

Multi State Proposal

Project Number: SDC 345
Project Title: Biobased Fibrous Materials and Cleaner Technologies for a Sustainable and Environmentally Responsible Textile Industry
Project Duration: October 2012-September 2016

1. Statement of Issues and Justification

1.1. Addressing national/regional priorities

Developing biobased products is a national priority. The USDA is devoting considerable efforts to promote the use of biobased products. In fact, USDA has a special “BiopREFERRED program” that “aims to increase the purchase and use of renewable, environmentally friendly biobased products while providing “green” jobs and new markets for farmers, manufacturers, and vendors.” Under this program, the USDA lists 11 different categories of products with several different product types in each category. Fibrous materials have been listed to a limited extent and are only included in the carpets and composites sections of the biobased programs. However, fibrous materials can be used to develop a wide range of biobased products that offer substantial benefits to farmers, the economy, biobased product industries and the environment. On a national level, the fibrous biobased products can replace synthetic polymer based materials and help to establish a biobased agricultural industry. Such an industry will rely on indigenously available renewable, abundant and inexpensive agricultural byproducts and coproducts. Such an effort will help to promote the “BiopREFERRED program” established by USDA.

In addition to adding high value to agricultural byproducts and coproducts, textile materials can help to protect the citizens, agricultural commodities and infrastructure from internal and external biological threats. As part of this research, high-performance textiles will be developed that defend public and healthcare personnel from biological hazards, as well as protect fire fighters and first responders from fire hazards. This part of the project will address three key focus areas as identified by The National Strategy for Homeland Security: 1) defending against catastrophic threats (bio-defense); 2) protecting critical public health and safety infrastructures; and 3) domestic counter-terrorism. In addition, this research will study the ability of textile materials to provide protection against healthcare associated infections (HAI) which have recently emerged as a major concern among health care professionals (Vigo, 2001; Liu, 2001).

Protecting farms and enhancing productivity are vital to the economic competitiveness and sustainability of our agricultural crops that is related to the Science Roadmap for Agriculture prepared by the National Association of State Universities and Land-Grant Colleges (NASULGC) and Experiment Station Committee on Organization and Policy (ESCOP). The biodegradable mulches that will be developed in this project will help to protect crops, reduce costs and decrease environmental pollution and achieve the goals set by NASULGC. The work on development and evaluation of eco-friendly meshes for insect control will reduce the use of chemical pesticides and shed light on alternative methods of pest management in our farms.

As Earth’s population increases, there has been tremendous increase in the demand of fresh water for farming, food preparation and processing, textile production, and daily use, etc. The cost of managing, treating, and delivering fresh water is rising very rapidly. The ‘green’ seed coatings will provide not only the boost to initial growth but protection from fungal attack. The

textile industry is a major consumer of fresh water. Ecotextile News reported in the 2009 April issue that, globally, approximately 1.2 billion cubic meters of water are used annually for cleaning and bleaching of textiles alone. Much more is used in dyeing, finishing and other textile processes. In addition, more than 2.4 billion lbs of dyes, detergents and auxiliary chemicals were used by world textile industries. However, current methods of continual purification of the wastewater has shown build ups of different contaminations and poor dyeing finishing performances. Conventional filtration, followed by acid base neutralization, has been used with very limited success. Electrochemical oxidation (Vlyssides, 1998) and ozonation (Ehud, 2006) are examples of newer technologies. Recently, a new photochemical and photo catalytic oxidation (PECO) technology has been developed at UW-Madison to convert aqueous ammonia into nitrogen gas (Ramsey, 2009). The potential application of this new technology to treat textile waste water will be studied as part of this proposed research.

1.2. Importance of the work

Adding value to agricultural byproducts and coproducts of biofuel production and finding sustainable alternative sources for existing petroleum-based products will have a substantial and long term impact on agriculture, the economy, the environment and energy independence and security of the United States. The five major food crops (corn, wheat, soy, sorghum and rice) in the United States generate about 300 million tons of lignocellulosic crop residues as byproducts every year. These byproducts currently have limited use. These abundant and low cost (\$50-\$80 per ton) agricultural byproducts are renewable and sustainable without the need for additional natural resources. It has been demonstrated that lignocellulosic byproducts such as corn stover, rice and wheat straw can be used to obtain high quality fibers suitable for textile, composite and other applications (Reddy, 2005, 2006, 2007; Huda, 2008). Cellulose from these lignocellulosics can also be used to generate regenerated cellulose fibers (rayon) or it can be utilized as reinforcing agent or filler to plastics and resins. Similarly, biofuel production from cereal grains generates abundant carbohydrate and protein containing coproducts that have limited applications. For instance, more than 10 million tons of distillers dried grains (DDG) are generated every year and DDG has a selling price of less than \$130 per ton, much lower than the current selling price of common thermoplastic polymers such as polypropylene and polyethylene (> \$2,000 per ton). It has been demonstrated that coproducts of biofuel production such as distillers dried grains (DDG) can be used to develop thermoplastics (Hu, 2011a,b).

Protective crop covering systems are used to optimize production and enhance the quality of fruits and vegetables. A key component of crop covering system is protective mulches that help to control weeds, maintain soil temperatures, conserve moisture and shorten the time to harvest crops. Currently, plastic mulches, primarily those made from polyethylene, are widely used. Traditional plastic mulches have initial cost, field removal and disposal issues. Removing plastic mulches from the field is estimated to cost \$600 per acre (Schrogen, 2004). Disposing the mulch in landfills is another problem since the synthetic polymers are non-degradable and burning the mulch is prohibited in many states. Therefore, it is ideal to have mulch that degrades in the soil (biodegradable mulches, BDMs) without affecting the crops, soil or the environment. In this research, non-woven biodegradable mulches will be developed from poly(lactic acid) (PLA) or polyhydroxyalkanoates (PHA) and their blends. PLA has been shown to be much more environmentally friendly than conventional non-biodegradable polymers such as polypropylene (PP) and polyethylene terephthalate (PET) in that PLA has documented to produce much less

greenhouse gas emissions, such as carbon dioxide, than PET. Protein based mulches, rich in nitrogen, also provide organic fertilizer for the new seedlings as the protein degrades. Further, the biodegradation rate of proteins may be controlled by crosslinking and/or by other additives. Proteins such as soy protein in the form of defatted soy flour are very inexpensive (\$0.2/lb).

