of the control of resin rheology by the introduction of long chain branching to polypropylene (PP) was investigated. It was found that the blending of an optimum amount of long-chain branched polymer to regular PP caused the stabilization of the melt blowing process. As a result, the formation of nonwoven fabrics consisting of fine fibers of rather uniform diameter distribution could be achieved.
Advantages/Disadvantages of 3D Warp Interlock Fabrics: A Review

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3D warp interlock fabrics can be used in several applications as fibrous reinforcement for composite material as well as fibrous material for protective solution. For each of these applications, product and process parameters can be adjusted to provide the required mechanical properties. Several research studies have highlighted the influence of product and process parameters, or a combination of them, onto the mechanical behavior of the 3D warp interlock fabrics. However, an overview of the main expected mechanical characteristics has not yet been provided to identify the advantages and disadvantages of such multi-layer woven structures. To fill this gap, we propose in this paper to summarize and put on the fore the main existing research results dealing with the several properties of these specific 3D structures. Based on our complete definition of these 3D warp interlock fabrics, more scientific knowledge on the influent parameters, leading to the required properties, will be provided. Thus, the objective of this paper aims at providing a more concise representation of their several quasi-static and dynamic mechanical properties to better understand their specific behavior. Several of our research works will strengthen the existing knowledge on dynamic behavior for protective solutions (Figure 1) and mechanical properties for composite material (Figure 2).

Figure 1. Prototype of female body armor made with para-aramid 3D warp interlock fabrics.
Figure 2. Forming behavior of thermoplastic 3D warp interlock fabrics.

KEYWORDS: 3D woven structures, mechanical properties, textile composite.

ACKNOWLEDGMENT
Authors would like to thank the French MoD (DGA) and EU commission (MAPPIC Project) to provide funds to conduct all these research projects.
Recently, fiber/yarn-based energy-storage systems have attracted enormous attention due to their remarkable promises in smart textiles and high-tech sportswear etc., mainly as more pliable energy-storage units being truly “wearable”. Scalable, inexpensive manufacturing processes that produce yarns capable of storing energy are required for new breakthroughs within the wearable electronics sector. For example, US Army is looking for the manufacturing processes capable of producing kilometers of energy-storage yarns that retain desirable mechanical attributes and can be knitted or woven into wearable fabrics. However, so far such processes are still missing. Current technologies include modified textile yarns, composite yarns containing metal wires, polymers, carbon nanotubes (CNTs), metal oxides, and conductive carbon yarns etc. However, several issues exist in current systems, including the biosafety of CNTs, the weight and corrosion of metal wires, and long-term stability of conductive polymers. These issues need to be addressed before they can be employed for practical use.

Here we introduce a unique process of making graphitic yarns (yarns made of graphitic carbon flakes) that has attracted enormous attention internationally. Yarns made of graphite flakes are lighter and more corrosion-resistive than metal wires, and more stable in electrical conductivity than polymers. More importantly, graphite flakes are generally considered to be biofriendly, since they have been used as the major component in pencils for decades. Furthermore, the process of making graphite fibers has several advantages: 1) large-scale availability of the wet-spinning dope, which is graphite oxide dispersions in water; 2) fiber/yarn precursors labile for various chemical/physical modifications; 3) graphite used as the major precursor, which is low cost and abundant in nature; 4) several prototypes have been demonstrated. This talk will introduce our recent research on process engineering and chemical modification of the detailed yarn structures for high energy density and mechanical flexibility.
MXene Fibers: The New Member of Functional Fibers Made from Two-dimensional Nanosheets

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Yarn-shaped supercapacitors (YSCs) once integrated into fabrics provide promising energy storage solutions to the increasing demand of wearable and portable electronics. In such device format, however, it is a challenge to achieve outstanding electrochemical performance without compromising flexibility. Metal carbides or carbonitrides called “MXene” has presented a distinct combination of very high conductivity (~10,000 S cm⁻¹), volumetric capacitance (~1,500 F cm⁻³), and hydrophilic properties. To date, however, MXene-based fibers or yarns that can be used for wearable applications have been relatively unexplored. Their small sheet size (<2 µm) and weak inter-sheet interactions have made it challenging to fabricate MXene-based fibers or yarns. Here, we present strategies to achieve fibers or yarns from Ti₃C₂ MXene. Our works include high-performance yarn supercapacitors fabricated by coating MXene on conductive carbon fiber bundles. This device showed length capacitance of ~132 mF cm⁻¹ higher than the literature reports (typically lower than 100 mF cm⁻¹) even at low mass loading of 2 mg cm⁻¹. We also used wet-spinning technique by taking advantage of the templating role of liquid crystalline (LC) graphene oxide (GO) to produce fibres with high MXene content of ~88 wt. %. These fibres demonstrate excellent flexibility and a high volumetric capacitance of ~341 F cm⁻³. We also employed bisscrolling technique to scroll MXene nanosheets within carbon nanotube helical corridors. Here, the yarns are predominantly MXene containing up to ~98 wt.% MXene yielding an areal capacitance as high as ~3,188 mF cm⁻², which exceeds the previously recorded performance for any fiber or yarn supercapacitor electrode. The supercapacitor prototype in asymmetric configuration reached a maximum energy and power densities of ~61.6 mWh cm⁻³ and ~5428 mW cm⁻³ respectively. We show that the MXene-based fibers and yarns are useful for powering small electronic devices when knitted or woven into a textile. Our works have introduced a new class of fibers that can be excellent candidates for integration within textile-based electronics and wearable devices.
Surface Properties of Carbon Nanotube Fibers: A Wetting Study

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Carbon nanotubes (CNTs) have received considerable attention owing to their unique 1D structure, and their exceptional mechanical and electrical properties combined with light weight. Assembling them into a fiber is a way to circumvent the difficulties of manipulation and dispersion of individual CNTs and to make them more accessible for applications, e.g. as reinforcement in polymer composites and as electrode casted with polymer electrolyte in energy devices. For these applications, the interfacial adhesion between the CNT fibers and the thermoplastic matrix plays a critical role, in particular for the mechanical performance of the composite materials. Therefore, accurate characterization of the wettability of CNT fibers is one of the most important cornerstones to improve the development of these composite materials.

In this work, the wettability of thin CNT fibers (15 to 20 µm in diameter) was studied by precisely measuring dynamic contact angles made between deionized water, ethylene glycol, and diiodomethane and single fibers by using a tensiometer combined with a synchronized optical observation [1]-[2] (Figure 1). The contact angle values obtained from both the optical and the force methods are in very good agreement validating our methodology. The optical method also allowed us to observe the pinning and fluctuation of the contact line, which were triggered by defects on the CNT fiber surface. Furthermore, a link between the contact angles measured at this scale (microscale) and the contact angles on individual CNTs [3] (nanoscale) was then derived by using a modified Cassie Baxter equation [4]. The surface energy components of CNTs were then obtained from the calculated contact angles, revealing that CNTs have a much smaller polar component than the non-polar component. This result was finally confirmed by X-ray photoelectron spectroscopy.

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ACKNOWLEDGMENT
This work was partially supported by the SIM Nanoforce program and KU Leuven Industrial Research Fund (IOF-KP/14/005). We also thank A-Tech System for the dry-spinning tool and their technical help. J. Wang thanks the China Scholarship Council (CSC) for offering scholarships.
Aerogels are among the least dense, approaching density of air, and the most porous materials with porosity over 99%, thus highly unique for many potential applications. While the most studied and commercially available aerogels are silica aerogels, interest in aerogels has been extended to include those based on carbon, organic materials, metals and metal oxides. Being able to shape aerogels into desired forms and dimensions is essential for their applications. The typically low tensile strength and Young's modulus of these aerogels, however, make them friable to be handled, used alone or to be integrated with other flexible, stretchable, and conformable materials, such as textiles. Nevertheless, the idea of incorporating three-dimensional (3D) aerogels into fibers or filaments are attractive from the standpoint of enabling fabrication into fibrous mats, woven and nonwoven fabrics, and flexible or wearable materials for thermal protective applications under either hot or cold extreme temperatures. Thermal insulation is among many of the diverse potential applications of cellulose nanofibril (CNF) aerogels. First, cellulose is among the lowest thermal conductive materials. The CNF aerogel cellular structure further limits air circulation, making them good convective inhibitors, while their low transparency to infrared radiation inhibits radiative transmission. Such distinguished thermal insulation potential of these CNF aerogels coupled with their unique resiliency and biological origin are particularly attractive for wearable thermal insulators. Therefore, shaping the 3D bulk aerogel into 1D fibers is envisioned to enable integrating CNF aerogels into such devices with scalable potential.

This paper presents the first continuous, strong, and coaxial aerogel fibers via wet-spinning of hollow cellulose acetate (CA) fibers (Figs 1a,b) and infusion of CNF aerogel precursor (Figs 1c,d). The multiscale porous sheath functions as a processable template and a surface layer to protect the integrity of the CNF aerogel from external forces or the environment (Figs 1e,f). Their fibrous form, optically-thick characteristics to infrared radiation (Fig 1g) and highly porous nature in both core and sheath offer unique thermal insulation characteristics. The specific combination of characteristics include high porosity (85%), high specific tensile strength (23.5 ± 2.5 MPa g cm⁻³), low density (0.2 g cm⁻³), wide working range (-20 to 150 °C), continuous and large-scale producibility, as well as biodegradability and cost-effectiveness. Both the porous sheath and the CNF aerogel core of the coaxial fibers play crucial roles in obtaining excellent thermal insulation performance when compared with the commercial cotton fabric of comparable thickness. Most significantly, the fabrication approach opens up new engineering approaches to fabricate nanocellulose aerogels in continuous filaments of varied fiber diameters and is scalable.

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ACKNOWLEDGMENT
The authors appreciate the funding support from the California Rice Research Board.
Carbon Nanotube/Polyvinyl Alcohol-coated Yarn: An Advanced Fiber for Multifunctional Textiles

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Textile, as a desired platform for wearable smart technology, can be integrated with smart element into the hierarchical levels of the fabric structure. However, for exploiting in the development of wearable devices, the sensors should be able to support the deformations of the textile simultaneously, without affecting the original textile characteristics such as softness, breathable and draping properties. Carbon nanotube yarn (fiber) made by twisting of axially aligned CNT assembly or floated CNT aerogel inherits the excellent mechanical, electrical as well as piezoresistive properties. Compared with the traditional rigid and brittle multifunctional fibers, the CNT yarn shows the advantages of light weight and high flexibility as well as the multiple sensing properties.

In this study, a multifunctional fiber based on carbon nanotube/polyvinyl alcohol (CNT/PVA) coated yarn, as shown in Figure 1, is manufactured, which employs good mechanical property, anti-abrasion, high conductivity, excellent strain and humidity sensitivity. CNT/PVA coated yarn with a core-sheath structure (inner pure CNT core and outer CNT-PVA sheath) exhibits a linear piezoresistive response, with a high gauge factor (the ratio of electrical resistance changes to strain change) of 2.36. Compared with the pure CNT yarn, the PVA coated yarn has an improved tensile strength by 71.8%, Young’s modulus by 157.3%, abrasion-resistance by 100%, and enhanced stability after cyclic loading. Moreover, the electrical resistance of the virgin humidity sensing material remains almost constant at low relative humidity (RH), and then increases sharply as the RH increases above 75%, showing a good humidity switch characteristic. Furthermore, the fabric strain sensor integrated with CNT/PVA yarn exhibited quick and precise response to the finger motion detection, demonstrating potential in wearable electronic.

