Appendix A

2007 S-1026 Regional Research Report
"Textile Materials and Technologies Addressing Energy, Health and Other National Security Issues"

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Objective 1 – To create barrier fabrics with novel finishes and processes for protection against biological threats.

Electrospinning apparatus was installed during 2009 and projects evaluated the impact of polymer and processing variables have been completed. The first of these involved elecrtospinning of Poly(ε – caprolactone) (PCL) with ampicillin and the second was electrospinning of PCL with Chitosan. In the first project, polymer molecular weight (MW), ampicillin concentration, polymer viscosity, polymer feed rate, spinning voltage, spinning distance, and fiber collector rotating speed were independent variables researched on their influence on spinnability, fiber diameter, fiber surface morphology, and yarn tensile properties. Results showed that, within spinnable range, smaller voltage which results in larger fiber diameter leads to larger yarn tensile tenacity. In the next project, the first step was to find a proper solvent to dissolve PCL and chitosan mixture to achieve a homogeneous polymer solution. Acetic acid, formic acid, chloroform, and their mixtures were tried and chloroform was chosen. Solutions with different polymer concentration, chitosan concentration, and solution viscosity were spun to obtain continuous fibers. Continuous fibers were collected from spinning solutions of 14% PCL plus chitosan from 2.5% to 12% concentration. By studying the fiber diameters, it was found that the fiber collector rotating speed had the largest influence among all the variables studied, including voltage, distance, and feed rate.

Objective 3 *To develop new bio-based textile products/processes to replace petrol-based materials*

A three-year, multistate and multidisciplinary research project, funded by NIFA Specialty Crops Research Initiative (number 2009-02484), is focusing on the evaluation of experimental and currently available biodegradable mulch (BDM) products, in both high tunnels and open field tomato production systems at three sites in the US (TN, TX, WA). The results of the degradation of the BDM's during Year 1 of this study, completed in 2010, are presented here.

Materials & Methods: The experimental design for this study was 5 x 2 x 3 with five mulches, two field conditions, and three field sites. The mulches, included an experimental white spunbond (SB) poly(lactic acid) (PLA) product, two commercially available BDM starch-based black film products (BioAgri and BioTelo), a black cellulose-based product (WeedGuardPlus) and conventional black polyethylene (PE) film (Pliant Corporation).

All products evaluated in this study, both BDM and non-BDM mulches, showed a loss of physical properties (strength and elongation) after exposure in high tunnel and open field environments. There were changes in MW indicating degradation of the products. SEM micrographs showed cracks and pitting of the starch based BDM's. There were no other patterns with regard to location or environment that could be determined based on mulch type. In comparing the influence of open field and high tunnel environments on strength loss, almost two-thirds of the samples had increased strength loss when in the open field. This result was expected as sunlight intensity and other weathering factors

are thought to increase degradation. Cellulose had the greatest loss of strength in the open field indicating that it was more readily degraded by sunlight and other weather factors when compared with the starch-based mulch products, SB PLA and PE film. The SB PLA did not show increased degradation compared with other BDMs studied here. Future work to evaluating a SB product with PLA of lower MW, finer fibers in the SB structure, and colored with black pigment is planned.

Publications:

Liu, H. and Leonas, K.K. Weight loss and morphology changes of electrospun poly(ε caprolactone) yarns during in vitro degradation. Fibers and Polymers, Vol. 5, No. 4, pp 10-19, 2010.

Liu, H., Leonas K. K., and Zhao,Y. Antimicrobial properties and release profile of ampicillin from electrospun polycaprolactone nanofiber yarns. Journal of Engineered Fiber and Fabrics; Vol. 11, No. 7, pp 1024-1031, 2010.