1.3. Consequences of not doing this research

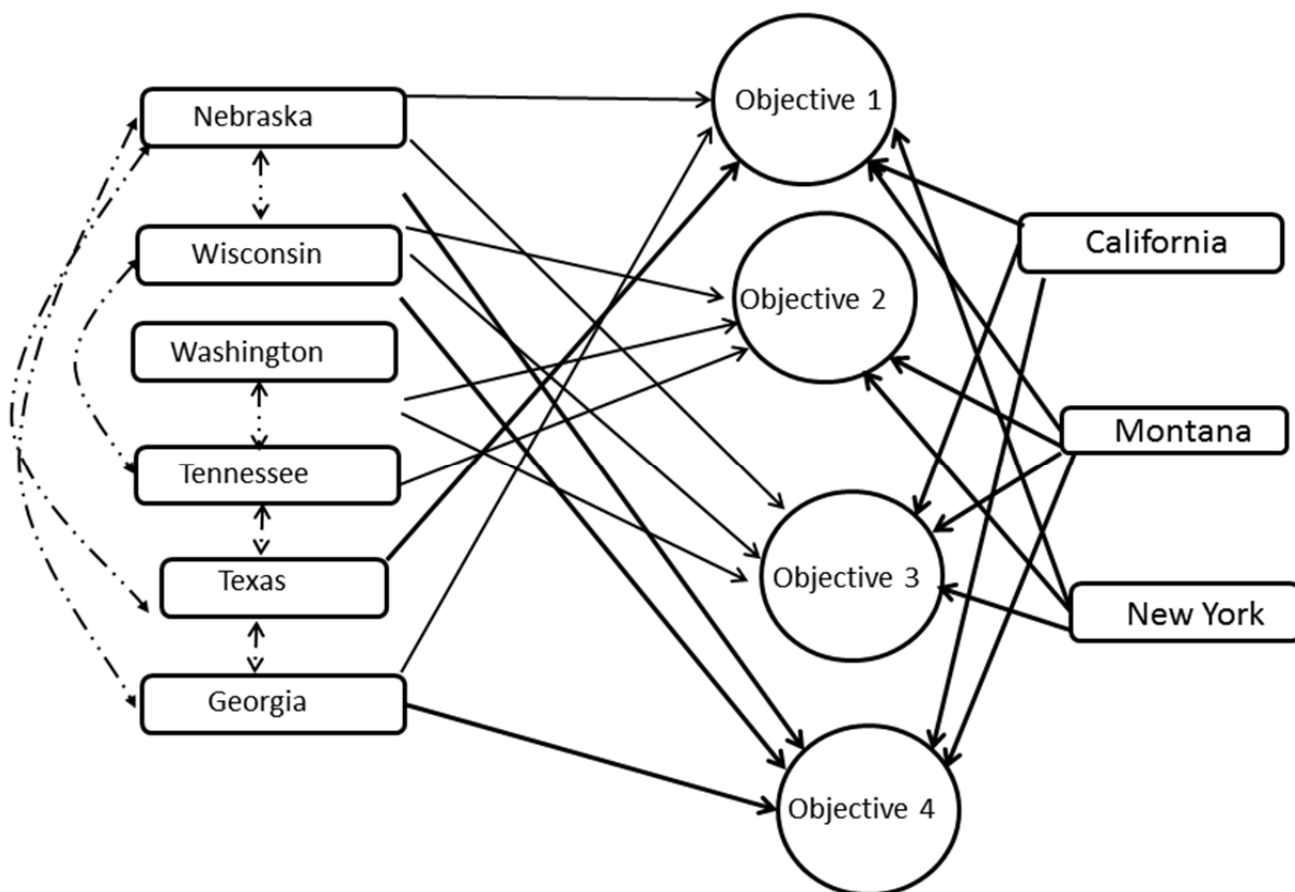
Developing biobased products from agricultural byproducts and coproducts as well as wastes has the potential to increase income from the crops while benefits the farmers and biofuel industries economically and reduces our dependence on petroleum based resources. The new value-added products will increase income from crop production, help to establish small businesses and create new jobs leading to economic benefits. Similarly, biobased products can replace synthetic polymers based products and benefit the environment. Adding value to the coproducts of biofuel production will help to reduce the cost of biofuels and make biofuels competitive to fossil fuels and reduce our dependence on foreign sources for energy. The complexity of pathways and issues in converting and utilizing agricultural byproducts and biofuel cobyproducts require a coordinated effort among researchers with complementary expertise to develop, evaluate and demonstrate the potential of biobased products for various applications. This multi-state proposal provides an opportunity for experts in biobased products to collaborate synergistically and develop unique biobased products. Without such an effort, it may be difficult to commercialize biobased products, improve income from agriculture, reduce cost of biofuels and have a sustainable and economically competitive biobased industry. Similarly, methods to conserve energy and water and develop novel technologies to treat textile waste water will help to reduce cost of textile processing and pollution and help to make the textile industry environmentally friendly. This will give a competitive edge to the US textile industry and, in addition, will potentially save billions of cubic meters of water.