ACKNOWLEDGMENT
This work was financially supported by the National Natural Science Foundation of China (Grant No. 51303025), Shanghai Natural Science Foundation (Grant No. 17ZR1400800), also funded by the Fundamental Research Funds for the Central Universities and DHU Distinguished Young Professor Program.
Study of Auxetic Woven Fabrics Based on Re-entrant Hexagonal Geometrical Structures

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The auxetic material is a special kind of materials that possess negative Poisson’s ratio (NPR), where the materials laterally expand when stretched and shrink when compressed. Auxetic textiles have been developed in the recent years including many areas that are yarn, fabrics, braids, non-woven and composites. However, the use of conventional yarns and machinery to produce auxetic fabrics has become more interesting, but a challenge to researchers. Most commonly, this approach is applied to knitted fabrics. Such fabrics may have many potential applications including maternity clothing, under garments, shape wear, under wear, leggings and sportswear. Compared to auxetic knitted fabrics, the development of auxetic woven fabric using this approach is still limited. This work reports a study on the fabrication and testing of auxetic woven fabrics based on re-entrant hexagonal geometrical structures. The re-entrant geometrical structure shows in Fig. 1(a), where the structure applied along the warp direction of fabric as highlighted in Fig. 1(c). Also, Fig. 1(b) shows the extended state of geometry where the structure will expand in the transverse directions resulting in the NPR effect. The NPR effect of fabric is discussed in terms of different extension directions, including warp direction, weft direction, and diagonal directions. In addition, the tensile repeating test was also conducted to observe the influence of repeated tensile loading on the auxetic effect of fabric. To conduct the tensile tests, the Instron 5566 tensile machine was used. The result that the NPR effect of the fabric varies significantly with extension direction, as shown in Fig.1(d). The highest auxetic effect was obtained in the warp direction in contrast to the weft and diagonal directions. Also, the results of tensile repeating test demonstrated that the fabric can retain around 46% of auxetic behavior even after 20 cycles of tensile loading shown in Fig. 1(e). It is suggested that the re-entrant hexagonal geometry can produce auxetic woven fabric where the fabric has NPR effect and keep auxetic behavior after many repeated uses.

![Fig. 1. Auxetic woven fabric developed: (a) free state of re-entrant geometry; (b) extended state of re-entrant geometry; (c) real fabric with geometrical unit cell; (d) NPR results (poisson’s Ratio vs tensile strain); (e) tensile repeating test results.](image-url)

ACKNOWLEDGMENT

This work was supported by the Research Grants Council of Hong Kong Special Administrative Region Government (grant no. 15205514).
Spider Silk-inspired Supertough Polyurethane/Urea Fibers

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Spider silks have been brought into the focus of research for decades due to its perfect balance of strength and toughness. Typically, Nephila spider dragline silk has a strength of 1.3 GPa, a breaking elongation of 40%, and a toughness of ~150 MJ m\(^{-3}\). Spider silks have β-sheet, α-helical, and random coil structures, where β-sheet nanocrystals have been identified as a key component responsible for its excellent mechanical properties. In this work, inspired by spider silks, β-sheet peptide segments were incorporated into polyurethane/urea chains to improve the material properties. The obtained polyurethane/urea fiber display a tensile strength of ~100 MPa, an elongation at break of ~750%, and a toughness of 389.7 MJ m\(^3\) (Figure 1), which is more than twice the toughness of Nephila spider dragline silk. We believe this work will provide new insight for developing high strength and toughness materials, which would have various potential applications such as wearable and implantable electrons, and biomedical devices.

Figure 1. Stress-strain curve of peptide-polyurethane/urea fiber.

KEYWORDS: Spider silk, β-sheet nanocrystals, polyurethane fiber, bioinspired.

ACKNOWLEDGMENT
This work was financially supported by the National Natural Science Foundation of China (51673162), Research Grants Council, University Grants Committee (PolyU 5158/13E), and Hong Kong Scholars Program (XJ2016052).
Directional Water Transport in the Trilayered Fibrous Membranes for Smart Moisture Wicking Textiles

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Directional water transport is a predominant part of functional textiles used for continuous sweat release in daily life. However, it has remained a great challenge to design such textiles which ensure continuous directional water transport and superior prevention of water penetration in reverse direction. Here, we report a scalable strategy to create trilayered fibrous membranes with progressive wettability by introducing a transfer layer, which could guide the directional water transport continuously and spontaneously, thus preventing the skin from being rewetted. The resulting trilayered fibrous membranes exhibited a high one-way transport index $R$ (1021%) and a desired breakthrough pressure (16.1 cm H$_2$O) in the reverse direction, indicating an ultra-high directional water transport capacity. Moreover, on the basis of water transport behavior, a plausible mechanism was proposed to provide insight into the integrative and cooperative driving forces at the interfaces of trilayered hydrophobic/transfer/superhydrophilic fibrous membranes. The successful synthesis of such fascinating materials would be valuable for the design of functional textiles with directional water transport property for personal drying applications.

Fig. 1. Directional water transport mechanism of trilayered fibrous membranes: a) Water drops onto the upward hydrophobic PU side and penetrates the membranes immediately. b) Water drops onto the upward superhydrophilic HPAN side and spreads rather than penetrates. Schematic illustration of the directional water transport mechanism: water is dropped on the upward c) hydrophobic PU side and d) HPAN side, respectively.

ACKNOWLEDGMENT
This work is supported by the Program for Professor of Special Appointment (Eastern Scholar) at Shanghai Institutions of Higher Learning (No. TP2016019), the Shanghai Rising-Star Program (No. 16QA1400200), the National Natural Science Foundation of China (Nos. 51473030, 51503028, and 51673037), the National Key R&D Program of China (No. 2016YFB0303200), the Innovation Program of Shanghai Municipal Education Commission (No. 2017-01-07-00-03-E00024), and the Fundamental Research Funds for the Central Universities (No. 2232016A3-03).
Breathable Multifunctional Materials for Dynamic Protection from Chem/Bio Threats

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New materials are required for the fabrication of advanced multifunctional garments that allow high moisture vapor transport rates (MVTR) while blocking toxic chemicals and biothreats. In particular, for in-the-field personnel protection from chemical and biological (CB) agents, smart dynamic materials are highly desirable that exhibit a reversible, CB-triggered, rapid transition from a breathable state to a protective state. Toward the realization of this new paradigm of adaptive protection, we are developing a chemical threat responsive material based on a surface-functionalized carbon-nanotube (CNT)-membrane, in which vertically-aligned CNTs function as the only pores in an otherwise impermeable, polymeric film. Response to the threat is triggered by direct chemical warfare agent (CWA) attack to the functional groups at the membrane surface, upon which the membrane switches from a highly breathable state in no-threat environment to a protective state by closing the CNT pore entrance to CWA permeation.

To demonstrate this concept, we first fabricated membranes with sub 5-nm CNT pores and quantified their breathability and rejection properties before functionalization with CWA-responsive polymers. Our results show that these membranes provide MVTR up to 11,000 gr/m²day, thus exceeding state-of-art breathable fabrics (eVent, GoreTex, etc.) even if the moisture conductive pores are only a few nm wide. Complete rejection of 3-nm charged dyes, 5-nm uncharged gold nanoparticles, and ~40-60-nm Dengue virus from aqueous solutions during filtration tests demonstrates that our CNT membranes provide a high degree of protection from bio-threats by size exclusion.

Then, we covalently grafted actuating polymers responsive to G-agent simulants to the surface of these CNT membranes. Upon exposure to simulants, these membranes switch reversibly from a breathable state with MVTR> 4,000 gr/m²day to a protective state with MVTR> 1,000 gr/m²day, and the material can be regenerated with a simple base treatment. Permeation tests reveal also that the membrane blocks ~99% of a nerve agent simulant in the protective state.

These results suggest that CNT membranes functionalized with CWA-responsive, actuating polymers could combine on-demand protection with high breathability in a single multifunctional material, and achieve the goal of a lighter, cooler, smarter protection of military and civilian personnel in CB contaminated environments.

ACKNOWLEDGMENT
This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. LLNL-ABS-xxx.
Long-term Stability of Copolymer Aramide Fibers

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Traditionally, soft body armor has been made from poly(p-phenylene terephthalamide) (PPTA) and ultra-high molecular weight polyethylene fibers. However, to diversify the fiber choices in the United States body armor market, copolymer fibers based on the combination of 5-amino-2-(p-aminophenyl) benzimidazole (PBIA) and the more conventional PPTA were introduced. Little is known regarding the long-term stability of these fibers, but as condensation polymers, they may have potential sensitivity to moisture and humidity. Therefore, characterizing the strength of the materials and understanding their vulnerability to environmental conditions is important for evaluating their use lifetime in safety applications. Ballistic resistance and other critical structural properties of these fibers are predicated on their strength. To accurately determine the strength of the individual fibers, it is necessary to disentangle them from the yarn without introducing damage. Three aramide-based copolymer fibers were selected for the study. The fibers were washed with acetone followed by methanol to remove an organic coating that held the individual fibers in each yarn bundle together. This coating makes it difficult to separate single fibers from the yarn bundle for mechanical testing without damaging the fibers and affecting their strength. After washing, Fourier transform infrared (FTIR) spectroscopy was performed on both washed and unwashed samples and the results compared. This experiment has shown that there are no significant variations in the spectra of poly(p-phenylene-benzimidazole-terephthalamide-co-p-phenylene terephthalamide) PBIA-co-PPTA1 and PBIA-co-PPTA3 after washing, and only a small variation in intensity for PBIA. This indicates that the acetone and methanol rinses are not adversely affecting the fibers and causing chemical degradation. Additionally, single fiber tensile testing was performed on the washed fibers to characterize their initial tensile strength and strain to failure and compare those to other reported values. Iterative procedural development was necessary to find a successful method for performing tensile testing on these fibers. Once the method development was completed, an ageing study was begun in which the fibers were exposed to various conditions of elevated heat and humidity, and the effect on their mechanical properties was evaluated. Preliminary results from this ageing study will be the subject of this presentation.
Physiological and Physical Strain Associated with Wearing Chemical Protective Clothing in a Hot Environment

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Chemical protective clothing (CPC) is one of the most important personal protective equipment (PPE) engineered to protect human beings from chemical and biological hazards. However, the wearer suffers substantial heat stress and physical strain due to the combined human-clothing-environment factors such as physical nature of wearer’s work, vapor impermeable and heavy CPC ensembles, and challenging thermal environment, which will limit working duration, lead to heat-related illness, and even cause injury and death. Therefore, there is an urgent need to investigate both the physiological responses and physical burden to better design the CPC and improve the benefits of the wearer.

METHODS

Eight participants were selected and each of them wore T-shirt shorts (Control) and a type of CPC (level B) while walking on a treadmill in a climate chamber at 40 °C and 70% relative humidity. During the testing, the physiological responses were measured continuously including heart rate, core temperature, and skin temperature. Based on the measured physiological responses, the body temperature, heat storage, physiological heat strain index, sweating rate, and tolerance time were calculated. The physical burden, such as perceived wetness, perceived hotness, restriction of movement, and rate of perceived exertion, was also recorded with survey questionnaire.

RESULTS AND DISCUSSION

The results demonstrated that the CPC imposed greatly physiological strain in comparison with the control in terms of core temperature (Fig.1), skin temperature, body temperature, heart rate, sweating rate, heat storage, and physiological strain index (Fig.2), which were consistent with several documented studies.