Miles, C., Inglis, D., Leonas, K, Moore-Kucera, J., Wxzelaki, A, Wallace, R., Hayes, D., Wadsworth, L., **"**Evaluating potential biodegradable mulches for high tunnel and field vegetable production." 2010 Agricultural Plastics Congress Proceedings, July 31 – August 1, 2010, Palm Desert, CA

Appendix B

S-1026 Multi-State Research progress Report
Georgia
June 1, 2010- March 1, 2011

Microencapsulation Technology Using Essential Oils to Produce Smart Textile Functionalities that Improve Human Health

PI: Suraj Sharma, Textiles, Merchandising and Interiors, University of Georgia, Athens, GA

Multi-State Objective:
This project supports Objective 1 of the multistate project outline for S1026: Textile Materials and Technologies Addressing Energy, Health and Other National Security Issues. The objective is to create barrier fabrics, with novel finishes and processes for protection against biological threats.

Progress Report:
The long-term goal of this research is to expand the knowledge base of microcapsules and to develop smart textiles that will thwart biohazards and improve human health. The objectives of this research project are:

1. Production of protein-based microcapsules loaded with plant-source active agents
2. Attach and test the efficacy of microcapsules for antimicrobial and antiallergenic effects

To accomplish these objectives, first study was conducted to identify the effectiveness of various essential oils. Clove and Thyme oils were found to be effective acaricides. Subsequently, simple coacervation method was used to develop microcapsules loaded with these oils. Factorial design was used to determine the effect of main variables (type of oil, oil to gelatin ratio, stirring speed and surfactant) on the size of microcapsules. Results showed that stirring speed and type of oil (viscosity effect) were the critical variables to control the size of microcapsules. In addition, it was observed that vacuum drying can be an effective process to prevent aggregation of microcapsules and attach mechanically to the fabric without any binder. In another study, through bioassay against HDM (Der.f), we observed that clove containing phenolic compound (85% eugenol) was more effective in reducing the number of house dust mites than thyme oil. Further study using rosemary and peppermint essential oils is undergoing and fumigation method promised their effect as an acaricides.
Impact: This study showed that coacervation method is useful to produce thermally stable microcapsules from natural-based materials. Microencapsulation approach seems cost effective and environmentally friendly to provide durable, long-term health effects of cotton textiles. Essential oils are obviously the alternative and effective source as acaricides in terms of their impact on environment and human health.

Presentations/Publications/Thesis:

Kim, Joo-Ran and Sharma, Suraj (2010). *Microencapsulation Technology Using Essential Oils to Produce Acaricides Against House Dust Mites*, Herman and Myrtle Goldstein Student paper competition, AATCC's International Conference, Atlanta, GA

Joo Ran Kim, M.S. in Textiles, Merchandising and Interiors with an emphasis in Textile Science – August 2010; Thesis Title: Microencapsulation Technology Using Essential Oils to Produce Acaricides against House Dust Mites

Kim, Joo Ran; *__Sharma, Suraj__. Microencapsulation Technology Using Essential Oils to Produce Durable Textile

Functionalities. Abstracts of Papers, 2011 Beltwide Cotton Conferences, Atlanta, GA, United States, January 4-7, 2011.

Kim, Joo Ran, and **Sharma, Suraj**, Acaricidal Activities Of Clove Bud Oil And Red Thyme Oil Using Microencapsulation Against House Dust Mites, *Journal of Microencapsulation*, 29(1):82-91, 2011.

Appendix C

2011 PROGRESS REPORT
S-1026 Multi-State Research Project
May 16, 2010-April 20 2011

PROJECT: **S1026 Textile Materials and Technologies Addressing Energy, Health and Other National Security Issues**

PRINCIPAL INVESTIGATOR: Dr. Yiqi Yang
Dept. of Textiles, Clothing and Design, &
Dept. of Biological Systems Engineering
University of Nebraska-Lincoln

PROGRESS REPORT:

Multi-State Project Objective: 3) To develop new bio-based textile products/processes to replace petrol-based materials

Nebraska Sub-Objective: b. Develop and optimize the appropriate PLA process and use conditions.

d. Develop new bio-based non-woven materials

Outputs

Activities
Our research on PLA has been broadened to the comparison of the properties and performance of PLA and PHBV. The main focus is on the applications of these PHAs as tissue engineering scaffolds.