1.4. Technical feasibility of the work

The PDs of this project are experts in their fields and have demonstrated the feasibility of the proposed work in their particular area of expertise. For example, researchers at the University of Nebraska-Lincoln (UNL) have demonstrated the use of the agricultural byproducts and coproducts to develop fibers, composites and thermoplastics. They have also demonstrated that the bioproducts developed in their laboratories are suitable for textile, medical and composite applications. Similarly, coproducts of biofuel production such as distillers dried grains with solubles (DDGS) have been used to extract high value chemicals and also to develop biothermoplastics. Developing regenerated cellulose fibers has been investigated by Texas-Austin and Dr. Chen is an expert in using lignocellulosic materials to develop regenerated cellulose fibers and films for potential use in textile, medical, food and other areas. Researchers at University of Wisconsin-Madison (UW-Madison) are experts in plasma treatment of textiles, textile dyeing/finishing and textile sustainability and Co-PI Karen Leonas at Washington State University and Co-PI, and Douglas G. Hayes at The University of Tennessee, Knoxville (UTK) are experts in developing biodegradable materials from poly(lactic acid) and other biopolymers for various applications including crop protection. Co-PI Suraj Sharma from the University of Georgia-Athens is an expert in utilizing proteins for high value composite and thermoplastic products. They have also studied textile wastewater quality by growing microalgae. Co-PI Anil Netravali at Cornell University has expertise in the areas of green composites, green mulches as well as seed coatings, all derived from agricultural and food waste products.

1.5. Advantages of doing the work as a multistate effort

This project has a wide range of Objectives starting from extraction of polymers, fabricating of products, characterization, developing applications and understanding the suitability of the products developed for various applications. Such a wide range of Objectives are essential to completely understand the potential of developing biobased materials and improving the safety and performance of textile based materials. However, this effort requires considerable expertise and resources that are not available to any one PI or in a single state. The PIs involved in this project are well known experts in their specialized areas. Combining the expertise of the PIs will provide an opportunity to develop a wide range of bioproducts and identify potential applications for the bioproducts in various areas. This will also enable the PIs to utilize and share the resources available to them more efficiently and ensure that the project develops useful products and technologies in a short period of time. As shown in the schematic below, the raw materials or products developed by one PI will be used by another PI to evaluate the suitability of the products for various applications. Similarly, products developed by one research group will be modified by another research group to improve the properties and potential applications. This collaborative research will enable us to have a thorough understanding of the potential of developing biobased products from agricultural byproducts and coproducts for various applications.



Schematic representing the contributions by the Universities to the Objectives (Solid lines) on page 6 and the collaborations between the participating Universities (broken lines)

1.6. Likely impacts from successful completion of the work

Successful completion of this research will lead to the development of novel bioproducts and biomaterials for the textile, composite, medical, automotive, agricultural and other industries, and efficient technologies to protect personnel from biological and fire hazards. Developing high value biobased materials will add substantial value to the agricultural byproducts and coproducts and therefore benefit the farmers economically. Such value addition will also help to reduce the cost of biofuels, reduce our dependence on non-renewable resources for fuels and decrease the adverse impact on the environment due to the use of synthetic polymers. It is expected that the products and technologies developed in this project will be unique and have high potential for commercialization in the near future. Small businesses can be established in rural areas to develop the biobased products developed in this research leading to job creation and benefit to the economy. Improved utilization of the abundant byproducts and coproducts from agricultural and biofuel production for high value applications will create an indigenous will help to reduce our dependence on foreign fossil fuels and increase national security. Similarly, having adequate protection against biological threats and fire hazards will help to safeguard citizens and vital installations from terrorism and accidents.

2. Related Current and Previous Work

A CRIS database search (April, 2011) did not show any major overlap with the proposed research. There were no reports on developing natural cellulose fibers or regenerated cellulose fibers from agricultural byproducts or coproducts. Similarly, there were no reports on developing fibers from plant proteins for textile, composite or medical applications. However, some reports are available on utilizing poultry feathers for various applications. Proposal 2009-00369 was to develop biodegradable nursery pots from feather keratin by injection molding. Proposal 2005-02675 developed carbon fibers from feathers and was terminated in 2009. There are no reports on developing thermoplastics from feathers by chemical modifications as proposed in this research. A USDA in-house project (2004-2008) examined the use of cotton gin byproducts as mulches and a current project 2009-02484 is examining the use of biodegradable mulches developed from PLA and PHA. Co-PI Leona's and Wadsworth are participating in the current PLA mulch project. In this project, the PLA-PHA mulches will be provided to Co-PI Sarmadi for plasma treatment to modify the surface of the non-wovens and the benefits of using plasma treatment for mulches will be evaluated. There are no reports on using algal proteins for bioproducts. Similarly, no reports were found on chemical modifications of lignocellulosics or coproducts of biofuel production for thermoplastic applications. There are no currently active CRIS projects on plasma treatment to improve the biological or antimicrobial behavior of textile materials.

Every state involved in this project has done some major work related to the development, characterization, finding applications and/or evaluating the feasibility of using biobased products for industrial applications. In the mid-nineties, Larry Wadsworth, retired professor of the University of Tennessee Department of Materials Science and Engineering and currently adjunct professor with the UTK Department of Biosystems Engineering and Soil Science (BESS), led a research project at UTK with Cargill, parent company of NatureWorks, to develop the first PLA nonwovens made by the meltblown and spunbond processes and has continued to make innovations with Co-PI Doug Hayes and Co-PI Karen Leonas in the use of PLA in nonwovens for crop mulch covers. Larry Wadsworth will serve on this multistate project as an adjunct