Furthermore, the level B reduced the tolerance time by 50% compared to the control. Wearing CPC interfered with wearer’s movements and perceived exertion due to the bulky design of CPC and its stiff and heavy material. The CPC created ergonomic problems compared to the control in trials. At the end of the exercise, subjects all felt very hard (PRE values between 17.40 and 17.75) to keep walking.

CONCLUSION

The vapor impermeable CPC caused considerable physiological strain and seriously physical strain compared with the control. The study is capable of providing guidance on the design and development of high-performance CPC.
Characterizing Thermal Protective and Thermo-physiological Comfort Performance of Fabrics Used in Firefighters’ Clothing Using 2D Intermediate Tests

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Evaluation of thermal protective and thermo-physiological comfort performances of firefighters’ clothing is essential to understand protection provided and heat stress induced by the clothing. Standardized bench-scale tests are less complex, convenient and inexpensive to evaluate the performances of fabrics used in the clothing (1D tests). However, these tests are limited regarding standard flame/radiant-heat exposure and configurations of tested fabrics. Therefore, more complex but standardized manikin tests are preferred to evaluate the performances of complete clothing (3D tests). Manikin tests for protective performance simulate more complex flash fire hazards but are expensive and accurate data analysis and interpretation is challenging. Considering the limitations of 1D and 3D tests, intermediate tests with simplified geometries have been developed for evaluating the performances of fabrics using cylindrical devices (2D tests). To date, 2D tests have not been studied in detail to characterize and understand the effects of fabric properties on protective and comfort performances. This study aims to fulfill this gap for a better understanding of the performances.

METHODOLOGY
A set of single- and multi-layered fabrics were selected and their physical properties (weight, thickness, air permeability, thermal resistance, evaporative resistance, water spreading speed) were measured using standard test methods. Next, thermal protective [in terms of amount of thermal energy transferred through the fabrics ($E_{trans}$) and percentage of burn injuries (Burn%)] and thermo-physiological comfort [in terms of Cooling Performance (CoolPerf) and comparative Time to Heat Stress (cTHEST)] performances of these fabrics were evaluated using 2D tests (Figures 1a and 1b). The relationship between fabric properties and performances were investigated to identify key properties affecting the performances.

RESULTS AND DISCUSSION
Weight, thickness, and thermal resistance positively affected protective performance but had a negative impact on comfort performances. Additionally, air permeability and evaporative resistance showed significant effects on protective and comfort performances. Thermal and evaporative resistances were found to have key roles for the protective and comfort performances, respectively.

CONCLUSION
This study provides an insight into the effect of fabric properties on protective and comfort performances using simplified intermediate 2D tests. This could lead towards developing new fabrics for better protection and comfort of firefighters worldwide.
Tensile Testing of Aged Flexible Unidirectional Composite Laminates

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Unidirectional (UD) laminates are incorporated in many body armor designs, and are constructed of thin layers of high-performance fibers held in place using binder resins and thin polymer films, with the fibers in each layer oriented parallel to each other. Armor is constructed by stacking such unidirectional layers in different orientations, typically alternating 0 degree and 90 degree layers. To date, only very preliminary work has been performed to characterize the ageing of unidirectional laminates and its effects on performance, particularly with regards to the binder resins. These resins have been seen to degrade when after ageing UD laminates showed visual signs of delamination and reductions in V50, which is the velocity at which half of the projectiles are expected to perforate the armor, in the development of the conditioning protocol used in NIJ Standard-0101.06. To accurately predict the long-term performance of armors constructed from UD laminates, a better understanding of the material property changes with ageing is necessary. As there are no current standards recommended for mechanically interrogating unidirectional (UD) laminate materials this work presents a new test methodology for accurately testing the mechanical properties of UD materials, as well as data for specimens aged up to 20 weeks at 70 C and 76% RH.
Membranes that can strictly resist the penetration of liquid water while easily transmitting water vapor have attracted considerable interest owing to their numerous applications, including protective clothing, wound dressings and outdoor tents and so on. Although several strategies such as biaxial stretching, phase separation, and melt blown have been developed, it remains an enormous challenge to fabricate membranes with best equilibrium of breathable and waterproof performance. Herein, we fabricated electrospun hydrophobic polyurethane (PU)/ Fluorinated polyurethane (FPU) fibrous membranes with tunable porous structure by regulating the relative humidity (RH). Significantly, the effects of porosity, mean pore size and maximum pore size on the water vapor transmission (WVT) rate and hydrostatic pressure have been investigated. Consequently, the fibrous membranes obtained at RH of 50% possessed an optimized porous structure with porosity of 60%, mean pore size of 1.6 μm and maximum pore size of 2.4 μm. The resultant fibrous membranes exhibited best equilibrium performance with WVT rate of 5.6 kg m⁻² d⁻¹ and hydrostatic pressure of 54.7 kPa, as well as good mechanical behavior with tensile strength of 12.5 MPa, indicating promising alternatives for many potential applications, especially in protective clothing.
The U.S. Army Natick Soldier Research, Development, and Engineering Center, in collaboration with the Product Manager for Soldier Clothing and Individual Equipment under the Program Executive Office-Soldier, is pursuing capabilities to revolutionize the next generation Soldier Combat Ensemble. Efforts will be focused on the incorporation of smart materials and wearable technologies into a Soldier ensemble in such a way as to create a capability platform that enhances Soldier awareness, performance, and lethality. The initial focus areas may include signature management, ballistic protection, load enhancement, Soldier-borne sensors, power storage, and power generation. Emerging technologies may consist of, or be integrated with, fibers, yarns, fabrics, and/or membrane-based materials. The Army will present its vision for a Soldier Combat Ensemble platform as a means for initiating future collaborations.
Posters
Smart Chair: Choice and Location of Sensors

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INTRODUCTION
Sitting is the most common position these days. You can sit and spend hours at work, driving long distances or resting. Studies have shown that sitting in a seated position for a long time could be bad for the health, causing muscle and back pains.

The chair, the bed, and the armchairs are the direct interface between the human body and the sitting position. These devices are generally formed of a metal or plastic frame, a foam sitting shape and a textile material. In this frame, connected textiles can be a means to improve the sitting position and to avoid the complications that could be induced. These connected textiles are mainly composed of smart material that can be used in different shape: fibers, threads, ink.

SENSOR INTEGRATION IN THE TEXTILE
Knowing the distribution of the pressure a sitting device is the goal of ergonomists and kinesics. The pressure distribution provides all the information needed to evaluate a sitting position such as pressure peaks, critical areas and posture defects. From these data, advice and improvement functions can be proposed. The measurement of the pressure on the sitting devices can be performed by acting on one of the elements that constitutes them; the metallic structure, the foam shape and the textile material. Currently, various materials are in the research phase, some of them are already marketed.

FLEXIBLE SENSOR TYPES
Depending on the nature of the application, the design of the pressure sensors could change. In our case, the sensor must be able to adapt to the seat surface, then flexibility is certainly the first criterion of them. There are generally three modes of operation:
- Piezoresistive sensor
- Piezoelectric sensor
- Capacitive sensor

INTEGRATION TECHNIQUES
Smart materials can be integrated using several techniques: weaving, knitting, embroidery and printing. The choice of technique depends on the application and its characteristics and the localization is directly linked to the parameter you would like to measure.

CONCLUSION
Up to now several technics have been tested and are under evaluation in order to know if they answer to the full requirement that has been set-up. Moreover, a localization study is currently running to be sure to place the sensor in the right place. The next step will be the electronic set-up and the data treatment that could be used for such kind of application.
Scalable Fabrication of Highly Carbonylated Nylon Nanofibrous via \textit{in situ} Citric Acid Grafting for Reversible Adsorption of Laccase by Chelated Copper Ions

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In this work, a novel laccase enzyme adsorbent was prepared by functionalization of activated nylon 6 with formaldehyde in an acidic medium and a metal chelating citric acid (CCA) was covalently attached to hydroxyl groups activated nylon surfaces by EDC/NHS to form coordinative complexes with copper metal ions. The resultant Ny-CCA-Cu (II) NFM exhibited an excessive laccase enzyme adsorption capacity of 220 mg g\textsuperscript{-1}, fast equilibrium within 6 h, and good reversibility. Taking advantage of the high specific surface area, the highly tortuous open-porous structure of nanofibers and accessible grafted carboxylate groups in situ and chelated metal ion to present excellent enzyme adsorption by chelation and coordination chemistry. Moreover, the released laccase enzyme keeps 85\% of its initial activity and Ny-CCA-Cu (II) NFM can be used more than 5 cycles without significant loading capacity changes. The nanofiber unique structure didn’t change by frequently reusing. The chemical and morphological structure for Ny-CCA-Cu (II) NFM were characterized before and after modification by FTIR, contact angle and SEM. Meanwhile, the adsorption performance was optimized by regulating buffer pH, the initial concentration of laccase enzyme and CCA content. The dynamic binding efficiency of 160 mg g\textsuperscript{-1} can be achieved driven solely by the gravity of protein solution. This simple approach may introduce a way for the design and development of robust, cost-effective and eco-environmentally adsorption membranes to meet the great demands for fast and efficient adsorption of positively charged proteins and matches well with the demands of the high yield and energy conservation in industrial scale.
Effect of Spinning Conditions on Mechanical Properties of Polyarylate Fiber in Melt-spinning Process

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Polyarylate fiber is one of commercial industrial fiber materials that have superior mechanical performance (high strength and modulus, low creep), as well as excellent durability in extreme environments (thermal stability at low/high temperature, water- and chemical-resistance). The polyarylate fiber products have been widely used for reinforcing materials such as bullet-proof wears, exterior materials of spaceships, and submarine cable. Polyarylate, thermotropic liquid crystalline polymer (TLCP), can be spun by melt-spinning process, and the spinnability and mechanical properties of polyarylate fibers are highly sensitive to process temperature and discharge characteristics (shear rate, hole size and etc.) in melt-spinning process, because TLCP show high shear thinning behavior.

Herein, we studied that the effect of spinning condition (spinning temperature, shear rate and etc) on mechanical properties of as-spun polyarylate fiber in melt-spinning process. As a raw material, polyarylate resin was provided by S company in Korea. The polyarylate was melted by using single-screw extruder and extruded through spinnerets by using gear pump. The spinneret with diameter of 0.15-0.3 mm and L/D of 4.0 was used and the spinning temperature was 290-310 °C. The as-spun fiber was collected by using low-speed winder. The obtained as-spun fibers were characterized by tensile test and SEM, and we could evaluate the effect of process conditions on the processability and mechanical properties of polyarylate fibers in the melt spinning process.
Highly-sensitive, inexpensive, and portable electrochemical biosensor was successfully developed for rapid detection of malathion residues in olive oil. The bio-tool has been fabricated based on genetically modified acetylcholinesterase immobilized onto nylon nanofibrous membranes decorated with citric acid via EDC/NHS. Owing to the unique properties of the membranes such as spatial structure, high porosity, and large surface area, the amperometric response of the biosensor improved by more than two folds. The biosensor exhibited high sensitivity against malathion residues with a detection limit (LOD) of $5.8 \times 10^{-11}$ M, this limit is lower than the maximum residue limit (MRL) set by international regulations. The biosensor demonstrated feasibility and practicality in commercial food safety measurements by successfully detecting malathion residues in olive oil after a simple liquid-liquid extraction.