Our research on new bio-based non-woven materials has been focused on the development of ultrafine fiber mats from zein for medical applications. More specifically, we have produced biocompatible and water stable zein nonwovens for drug delivery and tissue engineering. We have developed biocomposites using fiber mats from jute and switchgrass as reinforcements and plant proteins, such as soyproteins and wheat gluten as the matrix materials. We have also identified nontraditional natural protein nonwovens made by insects for potential applications in biomedical engineering and other arenas.

Products

We have made novel biocomposites reinforced with nonwoven mats of fibers from agricultural wastes such as switchgrass stems, and matrix materials such as wheat gluten and soyproteins. We have developed novel nonwoven scaffolds from zein for tissue engineering applications. We have discovered the potential application of new natural silk fibers from insects with properties similar to common silks currently available on market.

No graduated MS or PhD students

Outcomes

Change in knowledge
We, the first time in the world, have demonstrated that agricultural wastes such as switchgrass stems could be used as reinforcement materials for thermoplastic composites, especially for the light-weight composites.

We, the first time in the world, have developed 100% biocomposites using cellulosic fibers as reinforcement materials and plant proteins as matrix without using any chemicals as plasticizer.

We have proven that plant protein nonwoven scaffolds are excellent materials for cell culture and for drug controlled release.

We have demonstrated that silk nonwoven webs made by insects have properties very similar to commonly used silk fibers and has potential to be commercially used as new silk fibers.

Change in actions and conditions
Our findings continue to provide important information to researchers and industries for the selecting the appropriate renewable resources and application conditions for the development of fibrous materials and non-woven technologies in textiles, composites and medical industries. Our researches on biofibers, in general, allows us the leading position in the area. This provides us with funds for our graduate education and for our continuing researches. Our researches attract industries to contact UNL's Office of Technology Development for technology transfers.

Our researches on the biofibers provide opportunities for Nebraska to add billions of dollars to its economy, add jobs, and for the textile and materials industries to decrease their dependence on petroleum, and increase their sustainability.

Importance of the funds
The funds from this project were used to pay the students and research associates and to purchase materials and for testing. Without the money from this project, none of the researches could be accomplished.

Participants from Dr. Yiqi Yang's group
Narendra Reddy, Qiuran Jiang, Helan Xu, Yi Zou, Wei Li, Dan Tao and Chunyan Hu.

Targeted Audiences
textiles, agricultural and biological engineering, materials, polymers, biomaterials.

Publications

1. Reddy, N. and Yang, Y., Biocomposites Developed Using Water Plasticized Wheat Gluten as Matrix and Jute Fibers as Reinforcement . **Polymer International. 60**(4)711-716(2011).
2. Jiang, Q., and Yang, Y. Water Stable Electrospun Zein Fibers for Potential Drug Delivery. **Journal of Biomaterials Science: Polymer Edition. 22**, 1393-1408(2011).
3. Reddy, N. and Yang, Y., Completely Biodegradable Soyprotein-Jute Biocomposites Developed Using Water without any Chemicals as Plasticizer. **Industrial Crops and Products. 33**(1) 35-41 (2011).
4. Reddy, N. and Yang, Y., Structure and Properties of Ultrafine Silk Fibers Produced by Theriodopteryx ephemeraeformis. **Journal of Materials Science. 45**(24) 6617-6622(2010).

5. Reddy, N. and Yang, Y., Structure and Properties of Silk Fibers Produced by *Antheraea polyphemus*. **Journal of Biobased Materials and Bioenergy.** 4(4) 367-371(2010).
6. Zou, Y., Xu, H., and Yang, Y., Lightweight Polypropylene Composites Reinforced by Long Switchgrass Stems, **Journal of Polymers and the Environment**. **18**(4) 464-473(2010).
7. Jiang, Q., Reddy, N., Yang, Y. Cytocompatible Crosslinking of Electrospun Zein Fibers for the Development of Water Stable Tissue Engineering Scaffolds. **Acta Biomaterialia**. **6**(10) 4042-4051(2010).