faculty member with the UTK BESS Department. Also, a new soy and other grain protein based mulch with additives derived from corn stover and rice and wheat straw will be developed by Co-PI Netravali. These mulches also provide fertilizers as the protein biodegrades. Mulches that are compostable and potentially biodegradable will provide significant environmental benefits since synthetic plastic mulches readily undergo fragmentation and persist in the ecosystem, where they can adsorb hydrophobic compounds such as pesticides, leading to high local concentrations, and will reduce labor costs for mulch retrieval at the end of the growing season. Researchers at UNL have demonstrated that agricultural byproducts such as cornstover and wheat straw can be used to extract high quality natural cellulose fibers for textile, composite and other fibrous applications (Reddy, 2005, 2006, 2007; Zou, 2010; Huda, 2009). Dr. Chen has experiences in developing regenerated cellulose fibers. Co-PI Sarmadi has used plasma to modify and enhance the properties of textile materials for biological and fire protection. At UC Davis, major plant components, i.e., cellulose, hemicellulose, lignin, have been isolated from agricultural crop residues and food and beverage processing wastes in high purity and converted to several nanomaterials (Lu, 2012). Researchers at UNL have also shown that the byproducts can be used directly as reinforcement for composites using a novel composite fabrication method developed in their laboratories (Huda, 2008; Zou, 2010). Similarly, they have also shown that the coproducts such as DDGS generated during the production of biofuel can be used to extract high value proteins, carbohydrates and oil and also to develop biothermoplastics through chemical modifications (Xu, 2007; 2009; Hu, 2011a,b). Dr. Hsieh is an expert in fibers and materials chemistry and will contribute to Objectives 1, 2 and 4. Research at Cornell has focused on Green composites, mulches, seed coatings and green nanofibers for filtration (Chabba and Netravali, 2005, Huang and Netravali, 2006, Netravali, 2007, Huang and Netravali, 2008, Goda et al., 2009, Kim and Netravali, 2010, Takagi et al., 2011, Nakamura et al., 2012). The green nanofibers from soy protein can be effective not only in catching the fine dust but also bacteria and viruses. These can be used for personal masks to protect from bacteria and viruses. Dr. Belasco is an expert agricultural economist and will contribute to all the Objectives in this proposal.

3. Objectives

3.1. Objective 1: To develop novel biobased polymeric materials (NE, GA, TX, WI, MT, NY and CA)

- a. To develop fibrous materials from cellulose in lignocellulosic agricultural byproducts for textile, composite and super-absorbent applications
- b. To develop fibrous materials from the proteins in the agricultural coproducts for textile and composite and medical applications
- c. To develop fibrous materials from synthetic biopolymers for textile and composite applications
- d. To develop regenerated cellulose (rayon) from lignocellulosic agricultural byproducts
- e. To develop biothermoplastics and thermosets from the carbohydrates and protein in agricultural byproducts and coproducts for textile and composite applications
- f. To obtain algal proteins and develop biothermoplastics and thermosets from the algal proteins
- g. To evaluate the economics of the products and processes developed

3.2. Objective 2: To develop and evaluate biobased fibrous products for eco-friendly crop protection (TN, WA, NY, WI and MT)

- a. To develop biodegradable non-woven mulches from poly(lactic acid), polyhydroxyalkanoates (PHA) and their blends and sprayable mulches using protein processing wastes.
- b. To evaluate the economics of the products and processes developed

3.3. Objective 3: To develop and evaluate biobased products for health and safety applications (NE, WI, TN, WA, CA, MT and NY)

- a. Understand the potential of plant proteins as biomaterials for medical applications.
- b. To create barrier fabrics with novel finishes and processes for protection against biological threats
- c. To create newer fiber products and designs for textile and apparel products to address the fire safety issues.
- d. To develop nanofibers from proteins and starches and characterize their effectiveness for bacteria catching efficiency.
- e. To evaluate the economics of the products and processes developed

3.4. Objective 4: To develop and evaluate methods to remove dyes and finishing chemicals from textile waste water (WI, GA, NE, CA and MT)

4. Methods

4.1. Developing natural, regenerated cellulose and protein and synthetic fibers from the agricultural byproducts and coproducts

The lignocellulosic agricultural byproducts such as corn stover, wheat and soybean straw and cotton stalks will be used to obtain natural cellulose fibers for textile and composite applications (Reddy, 2005, 2006, 2007). The byproducts will be treated with alkali and/or enzymes to remove non-cellulosic substances and obtain fiber bundles. Treatment conditions such as concentration of alkali, time and temperature will be varied to obtain high yield (25-35%) and fibers with desired properties (lowest fineness, length between 1.5-3.0 cm and good strength and elongation). Conditions developed in our previous researches will be used as reference to extract the fibers (Reddy, 2005, 2006, 2007). Regenerated cellulose fiber will be developed from lignocellulosic feedstock using environmentally friendly solvents. We will use ionic liquid solvent system to dissolve the cellulose and recover the solvent after the fibers have been precipitated. The entire system will be closed loop which saves energy, reduces pollution and makes the whole system environmentally friendly. In addition pure cellulose and nanocellulose will be isolated and derived from agricultural residues and food/beverage processing wastes. Novel fibrous structures will be generated by self-assembling, physio-sorption, cryo- and thermal processes.

Proteins such as zein, soyproteins and wheat gluten in the agricultural coproducts and keratin in chicken feathers will be made into regenerated protein fibers for textile and medical applications. Biofuel coproducts such as distillers dried grains (DDGS), camelina meal and algae meal will also be used as sources for the proteins. The coproducts will be treated with solvents to extract the proteins with high molecular weight and viscosity required for fiber formation based on our experiences in extracting zein from DDGS (Xu, 2007). Proteins will be dissolved using solvents such as aqueous ethanol or urea and reducing agents. Concentrations of the proteins, chemicals, aging time and temperature will be varied to obtain a solution suitable for fiber spinning. Both

dry and wet spinning will be used depending on the solvents used for dissolution. In the case of wet spinning, a coagulation bath consisting of salts and acids will be used to precipitate the proteins. Fibers obtained will be washed thoroughly, drawn and stored for further studies. Synthetic biopolymers such as poly(lactic acid) (PLA) and poly(hydroxylalkonate) (PHA) derived from renewable resources will be used to develop fibers. These polymers will be melt spun into fibers using a laboratory fiber extruder (Reddy, 2008). Melting temperature, extrusion speed, collecting speed, drawing ratio etc will be modified to obtain fibers with properties comparable to fibers in current use.