**KEYWORDS:** Biosensor, nanofibers, acetylcholinesterase, pesticides, olive oil.
A Silicon-containing, Persistently Flame-retardant PA6 with Simultaneously Improved Anti-dripping and Mechanical Properties

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To overcome the easy migration and poor compatibility of additive flame retardants, a persistently inherent flame-retardant PA6 (FR-PA6), with 1, 3-bis(3-aminopropyl) tetramethyldisiloxane (MSDS), was first synthesized via controllable amidation and polycondensation reaction. MSDS played a reinforcement effect on condensed phase by insulating the external combustible environment. With the incorporation amount of 9 wt%, FR-PA6-9 could be extinguished after 6 melt droplets fall and passed UL-94 V-2 rating, while the level of PA6 was no rating (NR). Unexpectedly, MSDS enhanced the mechanical properties of FR-PA6 with the higher tensile stress and elongation at break than neat PA6. The inspiring results offered possibility that designing the FR-PA6 based composites by adding particles to achieve higher flame retardancy. Melamine cyanurate (MCA) nanoparticles as gas-phase synergistic agents were then blended with FR-PA6. The as-prepared FR-PA6/MCA composite with 12 wt% of MCA attained the limiting oxygen index (LOI) value of 32.7%, passed the V-0 level with only 1 melting droplet and sharply diminished the peak heat release rate (PHRR) (30.4%). Moreover, the composite still maintained the similar mechanical property to neat PA6 even though the addition of MCA deteriorated the mechanical property of FR-PA6. The special ‘condensed-gas phase’ flame-retardant mechanism of FR-PA6/MCA was also proposed through characterizing the residues and pyrolysis volatiles. This work provided a facile route to prepare a silicon-containing persistently flame-retardant PA6 with simultaneously improved anti-dripping and mechanical properties.

ACKNOWLEDGMENT
This work is financially supported by the National Key Research and Development Program of China (2016YFB0302700), Intelligent Manufacturing Program of China and the Fundamental Research Funds for the Central Universities (CUSF-DH-D-2018037).
Plasticizers have the characteristic of improving polymer processing by reducing melting temperatures and increasing free volume at the expense of mechanical properties—the tensile modulus of polymeric materials are greatly reduced. In contrast, antiplasticization describes process improvements without sacrificing tensile modulus. This study reviews the characteristics of small molecule, biomass derivatives and lignin that express antiplasticization. Matrix polymers of poly(vinyl alcohol), PVA, and polyacrylonitrile, PAN, were evaluated in the context of antiplasticization by aldaric acids (a ≤ 5% of polymer mass) and lignin at (5-50% of polymer mass). We employed the gel-spinning technique (for highly drawn fibers using vinyl polymers) to compare the effects of biomass derivatives on the drawability and mechanical strength of fibers prepared from polymers of 100-250 kDa in molecular size. PVA fibers had shown greater mechanical performance (as high as 1.4 GPa tensile strength, 49 GPa tensile modulus and 35 J/g toughness at 0.8% glucaric acid) versus that of neat control fibers (0.34 GPa, Young’s Modulus 21 GPa, and toughness 2 J/g). Further, the addition of aldaric acids improve the mechanical properties of PAN fibers, while also improving their draw ratio during fiber spinning. In this talk, we explore structural reasons for improved processing and fiber strengthening (see Figure 1).

Figure 1. Plastic failure of PVA fiber that was processed with ammonium glucarate is shown. The molecular interactions between PVA and antiplasticizer are represented in the schematic of crystalline polymer.

ACKNOWLEDGMENT
The Nonwovens Institute supported this research. This work was performed in part at the Analytical Instrumentation Facilities (AIF) at NC State University, which is supported by the state of North Carolina and the National Science Foundation (Award Number ECCS-1542015). AIF is a member of RTNN, a site in the National Nanotechnology Coordinated Infrastructure (NNCI). Pure Lignin Environmental Technology Ltd. and Kalion donated the lignin and glucarates used in this study, respectively.
Cellulose nanocrystals (CNCs) and nanofibrils (CNFs), most commonly derived by respective H₂SO₄ hydrolysis and TEMPO ((2,2,6,6-tetramethylpiperidin-1-yl)oxyl) oxidation, are hydrophilic and water dispersible but incompatible with organic liquids or hydrophobic synthetic polymers without additional surface modification. In this work, more hydrophobic cellulose nanofibrils (CNFs) were directly generated from cellulose by an efficient one-step telomerization using a unique multifunctional reagent, butadiene sulfone (BDS), followed by mechanical blending. BDS mimics dimethylsulfoxide (DMSO) and is a solid at the ambient temperature, melts at 65 °C, and decomposes into gaseous 1,3-butadiene and sulfur dioxide at or above 90 °C, thus multifunctional at equilibrium. In this process, 1,3-butadiene was telomerized at 110 °C with hydroxyl groups of amorphous cellulose in the presence of palladium into 2,7-octadienyl ether (ODE) and upon full fibrillation produced ODE-CNFs at ca. 90 wt.% yield, with the average length/width/thickness of 1.6(±0.5) µm/1.4(±0.3) nm/4.4(±2.2) nm, respectively. The surface ODE-group was characterized by ¹H NMR following procedure we established (d₆-DMSO, δ:1.47, 1.95, 2.07, 4.86, 5.29, 5.61, 5.90, 6.07 ppm) to have an overall DS of 0.17 based on integration and evident as weak alkene FTIR peak at 1660 cm⁻¹ (C=C stretches) and 3040 cm⁻¹ (H-C= stretches). The more hydrophobic ODE-CNFs could be dispersed in non-polar chloroform at 0.0005 w/v% and visualized as individual fibrils on hydrophobic graphite (Figure 1a). Hydrophobicity was further confirmed by water contact angles (CA) of 81.1 ° over dried 20 µL 0.02 w/v% ODE-CNFs (Figure 1c) and 94.0 ° over dried ODE-CNCs in CHCl₃ (Figure 2d). ODE-CNFs had similar thermal stability as cellulose, thus more thermally stable than TEMPO-CNFs. The size of CNFs can be tuned into finer and shorter CNCs (length/width/thickness; 284.0(±246.0) µm/2.6(±0.8) nm/1.0(±0.5) nm) by further hydrolysis with H₂SO₄ produced from adding water to SO₂. These data fully validate the telomerization reaction to be effective in generating more hydrophobic nanocelluloses. This simple one-pot reaction approach has proven to be highly versatile and tunable for both levels of hydrophobicity and forms of nanocelluloses, i.e., CNCs and CNFs, supplying broad range of organic dispersible and hydrophobic nanocelluloses for many applications involving organic formulations and synthetic thermoplastics.

Figure 1. (a) AFM image of ODE-CNf from the aqueous precipitate dispersed in CHCl₃ deposited on graphite; water contact angle on (b) mica; (c) 0.02 w/v % ODE-CNf in CHCl₃ dried on mica; (d) 0.02 w/v % hydrolyzed ODE-CNf in CHCl₃ dried on mica. The higher CA of CNCs attributes to their smaller aspect ratio.

REFERENCE
Fabrication of Bimetallic Nanoparticle Platforms for Fluorescence Enhancement

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Fluorescence enhancement is widely employed for the trace detection in many fields such as medical diagnosis, environment science and sensing because of its splendid specificity and sensitivity. Attributed to the unique localized surface plasmon resonance (LSPR) effect of metal nanoparticles (NPs) they are always designed to construct multifarious nanostructures to achieve enhanced fluorescence. The plasmon properties of metal nanoparticles play an important role in fluorescence enhancement, especially when the fluorescent dyes are located in proximity to the nanogaps between the closely distributed metal nanoparticles. Here, we present the manufacture of the bimetallic nanoparticle platforms using silver (Ag) and gold (Au) NPs to develop abundant narrow nanogaps at the vacant spaces on the prepared substrate. Owing to the strong plasmon coupling effect between the Ag and Au NPs, the Ag/Au hybrid platforms possess intense electromagnetic fields for fluorescence enhancement. These fabricated Ag/Au hybrid platforms which were decorated by the fluorescent dyes exhibited favorable fluorescence enhanced factors compared with control sample, and also improved the fluorescence enhancement efficiency of Au NPs added to the Ag NP platforms. Moreover, the finite-difference time-domain (FDTD) calculations further demonstrated the obviously enhanced electromagnetic fields at the nanogaps between Ag and Au NPs on the Ag/Au hybrid nanostructure in contrast to that on the individual Ag or Au NPs. Therefore, the bimetallic nanoparticle platforms prove to be feasible and effective to achieve the fluorescence enhancement and this work provides a promising strategy to fabricate the fabric-based metal enhanced fluorescence detection platforms on the large scale.
Study on Fiber Structure Development of High-molecular Weight PET in Islands-in-the-Sea Bicomponent Melt-spinning Process

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Bicomponent melt-spinning is one of the most widely used processes to produce high functional and special fibers such as having islands in the sea (SI) or sheath-core (SC) type cross section. In recent, many studies reported that the structure development of each polymer components was enhanced or suppressed in bicomponent fiber systems. The structure development of bicomponent fibers was strongly influenced by spinning conditions; such as spinning velocity or temperature, as well as surrounding environments; quenching air, polymer used, or interfacial area. However, the structure development of SI fibers at various spinning conditions was rarely studied.

Herein, we demonstrated that the structural development of SI fiber according to spinning velocity in high-speed melt-spinning process. As a raw material, HMW-PET with IV 1.18 dl/g and PP with MFR 25 g/10min were used for islands and sea components, respectively. The weight ratio of PP/PET components were 0.5:0.5 and the number of islands in single fiber was 74. The PP and PET were extruded at the spinning temperature of 290 °C and spinning velocity was 1.0-4.5 km/min. The hole diameter of spinneret were 0.5 mm and throughput rate was 2 g/min. To evaluate the structural development of SI fibers, prepared as-spun fibers were characterized by DSC, SEM, WAXD, tensile test compared with PET and PP single-component fibers. As results of structure analysis, we confirmed that the structure development of PP and PET components was suppressed and enhanced in SI fibers and the crystallinity and crystal size of PET component were significantly maximized by bicomponent fiber spinning systems.
Soluble Fiber Products

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We have created the first soluble fiber platform for personal and home care goods. The products are made from commercial polymers and surfactants that have been spun into fibers and converted into a host of products such as detergent, shampoo, conditioner, body wash, etc. This poster summarizes our first commercial execution of the platform, called DS3. In doing so, we are learning the many ways fibers can delight today’s consumers, both online and in retail stores.

DS3 is a next generation line of 8 unique travel-friendly cleaning products for your body & home that make life easier while significantly reducing the impact on the environment. DS3 is made with fiber spinning technology that eliminates water from the final product, removing 80% weight, 70% space, & 75% emissions. One liquid-free swatch works just as well as heavy, water-based cleaning products.

Each formula contains a proprietary blend of surfactants and polymers formulated specifically for the task at hand. In some cases, we have created composite structures comprised of particles and fibers into high basis weight nonwovens. We have found this combination of particles and fibers provides a highly mass-efficient means to deliver the cleaning agents at the appropriate concentrations. In addition, it also lends itself readily to the e-commerce channel due to the compact size, lightweight and no potential for leaking. We have also found that by producing a dry form, we no longer require the use of traditional stabilizers and preservatives, which is important for many consumer segments. An open question: Are there other applications this soluble fiber platform can serve?
Superabsorbent Skin Decontamination Patch

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BACKGROUND
Wet-decontamination methods such as water cleaning are an easy, quick, and effective means to remove most chemical residues from the skin surface. However, they frequently result in stratum corneum (SC) overhydration, which can reduce skin barrier integrity and accelerate the chemical absorption–penetration process (“wash-in” effect).

AIM
To develop quick and powerful water (vehicle) and chemical absorbent-binding methodology following water washing to enhance chemical decontamination/detoxification actions and prevent or reduce the “wash-in” side effect.

EXPERIMENT
Superabsorbent polymer (SAP) fiber (and/or powder) and detoxification agents such as metal organic frameworks (MOFs) were investigated with an in vitro human skin diffusion model following dermal exposure of \textsuperscript{14}C-paraoxon and \textsuperscript{3}H-water to evaluate decontamination efficiency (absorption, retention, and penetration) and detoxification action - acetylcholinesterase activity (AChE).

RESULTS
SAP applied immediately after water-washing significantly reduced chemical content in the SC, deep skin tissue, and penetration flux when compared with control (no decon) and dry decon (no wash) groups. \textsuperscript{3}H-water content did not change in both “water wash” SAP group and “dry” control. No “wash-in” effect was observed after applications of water wash and SAP. When SAP was combined with MOFs, AChE activity was ten-times higher than that in the corresponding control.

CONCLUSION
SAP is a strong absorbent for absorbing, retaining, and possibly binding water and chemicals in solution. It also reduces the SC water content to overcome “wash-in” effect. In combination with MOFs it can extend detoxification strength. Further study is warranted to demonstrate water wash and SAP/MOF decontamination efficacy and analyze related mechanisms.
Development of Ultra, Non-swelling Microporous-type Air Permeable/Waterproof Membrane Outside/Dry Inside Outdoor Product Designed for MOFI Through PU Hybrid Dry Process

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ABSTRACT

Water proof breathable membranes have numerous applications; however, their low wear resistance and low moisture tolerance limit their outdoor use. In this study we reported Ultra-Non-Swelling Microporous Waterproof and Breathable Poly-Urethane membrane through effective dry process; Evaporation induced phase separation method (EIPS). Emulsions and additives of membrane were optimized to achieve high abrasion resistance and mechanical performance without compromising on hydrophobicity and water vapor transmission. High mechanical and physical stability are attributed to non-swelling behavior and highly crosslinked chains in microporous membrane. With optimized recipe of formulation, UN-MWBP membrane sustained a tensile load of 350 kgf/cm² and abrasion resistance of 15,000 cycles. In addition to such superior mechanical stability, the membrane showed high breathability water vapor transmission 3,000g/m²/day and hydrostatic pressure of 3,500mm H₂O. Based on such promising results, PU can compete with existing commercial membranes for their facile synthesis, low cost, non-swelling behavior, high stability and commercial scale production.

Figure 1. Ultra-Non-swelling Microporous Water-proof and Breathable Poly-Urethane membrane for outdoor applications.

ACKNOWLEDGMENT

The results of this study are supported by the cooperation technology development project between the textile and textile industries in Ministry of Trade, Industry and Energy.
Solution Blown Coaxial Fibers for Drug-carrying Scaffold Applications

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INTRODUCTION: The study aims to develop biocompatible drug-loaded core-shell nanofibers via solution blow spinning to be applied as bioactive scaffolds. As the core material, water-soluble polyvinylpyrrolidone (PVP) was used, incorporating a hydrophilic fluorescence dye, sulforhodamine B (SRB) as a model drug. As the shell material, a biodegradable poly(ε-caprolactone) (PCL) was used with and without PVP porogen. For the porogen-added shells, PVPs in two different molecular weights (1.3 MDa and 40 kDa) were emulsified and mixed in the PCL matrix. The influence of porogens in the shell on the release rate of SRB was investigated. The viability of human epidermal keratinocytes (HEK) on the core-shell fibrous mat was investigated.

EXPERIMENT: Core-shell fibers containing a model drug within the core were fabricated via solution blow spinning (SBS). PVP/PCL core-shell fibers with three different shell compositions were prepared: PCL only (sample code: CS-N), PCL with low molecular weight (40 kDa) PVP (CS-L) and PCL with high molecular weight (1.3 MDa) PVP (CS-H). Surface wettability of the web, in-vitro SRB release test, and in-vitro cell test were performed.

RESULTS AND DISCUSSION: Transmission electron microscopic (TEM) images show clear boundaries between the core (PVP) and the shell (PCL or PVP/PCL blends), confirming the core and shell construction of the fibers (Figure 1). When PVP/PCL blend solution was observed for the miscibility and phase separation, a clear separation between PVP and PCL phase is shown, indicating that two polymers are immiscible. The PVP with 40 kDa produced uniform small-sized porogens, while that with 1.3 MDa produced uneven dispersion of larger porogens. From Figure 2, all samples exhibited an initial burst release followed by a sustained release of SRB. However, the release rate of the SRB was varied depending on the composition of the fiber shell. The CS-L displayed the fastest release of SRB; ~56 % of SRB was released during the first 30 min. After the initial burst, the release rate decreased. The overall drug release of CS-H was suppressed compared to CS-L. In contrast to the fibers with PVP/PCL shell, the fiber with monolithic PCL shell (CS-N) exhibited a significantly reduced initial burst (~ 3 % release during the first 30 min). HEK cells on CS-N formed two-dimensional monolayers, whereas HEK cells tended to form spherical clusters on CS-L and CS-H. Cells in 3D construction with spherical morphology indicate the maintenance of their functionality as the native tissue. The 3D cell growth was more apparent in CS-H, indicating that the addition of hydrophilic PVP (1.3 MDa) into the PCL shell created a beneficial environment for cells to attach, migrate, and proliferate.

CONCLUSIONS: The results demonstrate that core-shell fibers afford drug-loading bioactive scaffolds for biomedical application. The hydrophilic additive (PVP) in the hydrophobic shell matrix influenced the diffusion of the incorporated molecules, acting as a porogen in the aqueous medium. The increase of surface hydrophilicity by the addition of PVP was beneficial for cell viability and proliferation. The finding of this study provides fundamental guidance on web design for optimal bioactive release and cell regeneration.
Nanofibrous Membranes Containing Tea Tree Oil and Their Antimicrobial Properties

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INTRODUCTION
Because of the recent increase in the concern regarding human well-being and environmental pollution, the present demand for and development of environmentally friendly textile products using natural extracts are highlighted. Tea tree oil, which is a natural extract from the Australian native plant *Melaleuca alternifolia* (tea tree), is widely used because it has several advantageous properties such as antimicrobial activity, biosafety, and environmental friendliness. This research investigated the incorporation of tea tree oil into a polymeric matrix via emulsion electrospinning to develop antimicrobial and environmentally friendly textile materials. Poly(vinyl alcohol) (PVA), which is known as a biodegradable polymer and used as a medical material, was combined with tea tree oil for various applications in medical and healthcare textiles.

EXPERIMENTAL
We prepared an oil-in-water type of emulsion composed of tea tree oil and aqueous PVA solution as the oil and water phases, respectively. Electrospinning was performed using a vertical electrospinning setup. Various emulsion formulations and spinning conditions were used to identify the optimum processing conditions. The morphologies of nanofibers containing tea tree oil were observed by field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM) and confocal laser scanning microscopy (CLSM). Heat treatment was used without any additional chemical methods to improve the stability of PVA-based nanocomposite fibers in water. The release profile of tea tree oil from the nanofibers was examined using a thermal extractor and thermodesorption gas chromatography with mass spectrometric detection (TDS-GC/MS). The antibacterial effects of the nanofibers against *Propionibacterium acnes* (*P. acnes*) and *Staphylococcus aureus* (*S. aureus*) were assessed.

RESULTS AND DISCUSSION
SEM images showed that bead-free nanofibers containing tea tree oil were successfully fabricated through emulsion electrospinning. We observed a well-aligned core/sheath structure of the nanofibers containing tea tree oil in the core through TEM and CLSM. Among the applied thermal treatment conditions, the suitable heat treatment condition to improve the aqueous stability of the nanocomposite fibers was at 170 °C for 1 min. We investigated the release profile of tea tree oil emitted from the core/sheath structured nanofibers over 14 days. Additionally, the effect of drying and heat treatment on the release profile was examined. The results showed that although the drying and heat treatment after electrospinning reduced the emission of volatile organic compounds, Terpinen 4-ol, an active antibacterial component in tea tree oil, was released in a sustainable manner over 14 days. Terpinen 4-ol has an antimicrobial effect on acne and helps to ease the inflammation associated with acne. The core/sheath structured nanofibers containing tea tree oil presented strong antibacterial effects against *P. acnes* and a 99 % reduction rate against *S. aureus*.

CONCLUSIONS
We optimized the concentrations and electrospinning conditions to achieve a well-aligned core/sheath structure of PVA nanofibers containing tea tree oil. Nanofibers containing Terpinen 4-ol exhibited strong antimicrobial properties against *P. acnes* and *S. aureus*, indicating that tea tree oil loaded in the fibers still retained its antimicrobial properties even after it was subjected to a high electrical voltage and thermal treatment. These results demonstrate that the nanofibrous membranes containing tea tree oil have potential for use as medical nanotextiles and healthcare textiles.
β-Cyclodextrin-included Eco-friendly Nanofibrous Membranes for Efficient Removal and Release of Methylene Blue with Good Recyclability

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Considering the excellent thermo-mechanical properties, chemical stability and low cost, biodegradable aliphatic-aromatic copolyester is an ideal matrix functionalized for capturing pollutants in wastewater. In this work, biodegradable poly((butylene succinate-co-terephthalate)-co-serinol terephthalate) (PBSST) copolyesters with amino side group (-NH2) were first synthesized through copolymerization, followed by grafting carboxymethyl-β-cyclodextrin (CM-β-CD) into PBSST molecular chains via amidation reaction. The corresponding nanofibrous membranes were then fabricated by electrospinning as adsorbents for efficiently removing cationic dye methyl blue (MB) from aqueous solutions. The nanofibrous membranes exhibited high adsorption capacity for MB with good recyclability. The adsorption performance of nanofibrous membranes was fitted well with pseudo-second-order model and Langmuir isotherm model. The maximum adsorption capacity was 543.48 mg/g for MB along with removal efficiency of 98% after five regeneration cycles, much better than many other cyclodextrin-based adsorbents and other electrospun fiber adsorbents. The adsorption mechanism was proposed with the hydrogen bonds and host-guest complexation between MB and adsorbents. The adsorbents are of high adsorption capacity, eco-friendliness, cost effectiveness and easy operation, and present a great potential in disposing printing-dying wastewater.

Fig. 1. Schematic diagram of MB adsorption on the nanofibrous membranes.