Electrospinning will be used to produce ultra and nano scale fibers from the proteins and synthetic polymers for medical applications. Since soyproteins and wheat gluten do not dissolve in common solvents used for electrospinning, we will develop new technologies of dissolving the proteins and obtain electrospun scaffolds. Protein dissolution and electrospinning conditions will be modified to obtain scaffolds with the properties (fiber dimensions, pore size, porosity) desired for medical applications. Synthetic polymers will be dissolved in suitable solvents (chloroform, dimethylsulfoxide, dimethylformamide) and the polymers will be electrospun into fibers for various applications.

4.2. Crosslinking and imparting functional properties to the fibers

4.2.1. Crosslinking

The fibers obtained, especially the regenerated protein fibers, may not have the mechanical properties or water stability required for textile and medical applications. Carboxylic acids have proven to provide the required properties and are also biocompatible (Jiang, 2010;2012). We will crosslink the fibers using carboxylic acids (citric acid, butanetetracarboxylic acid). New method of carboxylic acid crosslinking without using catalysts will be considered to crosslink the fibers (Reddy, 2007). Crosslinking conditions such as concentrations of crosslinker, time and temperature of crosslinking will be optimized to obtain the desired improvement in fiber properties.

4.2.2. Antibacterial properties

Fibers will also be treated with plasma to impart antibacterial and fire-resistant properties to the fibers. The plasma functionalization will be done in a capacitively coupled parallel plate RF-reactor. The reactor has two disk-shaped electrodes with a gap of 10 cm with the lower electrode and the chamber grounded and the upper electrode is connected to the RF-power supply. It is equipped with 40 kHz (continuous) and 13.56 MHz (continuous and pulsed) power supply. The first class of polycationic surfaces generated will be quaternary ammonium groups. To synthesize quaternary ammonium groups on the surfaces, the inert substrates will be plasma functionalized to deposit nitrogen containing groups. These groups will be subsequently in situ or ex situ reacted with other simple organic molecules to form polycationic groups. The substrates after the plasma-functionalization will be characterized using Electron Spectroscopy for Chemical Analysis (ESCA), FTIR and AFM. The type of species generated in the plasma environment will be investigated using Residual Gas Analysis (RGA). Untreated and modified substrate surfaces will be evaluated for their ability to inactivate pathogens and inhibit biofilm formation. We will also evaluate the efficacy of the surfaces toward the aerosolized bacteria.

4.2.3. Fire resistance

The synthesis of cross-linked macromolecular structures on the surface of the cotton and their flame retardant activity has been successfully assessed with sodium silicate/ oxygen plasma (vacuum) treatments. However, the practical application of plasma requires the development of commercially available, reliable and lower cost plasma systems. Considering that, we propose to continue this research with a comparison between vacuum plasma treatments (what has been done until now) and atmospheric plasma ones. Therefore, the first approach would be using atmospheric plasma treatments to cross-link sodium silicate (SS) pre-deposited layers in order to produce flame retardant cotton. The second approach will be dip coating the substrates with polydimethyl siloxane (PDMS) thin films. Atmospheric plasma will play an important role in removing the methyl groups (which are flammable) from the structure of the PDMS and cross-linking it onto the surface of the cotton. Tetrahydrofuran (THF) will be used as solvent for PDMS. The third approach will be to synthesize poly-silicic acid (PSA) by a sol-gel polymerization of silicon alkoxides. PSA will be deposited onto the cotton substrates and polymerized into a cross-linked macromolecular structure using atmospheric plasma. Tetramethylorthosilicate (TMOS) and tetraethylorthosilicate (TEOS) have often been used as silicon alkoxides in the synthesis of hydrogels that polymerize into PSA. In our case, TEOS is preferred over TMOS because of the lower toxicity of the byproduct (ethyl alcohol). These plasma approaches will lead to high limiting oxygen index (LOI) cellulose materials that usually render flame retardant properties. The burning behavior will be investigated with the 45° angle auto flame chamber. Thermal properties and surface characterization will be studied using DSC, XPS, ATR/FTIR and SEM.

4.3. Characterizing the structure and properties of the fibers

Fibers obtained will be tested for their composition, fineness, length, morphological and physical structure and tensile properties using ASTM standards. Composition of the natural cellulose fibers will include determining the % cellulose, % hemicellulose, % lignin and ash using ASTM standards. Optical and scanning and transmission electron microscopes will be used to determine the longitudinal and cross-sectional features of the natural, regenerated and synthetic fibers developed. Physical structure in terms of % crystallinity, crystal size and dimensions will be determined using a X-ray diffractometer according to standard fiber diffraction studies. Tensile properties to be tested include strength, elongation and modulus. Electrospun fibers will be characterized for their porosity, pore sizes and tensile properties (compression strength and modulus). Protein fibers will be tested to determine their ability to withstand aqueous conditions at different temperatures and pH conditions using phosphate buffered saline (PBS) and enzyme solutions to evaluate their potential for medical applications. .

4.4. Processing the biofibers into textiles

The natural cellulose fibers obtained will be studied for their potential to be used for textile applications. The fibers will be bleached using hydrogen peroxide and the bleaching conditions will be optimized. Whiteness (CIE WI) of the fibers after bleaching will be measured using a Hunter Lab Ultrascan XE spectrophotometer (Reddy, 2011a). Two dyes from the major dye classes will be used to dye cellulose fibers (Reactive, direct, vat and sulfur) will be chosen to dye the fibers. Examples of specific dyes include Reactive Red 120, Direct Red 80, vat blue 1 and Lecuo sulfur black 1. The dyed fibers will be tested for wash, crock and light fastnesses, according to AATCC Test Methods 61-2A, 8 and 16-3, respectively.