ACKNOWLEDGMENT
This research was financially supported by Natural Science Fund of Shanghai City (18ZR1400500) and Application Fundamental Projects of China National Textile and Apparel Council.
Personal Thermal Management and Cold Weather Protection via Silver Nanowire and Hydrogel Modified Textiles

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²Harvard University, Cambridge, Massachusetts, United States

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The current protective ensemble for cold weather operations is the Extended Cold Weather Clothing System (ECWCS). It is a Soldier system that provides personal insulation in temperatures ranging from 4 °C to -51 °C, but this is only achieved through the use of seven layers totaling 12.8 lbs. An ongoing need in cold weather military clothing is novel textiles that could provide protection from extreme climatic operating environments, especially arctic cold weather. The main challenges with current garment systems are overdressing, moisture from sweat, discomfort from warmth following strenuous activity or entering heater areas and loss of manual dexterity from wearing gloves and mittens. A silver nanowire (AgNW)-coated electronic textile has been developed that provides multiple protection capabilities. Different fabric materials were coated with the nanowire ink and the thermal properties were examined. Scanning electron microscopy and Fourier Transform Infrared Spectroscopy revealed a conductive network of silver nanowires (~105 nm diameter) that were highly thermally insulating with a reflectance of >24% observed for 100% cotton indicating that AgNW-cloth is an effective IR reflector suitable for personal thermal management. The results of our research suggest that AgNW coated textiles are not only highly thermal insulating because it reflects infrared radiation but it also allows heating of the textile. This nanowire-coated textile could efficiently provide protection from extreme climatic operating environments. Fundamental research is underway that examines the potential incorporation of both AgNWs and thermoresponsive hydrogels into textile based systems. Inclusion of hydrogels into the textile will help with moisture management and the absorption of sweat away from the body during operations at freezing temperatures. Three categories of hydrogels are being studied to determine feasibility: polyethylene glycol (PEG), poly(N-isopropylacrylamide) (PNIPAAm), and polyacrylic acid (PAA). Different formulations of each hydrogel in bulk or microgel form will be examined to determine morphological effects due to freezing temperatures and rehydration studies after storage of the hydrogels at below freezing temperatures. Optical microscopy, SEM, and DLS will be utilized to study hydrogel morphology and behavior at various cold temperatures.

ACKNOWLEDGMENT
We would like to thank the Natick Soldier Research, Development and Engineering Center (NSRDEC) for funding the work described in the abstract.
Fabricating Durable, Fluoride-free, Water Repellency Cotton Fabrics with CPDMS

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Fabric is dramatically increasing due to environmental and human safety concerns. The current approach to produce water repellent cotton fabrics is applying fluorocarbon compounds in topical treatments. However, the fluorocarbons employed in the water repellent treatments on cotton fabrics have environmental concerns and environmentally friendly technologies are desperately sought. Herein, we report a new approach of fabricating water repellent cotton fabrics with proper washing durability by using poly{dimethylsiloxane-co-[2-(3,4-epoxycyclohexyl)ethyl]methylsiloxane} (CPDMS) as a hydrophobic agent. Benefiting from formation of robust ether bonds between CPDMS and cellulosics, the cotton fabrics could gain promising durability for daily laundry. The resultant cotton fabrics demonstrated simultaneously desired waterproofness (hydrostatic pressure up to 22 mbar), durability (hydrostatic pressure stabled at 12 mbar after five laundry cycles), and breathability. Moreover, the CPDMS modified cotton fabrics also exhibited robust physical property with tensile strength retention up to 73%.
Effect of Moisture Content on Thermophysiological Properties of Terry Knitted Socks Followed by Thermal Resistance Comparison Among Different Skin Models

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¹Faculty of Textile Engineering, Technical University of Liberec, Czech Republic
²Faculty of Textile Technology, University of Zagreb, Croatia
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Socks comfort has great significance in our daily life. This significance even increased when we underwent a work of low or high activity. It causes the sweating of our body with different rates. In this study, terry socks with differential fibre composition were produced on the same knitting machine with homogeneous yarn linear densities and wetted to a saturated level. Then after successive intervals of conditioning, the socks are characterized by thermal resistance, thermal absorbivity and relative cooling effect in dry and wet states. Hydrophobic have best results followed by semi hydrophilic and hydrophilic. Thermal resistance in dry state measured on Thermal Foot Model has a correlation ($r^2$) of 0.64 and 0.55 is observed with Permetest and Alambeta, respectively.

OBJECTIVES
• To investigate the effect of moisture content on thermal resistance, thermal absorbivity, and relative cooling.
• Thermal resistance comparison among TFM, Alambeta, and Permetest.
• To measure the tests in extended state for real simulation of wore sock on human foot or TFM.
• Image analysis of socks with / without extension by using ImageJ software.

MATERIALS AND METHODS

Table I. Sock Samples

<table>
<thead>
<tr>
<th>Fibre Composition (%)</th>
<th>Sock Codes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton 86%, Polyester 12.74%, Elastene 1.26%</td>
<td>T1</td>
</tr>
<tr>
<td>Viscose 85%, Polyester 13.65%, Elastene 1.35%</td>
<td>T2</td>
</tr>
<tr>
<td>Polyester 98.56%, Elastene 1.44%</td>
<td>T3</td>
</tr>
<tr>
<td>Nylon 81%, Polyester 17.29%, Elastene 1.71%</td>
<td>T4</td>
</tr>
<tr>
<td>Polyproplene 83%, Polyester 15.29%, Elastene 1.71%</td>
<td>T5</td>
</tr>
<tr>
<td>Wool 89%, Polyester 10.1%, Elastene 0.99%</td>
<td>T6</td>
</tr>
<tr>
<td>Acrylic 90%, Polyester 9.1%, Elastene 0.9%</td>
<td>T7</td>
</tr>
<tr>
<td>Coolmax 85%, Polyester 13.65%, Elastene 1.35%</td>
<td>T8</td>
</tr>
<tr>
<td>Micro Nylon 79%, Polyester 19.11%, Elastene 1.89%</td>
<td>T9</td>
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</table>

Note: T2 and T8 Used Only for Thermal Resistance Comparison

Table II. Test Methods

<table>
<thead>
<tr>
<th>Testing Parameters</th>
<th>Method/Equipment</th>
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<tr>
<td>Thermal Absorbitivity</td>
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<tr>
<td>Thermal Resistance</td>
<td>Thermal Foot Model, Alambeta, Permetest</td>
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<td>Relative Cooling</td>
<td>Permetest</td>
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</table>
Table III. Thermal FM, Permetest, and Alambeta Parameters

<table>
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<tr>
<th>Parameter</th>
<th>Foot Model</th>
<th>Permetest</th>
<th>Alambeta</th>
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<td>Relative Humidity</td>
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<td>65 ± 5 (%)</td>
<td>65 ± 5 (%)</td>
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<td>35 (°C)</td>
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<tr>
<td>Chamber Door</td>
<td>Open</td>
<td>Closed</td>
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<tr>
<td>Membrane</td>
<td>FM Glued with a Semi-permeable Membrane</td>
<td>Permetest Already has GORE-TEX Membrane. A Polythene Sheet Used to Block Moisture Permeation.</td>
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<tr>
<td>Isothermal Conditions</td>
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<td>Not Applicable</td>
<td>Measuring Head is Covered by Isothermal Ring</td>
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RESULTS AND DISCUSSION

Thermophysiological Properties in Wet State

Fig. 1. Effect of moisture content on thermal resistance.

Fig. 2. Effect of moisture content on thermal absorbitivity.

Fig. 3. Effect of moisture content on relative cooling (%).

Thermal Resistance Comparison with Different Skin Models

Fig. 4. Sample preparation for measurement in extended state.

Thermal Resistance (m².K/W) Correlation

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Foot Model</th>
<th>Permetest</th>
<th>Alambeta</th>
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<tr>
<td>Thermal Resistance (m².K/W)</td>
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<tr>
<td>[Graph showing thermal resistance comparison]</td>
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Thermal Resistance (m².K/W) Correlation

<table>
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<th>Foot Model</th>
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<th>Alambeta</th>
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<td>Thermal Resistance (m².K/W)</td>
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<tr>
<td>[Graph showing correlation]</td>
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Fig. 5. Thermal resistance comparison.
Image Analysis for Porous Area (%) by ImageJ

Fig. 6. Image analysis.

Table IV. Image Analysis Data

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<th>Sock Code</th>
<th>Area</th>
<th>Mean</th>
<th>Min</th>
<th>Max</th>
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<th>MinThr</th>
<th>MaxThr</th>
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CONCLUSION
A higher value of moisture leads to decrease the thermal resistance while relative cooling effect and thermal absorptivity increase to a large extent. Every sock follows a different trend line while tested after consecutive conditioning intervals as shown in the above figures. Only sample T5 could resume its thermal resistance after 6 hours of conditioning. In the case of thermal absorptivity and relative cooling T1 and T2 is the worst one. Thermal resistance measured in an extended state by Alambeta and Permetest is comparable with Rct (m².K/W) measured by thermal Foot Model with the coefficient of determination (R²) of 0.55 and 0.64 respectively. Image analysis results show that socks should be tested in extended state for real simulation of wearing.

ACKNOWLEDGMENT
This work is supported by the Ministry of Education of the Czech Republic within the SGS21251 at the Technical University of Liberec.
Development of a Conformal Textile Antenna for Thermotherapy

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Hyperthermia therapy is a cancer treatment, where body tissue is exposed to a mild heat (typically within 39 ~ 45 °C) to deactivate and damage cancer cells. Heat produced during hyperthermia therapy can also be beneficial to promote the effects of conventional cancer treatments such as chemotherapy and radiotherapy. The current challenges of hyperthermia therapy include development of a patient-friendly heating technique to enable a longer treatment duration for a more effective treatment.

The objective of this research is to present a conformal patch antenna fabricated with textile materials for a comfortable hyperthermia device in breast cancer treatment. The proposed textile antenna follows a female breast contour and consists of copper-coated woven polyester for a radiating patch and a ground plane and woven polyester for a substrate and a padding. The dimensions of the textile antenna were optimized using a 3D full-wave electromagnetic (EM) simulation software.

As a model study, EM waves generated by the single antenna were calculated, and the specific absorption rate (SAR) was simulated by incorporating a female breast phantom with an input power of 1 W (Figure 1(a)). Thermal simulations were also performed to estimate temperature increments over time (Figure 1(b)). Finally, measurements were followed by physical prototyping of the textile antenna and breast phantom under the same conditions to validate simulated results at the measurement depths of 5 mm and 15 mm in tissue (Figure 1(c)).

![Figure 1](image)

Figure 1. (a) Simulated SAR distribution in a breast phantom, (b) simulated temperature increment after 15 minutes of heating, and (c) simulated and measured temperature increments at depths of 5 mm and 15 mm.

From simulations, we observed temperature rises by 6.7 °C and 3.5 °C at 5 mm and 15 mm depth, respectively, after 15 minutes of heating. On the other hand, the measurements showed that temperature rises at these locations were 3.3 °C and 1.9 °C, respectively. The measured temperature increments were smaller than the expected values most likely due to heat leakage into the ambient air and limited fabrication accuracy. It would be possible to engineer the thermal effect by increasing the input power and/or adding a couple of patch antennas. The research demonstrated that hyperthermia therapy could be administered in a wearable form with the proposed textile patch antenna.

ACKNOWLEDGMENT

The authors would like to thank Dr. Jacob J. Adams, Mr. Vivek T. Bharambe, and Mr. Bill Zhou from the Department of Electrical and Computer Engineering at North Carolina State University for their support during the EM simulations and the temperature measurements.
Plasma Surface Modification of Electrospun Poly(lactic acid) (PLA) Nanofibers for Protein Immobilization

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In this work, plasma treatment was used to functionalize the surface of electrospun poly(lactic acid) (PLA) nanofibers for proteins immobilization. The effect of various plasma treatment parameters such as time and voltage on the chemical structures of the nanofibers was examined. Methylene blue adsorption test was used to characterize the surface chemistry of the nanofibers. Also, protein binding to the nanofibers surface was examined by static and dynamic adsorption experiments. BSA was used as a model protein for this study. Oxygen plasma treatment effectively created carboxylic acid functional groups on the surface of the PLA fibers. The data also showed that a dynamic binding of protein to PLA via bioconjugation chemistry yielded the best results in terms of maximizing specific covalent binding and minimizing nonspecific adsorption. Covalent binding of proteins on nanofibers can allow for high sensitivity biosensing thanks to high surface area and porosity of nanofiber fabrics. The obtained results widen the application of PLA nanofibers in biosensor and biomedical products.

Figure 1. Confocal fluorescent images of PLA (A); PLA treated with plasma, Sulfo-NHS, and dynamic BSA-FITC binding (B); PLA treated with plasma and dynamic BSA-FITC binding (C); PLA treated only with dynamic BSA-FITC binding (D).

ACKNOWLEDGMENT
The authors thank Cornell University for letting us use their testing facilities.
Surface Segregation of Metal Oxide Nanoparticles in Polypropylene Fibers and Films

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Metal oxide nanoparticle applications include catalysis, hazardous gas adsorption and optoelectronics. Metal oxide-decorated fibers and fabrics have potential uses that include chemical-biological toxin degradation, gas filtration and chemical sensing. The goal of the present work is to investigate parameters that affect surface segregation of metal oxide nanoparticles in polymeric films and fibers. Of particular interest is the development of methods for preparing surface-active metal oxide nanoparticles from melt-extruded blends of polymers/metal oxides. Fundamental questions include how the molecular weight and chemical structure of the polymer, along with the size and shape of the nanoparticles affect surface segregation. Initial studies have focused on polypropylene (PP) and zinc oxide nanoparticles (nano-ZnO). Annealing of PP films and fibers containing 1-3 wt. percent of nano-ZnO at various temperatures in air and in ultra-high vacuum (UHV) has been performed to affect surface segregation of the nanoparticles. Cross sectional images of annealed fibers indicate that surface segregation of the nanoparticles occurs, but only in the near-surface region. Energy dispersive X-ray spectroscopy and X-ray photoelectron spectroscopy have been employed to obtain quantitative elemental composition of the surface of annealed fibers and films. It is found that surface enrichment of ZnO nanoparticles is affected by various parameters, including the presence of adsorbed polar groups (e.g., carbonyl groups) and the Mw of the PP. Annealing oligomer and polymer films in air leads to surface segregation of nano-ZnO, with partial oxidation of the PP on the surface. Ozone exposure introduces polar groups on the surface of spin-cast films, and heating ozone-treated films in UHV leads to varying degrees of surface enrichment, depending on the polymer's molecular weight. Confirmation of the surface activity of the segregated nano-ZnO has been performed using confocal fluorescence microscopy of fibers exposed to a fluorescent dye containing a functional group that selectively reacts with the nano-ZnO.
An Experimental Study on Optimal Washing Conditions for Sustainable Laundry

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INTRODUCTION

In recent years, there are increasing interest and study to improve the sustainability of textiles products in all stages of production, distribution, use & care, and disposal. The use & care stage of a textile product impacts on its sustainability more significantly, than any one stage of the product’s life cycle, due to repeated washing and drying. This study aimed to determine efficient washing conditions, with high detergency, to reduce energy consumption from excessive washing and improve the sustainability of the washing stage.

EXPERIMENTAL

Detergency was measured at various washing temperatures (20°C, 40°C, and 60°C) and times (10 min, 20 min, and 30 min) using standardized soiled fabrics, i.e., 100% cotton, polyester/cotton (65/35%), and 100% polyester woven fabric soiled with pigment/sebum, carbon black/mineral oil, soot/mineral oil, cocoa, and red wine. The washing method such as detergent, detergent concentration and measurement of reflectance was basically in accordance with IEC 60456 standard.

RESULTS AND DISCUSSION

The detergency at the washing condition of 20 ℃ and 30 min was higher than that at 40 ℃ and 10 min. Also, the detergency at the condition of 40 ℃ and 30 min was higher than that at 60 ℃ and 10 minutes (Figure 1).

This may be because the reduced washing effect at low washing temperatures was complemented by the increased mechanical action over the long time. Based on the drum washing machine, additional energy of 1541 Wh is needed to raise the washing temperature from 20°C to 60°C, whereas 20 Wh of energy is required to increase the washing time from 10 minutes to 30 minutes. Therefore, it is expected that the energy consumption of the washing machine can be reduced by setting the washing condition with a low washing temperature and a long washing time.

In comparison of detergency of standardized soiled fabrics, i.e., 100% cotton, polyester/cotton (65/35%), and 100% polyester woven fabric, it was found that the washing temperature and time having the same detergency were different from each other. In comparison of the detergency according to the type of soils, the washing temperature and time having the same detergency were also different from each other.

Figure 1. Detergency of cotton fabric(a), Cotton/PET 65/35(b), and PET fabric(c) according to washing conditions.

CONCLUSION

From the results that the optimum washing temperature and time vary according to the type of fibers and soils, that energy or detergent have been consumed more than necessary in actual laundry. The sorting of laundry was confirmed to be sustainable washing method to prevent excessive washing and reduce energy waste. These results of this study provide empirical basis on sustainable clothing laundry and care.
Protonation of Cellulose Nanofibril Aerogels: Synergistic Enhancement of Amphiphilic Superabsorbency and Immersed Stability

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Super ultra-light weight (5.0 mg cm$^{-3}$) and ultra-porous (99.7%) amphiphilic aerogels with highly interconnected and open-cell pore morphology to absorb 190, 133, and 227 g g$^{-1}$ water, decane, and chloroform/dried mass, respectively, were fabricated from self-assembling of 0.6 wt% cellulose nanofibril (CNFs) aqueous suspension as effected by varying the extent of protonation on C6 surface carboxyls under facile slow-freezing and freeze-drying. The same TEMPO oxidized and mechanically blended CNFs with identical geometries and level of oxidation had their surface carboxyls protonated to 11, 46 and 100% carboxylic acid. At 0.6 wt%, TEMPO oxidized CNFs of ultrathin diameter (1-2 nm), high aspect ratios (several hundreds) and abundant polar surface hydroxyls and carboxyls behaved similar to thixotropic aqueous soluble polymers until freezing (-20 °C, 15h) and freeze-drying (-47 °C, 0.05 mbar, 4 days). Upon freeze-drying, the spaces once occupied by ice crystals were well preserved to derive macroporous aerogels with uniform honeycomb-like cellular pore morphology, though becoming more highly interconnected and open-cell with increasing protonation. The conversion of sodium carboxylates to carboxylic acids on TEMPO-CNFS prior to fabrication into aerogel conferred extraordinary enhancement of aerogel density (8.1 to 5.0 mg cm$^{-3}$), porosity (99.5 to 99.7%), and volume (1.0 to 1.3 cm$^3$) to allow improved accessibility and interconnectivity of pore morphology, accounting for 91, 88, and 82% of the theoretical absorption capacity of water, decane, and chloroform. While cellular walls became thinner (522 vs. 825 nm) and the diameter of macropores enlarged (228 vs. 207 µm), protonation also proved to enhance the long-term immersed stability of aerogels in aqueous media, including preservation of dimensional integrity and water-activated shape recovery. Aerogels also showed superior long-term immersed stability in acidic aqueous media, as well as nonpolar and aliphatic decane. In addition, the interfacial properties of borosilicate glass and polypropylene tube molds showed important effects on aerogel morphology and surface wetting behavior to be crucial considerations to aerogel manufacturing.

KEYWORDS: Cellulose nanofibrils, aerogels, surface carboxyl protonation, amphiphilic, superabsorbents, self-assembly, immersive stability, TEMPO-mediated oxidation.

REFERENCES
To evaluate compatibility between a substrate and a thermoplastic polymer, the established methodology is to estimate their surface composition in terms of surface energy components, utilizing the results of contact angle measurements of probe liquids onto substrate and polymer surfaces at room temperature. Using this methodology, polymer surfaces are studied in solid state, however, during spreading of polymers on a substrate, polymers are in molten state and at high temperature, having different surface energies and more complex polymer/substrate interactions due to polymer chain mobility.

This poster presents a model study with practical relevance to predict polymer/substrate compatibility including contact angle measurements at high temperature directly performed between molten thermoplastics; polypropylene (PP), polyvinylidene fluoride (PVDF) and maleic anhydride-grafted polypropylene (MAPP), on smooth glass fibers and plates. The values of total surface energy of thermoplastics at high temperature (260°C) are down to 57% of that measured at room temperature, which has a strong influence on the wetting prediction. Surface energies of both the polymer and the substrate were found not to be the only factor controlling the wetting behaviour of molten polymers and the level of adhesion with the substrate, but also some intrinsic characteristics of the polymer melt play a role. We also observed that the wetting behaviour of molten MAPP is affected by the maleic anhydride (MA) content, demonstrating dramatically different results to room temperature measurements, which is suspected to be due to the formation of covalent bonds of MA groups with the glass surface enhancing the interface strength beyond the shear strength of MAPP.

Figure 1: Snapshots illustrating the spreading of a PP drop on a glass slide at 220°C (Left) and the corresponding contact angle, volume, and radius variations versus time (right).

Figure 2: MAPP polymer drop after cooling down to room temperature, where fragments on the glass surface of the fractured polymer matrix can be observed (red arrows).
Fumigants are a group of highly toxic vaporous pesticides that are commonly used in agriculture productions to control the growth of pests in the soil for maintaining a good production yield of crops such as strawberry in California, US. The high vapor pressure of fumigants makes them readily emit into the atmosphere and diffuse to vicinal areas, becoming a serious hazardous concern to farm workers and residents, especially to children. The development of a being easy-to-use, portable and cost-effective sensor system is urgent to protect farm workers and residents from the over-exposure of these acute toxicants. While current detection methods are mostly instrument-based and difficult real-time monitoring of the hazardous chemicals, we have been focused on the development of colorimetric sensors based on fibrous materials to detect ultra-low concentrations of fumigants in the air. The poisoning or detoxification effects of various toxicants in the human body that can generate color changes were applied as the detection reaction with the merits of a fast response time and ultra-high sensitivity. For instance, biological thiols and 4-(p-nitrobenzyl) pyridine were studied as the sensing agents to produce color signals according to different fumigants with a concentration range from ppm to ppb level. In order to achieve ultrasensitive detections of vaporous fumigants, different fiber-based materials with various specific surface areas were developed as the indispensable sensor matrix to maximize the vapor adsorption, thereby enriched the fumigant concentration on the sensor surface to kinetically accelerate the color change and significantly lower the detection limit. In this presentation, the optimization of the color generation