Yarns (ring and rotor spun) will be developed from the natural cellulose fibers. Our goal will be to develop yarns made entirely from the fibers developed in this research. However, we will also consider blending the fibers with cotton and/or polyester to enable better processing of the fibers. Pilot-scale equipments available in the PIs laboratories or at other facilities in the United States will be used to complete the yarn production. The properties such as tensile strength, elongation, imperfections and faults in the yarns developed will be compared to similar yarns made from cotton or cotton blends. Small samples of fabrics will also be developed is satisfactory yarns are obtained and the properties of the fabrics will be investigated.

4.5. Completely biodegradable composites from the agricultural byproducts and coproducts

The cellulose fibers obtained from the agricultural byproducts will be used as reinforcement for composites and coproducts of biofuel production such as DDG or canola meal or other synthetic biofibers or biopolymers as matrix to develop 100% biodegradable composites (Huda, 2008; Zou, 2010; Reddy, 2011b,c). Similarly, proteins in the agricultural coproducts can be used as matrix materials with or without chemical modifications (Reddy, 2011b,c). The proteins will be used as matrix and natural cellulose fibers obtained from the agricultural byproducts as described in Objective 1 will be used as reinforcement resulting in completely degradable composites derived from agricultural wastes. In addition, the synthetic biopolymers (PLA-PHA) will be used as matrix and the cellulose fibers obtained in Objective 1 can be used as reinforcement. We also plan to use starch and protein based thermoset resins and reinforce them with cellulose fibers derived from corn stovers, rice and wheat straw and other fibers to fabricate composites with excellent properties. Nanocellulose will be incorporated with biopolymers and inorganic substances to fabricate hybrids and nanocomposites. Composite fabrication conditions will be optimized and properties of the composites developed will be determined as described in Objective 1. The composites will be tested for flexural, tensile, impact resistance and acoustic properties according to ASTM Standards and the properties will be compared to similar composites in current use.

4.6. Chemical modification of biopolymers for thermoplastic applications

The lignocellulosic byproducts and the coproducts of biofuel production are non-thermoplastic. The agricultural byproducts will be chemically modified by etherification using acrylonitrile and by grafting vinyl monomers. Etherification conditions will be optimized to achieve thermoplastic materials with low amounts of chemicals. Grafting will be done using methyl, ethyl and butyl acrylates and methacrylates. Grafting conditions will be optimized to obtain high % grafting. The influence of grafting conditions on the % homopolymers and grafting efficiency will be studied.

The optimal algal cell disruption method for efficient protein extraction with minimal denaturing will be determined using three non-thermal or low energy methods that allow high throughput if scaled up to commercial levels. These include combination of osmotic shock and homogenization, high frequency ultrasound and enzymatic digestion. The cell disruption efficiency will be determined by measuring the amount of protein and nucleic acids found in the algal supernatant. Additionally, the supernatant will have its electroconductivity and total dissolved solids (TDS) measured using a water quality meter which will be correlated to the protein/nucleic acid measurements. The concentrated algal biomass from each sample will then

be analyzed by the differential scanning calorimeter (DSC) to determine the degree of protein denaturing induced by each treatment.

The cell disruption pretreatment will enhance the extraction efficiencies of proteins or lipids by compromising the recalcitrant cell wall and allowing improved mass transfer in the extraction medium. A portion of the disrupted biomass will be used directly in injection molding for whole cell biocomposites. Another portion of the disrupted biomass will undergo a hexane solvent extraction to remove the algal lipids. The remaining oil-extracted biomass is known as the protein concentrate and this fraction will be used for the formation of protein-concentrate biocomposites. Depending on the type, molecular weights, and distribution of proteins, the protein isolate will be used as a combination of all algal proteins and, if viable, further isolation and concentration will be used to provide a more purified protein isolate sample for use in the formation of protein isolate biopolymers. The protein derived may also be crosslinked to form thermoset resins and further processed into nanocomposites by incorporating nanoclay or nanofibrillated cellulose (NFC) or cellulose nanocrystals.

The chemically modified agricultural byproducts and coproducts will be compression molded to form plastics. Compression molding conditions such as temperature and time of exposure and pressure during compression molding will be varied to melt the biopolymers and develop plastics. Plasticizers such as glycerol will be added if necessary to facilitate the melting of the proteins. The bioplastics developed will undergo in-depth material property analyses using differential scanning calorimetry (DSC), thermo-gravimetric analysis (TGA) and dynamic mechanical analysis (DMA) and mechanical testing such that a quantitative database can be developed. The objective is to establish correlations between thermomechanical process parameters, bio-resin formulation and blending with the static and dynamic mechanical properties of the material. Environmental analysis will evaluate the susceptibility of the samples to moisture, and finally a biodegradation analysis will determine the materials weight loss and physical degradation over time under natural soil conditions. Bioplastic samples formed will be subjected to ASTM test methods to evaluate water and soil degradation. The information generated from these analyses will be needed to establish the feasibility of the bioplastics for compostable plastic materials. Bioplastics and composites will also be characterized using composting set up at Cornell University.

4.7. Medical applications of the Biofibers

The normal and electrospun protein fibers developed in Objective 1b and the biothermoplastic fiber developed in Objective 1d will be studied for potential medical applications such as tissue engineering and controlled drug release (Reddy, 2009b; Jiang, 2010). The attachment, growth and proliferation of the cells on the protein fibers will be tested using MTS and DNA assays. The potential of using the protein fibers for controlled drug release will also be studied. Drugs such as metformin and diclofenac that are used to treat diseases such as cancer will be loaded onto the fibers (Xu, 2010). The kinetic and thermodynamic parameters of drug loading such as diffusion coefficient, activation energy for diffusion, affinity, sorption enthalpy and entropy will be calculated. This will provide data to determine the capacity (amount) and the rate at which the drugs can be loaded.