ACKNOWLEDGMENT
Department of Pesticide Regulation of California EPA financially supports the research.
Spider dragline silk is a spectacular biopolymeric fiber with mechanical properties stronger than steel and tougher than Kevlar. It can be softened, contracted and made stiff at different conditions that helps the fiber to function properly. Supercontraction is one of the characteristic properties of the dragline silk where the unrestrained fiber can contract up to 50% of the original length to generate ample stress under influence of humidity. Additionally, the fiber can also exhibit cyclic repeatable contraction when re-extended, dried and exposed to humidity. Spiders are cannibalistic in nature and they can’t be farmed like silkworm to harvest their silk. To overcome this limitation alternative methods using genetic engineering has been applied to produce recombinant spider silk proteins. Wet spinning methods using non-miscible solvent as coagulant has been widely adopted to spin recombinant spidroins into fiber. The use of dehydrating coagulants replaces the bound water molecules in the protein and exposes hydrophobic side chains to surface by flexing hydrophilic chain. This restricts the mobility of water molecules to penetrate inside the disordered semi-amorphous regions to initiate humidity response similar to natural fibers. Recent advancements in understanding of the principles involved in natural spinning have encouraged development of specialized artificial spinning methods that tries to apply chemical and physical processes found in nature. In this work, we had studied biomimetic spinning of recombinant spider dragline silk protein to explore their response to their humidity. Chimeric recombinant spider dragline silk proteins MaSp1 and MaSp2 are expressed in E. Coli and spun into fibers mimicking physiological conditions in spider spinning duct. Engineered NT & CT of spider silk proteins initiate lock-trigger mechanism to form fiber when exposed in pH and ion gradient. Interestingly, biomimetically spun fibers using ion-exchange strategy demonstrated cyclic contraction in response to humidity. Furthermore, the fibers were able to memorize the stress applied to them that can be retrieved upon repeated wetting and drying under controlled conditions. This smart behavior of spider silk fibers shows potential future in various applications namely sensors/ actuators and other smart biomedical devices.
Cellulose is a renewable source that presents many benefits such as good mechanical properties, low environmental impact, ease of manipulation, availability and low cost, among others. Due to these properties, cellulose fibers have been largely studied as reinforcements for composites, both polymeric and ceramic, being the latter less common. Nonetheless, the use of such reinforcements in cementitious matrices presents a challenge since the alkalinity of the cement-curing process degrades the reinforcement, leading to composite materials with poor durability. In this sense, most research in these materials has been focused on increasing their durability to wet/dry cycling. Several strategies have been considered to that purpose, such as fiber treatment, matrix selection, composite processing, and reinforcement size and structure.

On the other hand, cellulose-based reinforcements can be added in cementitious composites at any level: macro- (fabrics or fibers), micro- (pulps and microfibrillated fibers), and nano- (nanofibers or cellulose nanocrystals). Although the use of pulps and microfibrillated fibers has shown to improve the mechanical performance of cementitious composites, even greater strength and ductility can be achieved at the macroscale with the use of nonwoven fabrics while producing lightweight materials. The addition of low amounts (< 1 wt.%) of nanocelluloses can even produce a modification of the curing process, leading to more durable cement composites.

In this work, the results concerning to the reinforcement of calcium aluminate cement (CAC) pastes with nonwoven fabrics on the one hand and nanocelluloses on the other are presented, covering in each case a combination of few of the aforementioned strategies, and showing the influence of each approach in the final durability to accelerated aging of the final material.

ACKNOWLEDGMENT
This work was financially supported by the Ministerio de Economía y Competitividad, MINECO, Government of Spain [project BIA2014-59399-R].
Pressure-responsive PET Fabrics via Constructing Conductive Wrinkles at Room Temperature

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Thanks to the booming development of wearable electronics, finding a facile approach to integrate electronics to textiles becomes attractive. However, many disadvantages such as trivial fabrication steps, limitations of textile substrate types and unavoidable damage to textiles’ natural properties, have hampered the development of wearable electronics. In this work, pressure responsive PET fabrics were produced through a facile fabrication of conductive PET fabrics. Firstly, fine conductive adhesives (CA) with good printing performance were prepared by utilizing alcohols-assisted dispersion to prevent Ag NPs’ aggregation tendency in waterborne polyurethane (WPU). Then, screen printing on PET fabrics and subsequently treated with solvent system that consisted diallyldimethylammonium chloride (DADMAC), dichloromethane (DCM) and ethanol to construct conductive wrinkles at room temperature. With the help of WPU’s swelling, the conductive path was generated by Ag NPs spontaneous aggregation and the as-prepared conductive PET fabrics demonstrated outstanding electric conductivity (0.01 Ω·m). Finally a simple self-assembly PET fabrics pressure sensor based on the conductive PET fabrics exhibited superior performance, including the synchronous response to thumb pressing (frequency was 0.5 Hz), precisely distinction current of different weights, decent bending stability, fast response (63 ms) and relaxation time (23 ms), simultaneously possessing advantages of low cost and facile fabrication process. Based on its superior performance, the PET fabric sensor was developed to monitor the human fist behavior at different frequency (0.25 Hz and 1 Hz) and its good real-time response current, meeting the potential application requirements in wearable electronics and “smart textiles” for monitoring human’s movement behavior.

KEYWORDS: Conductive adhesive; PET fabrics; conductive wrinkles; room temperature; pressure sensor.
Ultrasonic Effect on Morphology of Silk Fibroin Microspheres Prepared by Emulsion Cross-linking Process

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Silk fibroin blank microspheres prepared by emulsion cross-linking process are prone to aggregate due to the large particle size. In order to solve this problem, ultrasonic treatment is employed during the emulsion cross-linking process to reduce the particle size. The particle size distribution of microspheres was measured by Malvin NanoZS90 laser particle size analyzer, meanwhile, scanning electron microscopy (SEM) was used to detect the morphology. The results showed that without the ultrasonic effect the microspheres prepared via emulsion cross-linking process had the average particle size of 15.08 μm with the standard deviation (SD) value of 0.515, and the aggregation was very obvious as in Fig. 1. However, after ultrasonic assistance, the average particle size decreased with the increasing of ultrasonic frequency and ultrasonic power. The particle size of the microspheres prepared with the ultrasonic at 45 KHz and 100 W reduced to one third of the original. The SD value of microspheres was also decreased, indicating a more uniform microspheres size. As shown in Fig. 2, the effect of ultrasonic assistance is quite dramatic and the aggregation was effectively.

(1) The particle size distribution spectra and microscopy of microspheres prepared via emulsion cross-linking process.

(2) The particle size distribution spectra and microscopy of microspheres prepared via emulsion cross-linking process assisted with ultrasound.

ACKNOWLEDGMENT
The work was supported by National Natural Science Foundation of China (51503002), Natural Science Foundation of Anhui province (1608085QB43), Key Project of Natural Science Research of Anhui Province (KJ2016A796) and the young and middle-aged top-notch talent project of Anhui Polytechnic University (2016BJRC007).
The development of methodologies to fabricate textiles covered with metal oxide nanoparticles has potential benefits for Soldier protection. Metal oxide nanoparticles offer substantial potential as photocatalytic, sensing and smart materials; however, a significant challenge has been to incorporate them into a cost effective, scalable matrix that facilitates accessibility for numerous applications. Our work centers on employing standard melt extrusion principles for incorporating metallic nanoparticles into polymer matrices to create functional fibers. The fibers can be knitted or woven into swatch materials, and post-treated if desired, to expose the metallic nanoparticles at the surface for enhanced accessibility and reactivity. Here, we describe the use of a bi-component melt extrusion system, incorporating a sheath/core configuration to produce polypropylene (PP) fiber in order to fabricate swatch materials. The first method consists of melt extruding a blend of metal oxide nanoparticles and PP into fibers that are subsequently knitted into swatch materials. This method was evaluated for zinc oxide (ZnO) nanospheres and nanorods. X-ray photoelectron spectroscopy (XPS) and field emission – scanning electron microscopy (FE-SEM) showed that the nanoparticles were in the near-surface region as well as in the bulk of the fibers. However, the particles were shown to be photo-catalytically inactive, likely due to site blocking by PP or processing additives, such as fiber lubricants. The second method consists of plasma treating virgin PP swatches in air, immersing them in metal oxide colloidal suspensions, ultra-sonicating them to remove loose nanoparticles and drying the swatches. XPS confirmed that plasma treatment forms carboxylate groups on the surface that bond the metal oxide nanoparticles to the swatch. This method was used to functionalize swatches with ZnO and titanium dioxide (TiO2) nanospheres and ZnO nanorods. Metal oxide functionalization was found to induce dramatic changes in the thermal decomposition temperature of the PP swatch and in the water contact angle, rendering the swatch hydrophobic and producing photo-catalytically active PP fabrics. Photoluminescence of the ZnO nanoparticle swatches was measured and shown to change upon exposure to sulfur dioxide, thus demonstrating the potential of metal oxide functionalized swatches as a gas sensing platform.
Highly Fluorescent Luminescent Carbonized Polymer Dots Prepared via a Stabilizer-free Dispersion Synthesis Method

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In recent years, carbon dots (CD) as new fluorescent nanomaterials have attracted much attention due to their unique properties. Herein, highly fluorescent carbonized polymer dots (CPDs) were prepared following a hydrothermal synthesis method by using citric acid as a carbon source and tris(2-aminoethyl)amine as a nitrogen source in the presence of pure water under relatively low temperature. The CPDs could emit blue fluorescence with a maximum quantum yield (QY) of 45.3%. Compared with ammonium hydroxide and urea as different nitrogen sources, CPDs prepared from using tris(2-aminoethyl)amine showed better stability. Results indicated that the tris(2-aminoethyl) amine could not only passivate the surface of CPDs, but also participate in the formation process of CPDs. The CPDs showed the characters of excitation-dependent, quenching when complexed with Fe3+, Cu2+, Ag+, which can be used as an efficient metal ions identification sensor.

KEYWORDS: Fluorescent; carbonized polymer dots; hydrothermal synthesis; quenching.
Functional Peptide-based Materials for Biomedical Applications

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It is recognized that drug and gene delivery systems have numerous advantages for controlled drug release, including prolonged duration time, reduced side effect, improved drug bioactivity and enhanced therapeutic efficiency. To enhance the bioavailability of therapeutics and to deliver therapeutic agents to particular tissues and cells, the ideal drug and gene delivery systems should have desired functional properties, including target ability and stimuli responsibility.

Our studies focused on the functional peptides for biomedical applications. For example, various targeting peptides were used for targeted drug/gene delivery. Enzyme sensitive peptide was used to give the delivery systems with protease responsibility. To achieve enhanced synergy effects, double/multiple stimuli-responsive systems have also been developed. The physical/chemical properties and drug/gene delivery behaviors in vitro or in vivo of peptide based delivery systems were investigated in detail. The results showed the drug and gene delivery systems exhibited improved drug controlled release and enhanced gene transfection efficiency.
Development of Highly Sensitive Nanofibrous Portable Biosensors for Detection of Trace Amount of Toxicants

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Conventional toxin detection methods, including liquid chromatography (LC) or enzyme linked immunosorbent assay (ELISA), are instrument-dependent, time-consuming, and complicated-operation, limiting their applications in onsite and instant detection. Here, we report fabrication of a portable, eye-distinguishable, and highly sensitive biosensor by applying indirect competitive immunoassay (ic-ELISA) on nanofibrous membranes. First, optimized morphological structures of the membranes were obtained by electrospinning poly (vinyl alcohol –co-ethylene) with controls; then the surface structures of the membranes were chemically modified with the morphology intact; afterward, antibodies were immobilized onto the membranes through covalent bonding with active protein conformations retained. Biological activities of the antibodies on the nanofibers were investigated under different modification conditions. Colorimetric signals of trace analytes can be observed on the membranes by naked eyes within 30 mins, and the Limits of detections (LOD) of the analytes were at 0.05ppb for chloramphenicol (CAP) and 0.1ppb for amatoxin. The successful design and fabrication of these biosensor materials provide new insights into the development of portable and practical sensors for toxicants in foods and environment.