Antimicrobial layers will be synthesized in plasma environment under low-pressure. Substrates used for these modifications would include cellulose (cotton fabric, filter paper), medical grade silicone rubber, polyurethane and polyethylene. University of Tennessee will produce some of the non-woven fabrics. Samples will be cut into one inch diameter discs and sterilized by autoclaving prior to treatment.

Recently it has been possible to electrospin soy protein into nanofibers (Cho et al., 2010, Cho et al., 2012). Since the soy protein contains amino acids that carry charge, the nanofibers can easily attract dust particles and bacteria. It needs to be seen how well they attract viruses as well. Such nanofiber based filters would be attractive for personal protection masks as well as in hospitals, particularly surgery rooms to prevent bacterial infections which are common after surgery. We will prepare nanofiber based filter media by electro-spinning process and characterize the bacteria filtering efficiency (BFE) of nanofilters using a newly developed laboratory technique that provides quantitative results. Special aerosol will be prepared for this test using non-pathogenic *E. coli* (gram -ve) and *S. aureus* (gram +ve) bacteria strains as well as reovirus and influenza virus.

4.8. Biodegradable non-woven mulches

Spunbond and meltblown nonwoven BDM prototype's will be developed from PLA and other PHAs in this study. The SP and MB nonwoven BMD can be produced from biodegradable polylactic acid (PLA) and polyhydroxyalkanoates (PHA). These nonwoven fabrics can be made to meet specific width, color, and fiber diameters which can be altered as needed in response to ongoing research findings. The mulches developed will be sent to UWM to modify the surface using plasma treatments. BDM degradation will be evaluated based on changes in physical, mechanical and chemical structure at macro- and microscopic levels. Mechanical properties of biodegradation include strength, elongation, tear strength and thickness, while microscopy techniques are used to observe physical changes (surface etching, pinpoint holes and percent crystallinity). The MB blend with 75% PLA and 25% PHB, MB 100% PLA and a similar weight spunbond (SB) 100% PLA nonwoven fabrics will be evaluated in a greenhouse in plastic trays beneath 2cm of soil with manure compost treated organic farm soil at low and high moisture levels and with and without added enzymes to enhance biodegradation. Samples will be analyzed by Gel Permeation Chromatography (GPC) for molecular weight loss and by scanning electron microscopy (SEM) will be used to observe fiber breakage by Co-PI Doug Hayes at UTK. Similar specimens will be analyzed by Co-PI Karen Leonas at WSU-Pullman to measure changes in tensile strength and elongation-to-break, tearing strength, and changes in weight and thickness, as well as SEM analysis to observe any effects on gross morphology. Preliminary results have been reported in a manuscript recently submitted for publication (Wadsworth, submitted 2012). Biodegradable protein based hydromulches will also be developed. These can be formulated using crosslinked proteins blended with other additives including fibers derived from corn stovers, rice and wheat straw. These have special advantage as organic fertilizer as the protein degrades. Another advantage is that they can be sprayed on ground or around plants. In addition, other additives such as fungicides, short-lived pesticides, etc. may be easily added to protect the plants.

4.9. Dye waste water treatment

Wisconsin will perform dyeing of fabrics and use PECO to treat the waste water. Wisconsin will also monitor the pH before and after treatment, and use spectrophotometer to analyze the color of fabrics, the wastewater color and to detect the presence of organic compounds. University of Georgia will grow microalgae in the PECO treated water by Wisconsin, from consortia of strain identified previously using Dalton Carpet wastewater. Georgia will also use LC-MS to determine the extent of organics removal through this algal bioremediation process. Nebraska will use liquid chromatography to separate the degradation products, followed by FTIR, ¹H-NMR, ¹³C-NMR, and Mass spectrometry on treated and untreated wastewater provided by the UW-Madison. Since there will be much work involved in the analyses, this work first will focus on reactive dyes. Reactive Blue 19 will be used for initial study due to its simple structure and popularity. After the study of Blue 19, Reactive Red 120 and Black 5, will be studied because they are more complicated, but very popular in the industry. The reason to study reactive dyes is that it is the mostly used dye class in the world, and the dye with the least fixation, or highest concentration left in the used bath. Other classes such as direct, basic and disperse dyes will be studied after reactive dyes.

4.10. Evaluating the economics of the processes and products developed

The economics of the processes and products developed will be studied and potential impact on agriculture, economy and biobased products will be estimated. Costs and benefits of new bio-based plastic products arise at both the firm level and the market level. The economic assessment would compare the private net benefits (i.e., profits) from bio-based plastic products relative to traditional products in terms of reduced labor, no disposal fees, and reduced pesticide usage. This approach will follow standard process and enterprise budgets approaches, which will be refined and adapted as needed. At the market level, the switch from oil-based plastics to bio-based plastics begets the following questions. The importance of reduced demand for oil-based products (i.e., less bio-based mulches) impact oil prices or, more importantly, the relationship between oil prices and commodity prices will be estimated. With the proposed increase in demand for bio-based products, the impact on crop acres, prices and land values will be studied. For example, ethanol policies have had an important impact on corn prices. Similar impacts of bio-based products developed in this project on input price and profit volatility for producers will be investigated. The requirement of land for bio-based product development, competitiveness of the products, potential value addition and magnitude of value addition, costs and benefits of using the biobased products as public goods will be answered. Environmental assessment in terms of public benefits and reduction in burning and disposal and greenhouse gas emissions will also be a part of the economic analysis.

5. Measurement of Progress and Results

5.1. Outputs

Major objectives of this research will generate outputs in the form of products. Objective 1a will produce cellulose fibers, protein fibers will be obtained from Objective 1b, synthetic fibers will be obtained from objective 1c and 1d and biothermoplastics will be obtained from Objective 1e. Biothermoplastics will be developed from algal proteins in Objective 1f. Overall, Objective 1 will produce products that will be evaluated quantitatively for performance properties in comparison to similar products currently available on the market. Objective 2 will produce eco-friendly non-woven mulches that are expected to replace their synthetic counterparts. Objective

3b will develop textiles containing novel finishes that can protect against biological threats and Objective 3c will develop textiles and apparels with good fire resistance. Objective 4 will develop treatment methods to efficiently remove dyes and auxiliary chemicals. Results obtained will be used to publish papers, presentations at conferences and also for thesis and dissertations.

5.2. Outcomes or projected impacts

The cellulose, protein and green fibers and composites developed from the agricultural byproducts, coproducts and wastes are expected to reduce our dependence on natural resources for fibers and fibrous products. This will not only help to add more value to our agricultural crops, benefit the environment but also improve the economy creating “green” jobs. Understanding the potential of using fibers derived from agricultural crops for medical applications will develop unique biomaterials and could lead to reduction in medical costs. The biodegradable mulches can reduce the cost to farmers, reduce disposal problems and also increase the productivity from the farms. Though the current cost of biodegradable covers is higher than petroleum based films the cost is expected to decrease with higher adoption rates and increased volume of production of bio-based covers. In addition, two potential environmental and energy reducing benefits of using biodegradable covers over traditional plastics are less dependence on petroleum and no hassle of final disposability. Results from this research will be published in appropriate peer reviewed journals and also presented in textile, bioproduct, biomaterial and related meetings and conferences. Any intellectual property generated from this research will be protected through disclosures, patent applications and/or publications. The PIs of this project have close relationship with the industry and will be communicated to the industry after the required confidentiality or co-operative agreements are made. The new products and processes developed will be protected for intellectual rights at the institution(s) that collaborate with each other. Patent disclosures will be submitted to the respective universities for patent considerations. Students and other personnel working on the project will also be included in the patent according to the rules at the respective institutions..

5.3. Milestones

October 2012 to September 2014. Complete development of natural/regenerated cellulose fibers and processing natural fibers into textiles and composites (NE, TX, CA, NY). Complete synthesis of macromolecular structures containing quaternary phosphonium and ternary sulfonium groups on synthetic and natural fibers and fabrics and incorporate reactive halogens on natural and synthetic polymeric surfaces using plasma (WI). Complete optimizing algal protein extraction (GA, NY). Develop and characterize PLA-PHA and soyprotein mulches, conduct initial trials on biodegradation (TN, WA, NY). Identify methods to remove dyes and chemicals from waste water.

October 2014 to September 2016. Develop regenerated protein fibers, electrospun protein fibers and thermoplastics from proteins and conduct medical application studies for the plant protein based biomaterials (NE, CA). Characterize plasma generated surface functionalities and surface topographies, evaluate antibacterial activity on modified substances, study plasma-mediate crosslinking of poly(vinyl) benzylic chloride (PVBC) on polymeric surface and evaluate antibacterial activity of quaternized PBVC films (WI). Develop and evaluate algal protein bioplastics (GA). Complete evaluation of PLA mulches (TN, WA). Develop and evaluate efficiency of removing dyes and chemicals from textile waste water.

6. Projected Participation

Please see Appendix E

7. Outreach Plan

Members of this multi-state project have very strong publication records and close association with the industries. The members will communicate regularly with each other and meet as a team annually to share the results and products generated as well as plan and coordinate joint effort. Results from this project will also be made accessible to the scientific community, farmers and general public through publications, presentations at national and regional meetings, special technical reports and annual reports. A comprehensive bulletin will be published after completion of the project. Technologies developed in this project will be made available to the textile, bioproduct, biomaterial, non-woven and other related industries for technology transfer and licensing. All participating institutions have units that look after the IP and their licensing. The PIs are members of leading national organizations related to their scientific discipline and also regularly meet with industry leaders. The PIs will communicate their findings personally with industry representatives. The PIs will also publicize their findings by providing samples of the products developed to industries and anyone interested in their research.

8. Organization/Governance

The proposed list of technical committee members for this project is listed in Appendix E. For those states having more than one participant, one member will be designated as the voting member, as determined by that institution or AES director. The officers in the committee will consist of a chair, vice-chair and secretary. The officers along with the project administration advisor, USDA-CSREES representative, and USDA-ARS administrative advisor will serve as the executive committee. The advisors will be non-voting members. The general operational procedures will be followed as in the CSREES Manual for Cooperative Regional Research. Officers for the first year will be elected at the first organizational meeting after the project has been approved. After the first year, the election of officers will take place at the annual technical committee meetings in the fall. The chair is responsible for notifying the members of the date and place of the annual meeting, preparing an agenda, presiding over the annual meetings. The chair will also be responsible for writing the annual report for the year he/she has served as the chair. The vice-chair will assist the chair with performing the duties of chair and make arrangements for the annual meetings. The secretary will be responsible for correspondence related to the technical committee as deemed necessary as the chair or vice-chair and taking/distributing minutes at the annual meetings to the technical committee members and advisors. The duties of the technical committee are to coordinate the research and other activities related to the project. The technical committee will meet annually for the purpose of coordinating, reporting, and sharing research activities, procedures, and results, analyzing data, and conducting project business. The administrative advisor will be responsible for sending the technical committee members the necessary authorization for all official meetings. Subcommittees and meetings may be designated by the chair, if needed, to accomplish various relevant research and administration tasks such as research planning and coordination, the development of specific cooperative research procedures, assimilation and analysis of data from contributing scientists, and publication of regional or other bulletins and reports.

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