FINAL REPORT
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Extrusion and Molding of Proteins Fractions and Fats Derived from Animal By-Products for Packaging and Structural Applications

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Objectives: For proteins derived from animal byproducts, the primary objectives of this research project were to:

(i) establish a protocol for the systematic characterization of the proteins, and

(ii) process these fractions into desired forms such as films/sheets.

Project Overview:

Materials

Feather meal grade protein samples were provided by American Proteins, Inc., Cummings, GA. Since the feather meal had a high protein concentration of about 80 wt% protein, it was used throughout the first-year study. Glycerol was used as a plasticizing agent (Fisher Scientific Chemicals, Fairlawn, NJ). Other protein/plasticizer candidates will be considered in the future.

Thermal Processing

Protein sheets were made via thermal compression technique that involved six major variables: temperature, force, time, type of plasticizer, amount of plasticizer, and degree of mixing. The desired amounts of protein and glycerol were first mixed in a mortar and pestle followed by intensive mixing in a Haake mixer. A Carver 30-12-28T compression press was used to produce samples by placing 5-10 g of material on a thin aluminum plate that had been treated with a mold release (Yellow Label Bomb Lube), a commercial product that prevents the finished film from sticking to the plate. Based on literature studies, a temperature range of 150 and 200 ˚C and a minimum protein content of 65 wt% were used.

Microstructural and Tensile Analysis

An optical microscope (Olympus BX60, Olympus America Inc., Melville, NY) was used to examine the surface textures of films. Small portions of films were cut and placed on microscope slides and then examined. The procedure outlined in ASTM D 638-94, *Standard Test Method for Tensile Properties of Plastics* (ASTM 1995) was generally followed to test the biopolymers. Samples were cut to 3 inches length and 0.5 inch width. Samples were then inserted into the clamps of the tensile tester, and tested at a constant strain rate of 0.200 inch/min. Upon sample failure, the breaking stress and strain to failure were recorded. Modulus of elasticity was determined by measuring the slope of the linear (elastic) region of the stress-strain graph.

Impacts and Significance:

The results from this study indicate that a good set of conditions for making films are a composition of 65-75 wt% feather meal and 35-25% glycerol and processing temperatures between 140 and 160˚C. A compaction force of approximately 15000 lb and processing times between 2 and 6 minutes were found suitable for producing samples. Unplasticized films were thick, dark brown, and opaque. By adding plasticizer and refining the processing technique, the film appearance improved significantly. Films from the last three batches were a much lighter color and, while they were still brown, they were fairly transparent.

Films made at higher temperatures for longer times tended to be most brittle, and often also had black streaks on them to signify probable partial decomposition in the sample. Samples with low plasticizer content also were more brittle, which is to be expected, as the most brittle films are those with no plasticizer at all.

Optical micrographs showed that samples with increased plasticizer levels were usually smoother than those with higher plasticizer concentration. This is also to a degree noticeable just by observation. Samples with low plasticizer content had a mildly grainy surface texture to them. Those with more plasticizer were generally smooth, and sometimes felt slightly greasy when touched.

Water solubility of the protein films was determined by immersing the feather meal-derived samples in either boiling water at 100°C or in NaOH solutions maintained at 60°C. The films did not dissolve in water and 0.0012M NaOH, as evidenced by gel fractions of 0.88 and 0.89 respectively. But, 0.1M NaOH dissolved much of the sample leaving behind a gel fraction of approximately 0.27. In an effort to reduce the water sensitivity of the feather-meal derived films, the samples were UV irradiated (22 J/cm^2 UVA) in a high-intensity ultra violet radiation chamber. The UV irradiation showed no significant effect for films that were tested in the stronger caustic solution, but showed improved water resistance under pure boiling condition and also for the dilute caustic solution.

Conclusions

High processing temperatures enhance the plasticity of materials, but this increasing plasticity is offset by increasing thermal degradation of the biopolymers. Water solubility of the films was measured after controlling the degree of crosslinking by rapid UV-radiation treatment (22 J/cm^2 UVA) in a high-intensity ultra violet radiation chamber. Preliminary research in our lab has shown that UV treated biopolymer sheets did not dissolve in water and 0.0012M NaOH, as evidenced by gel fractions of 0.88 and 0.89, respectively.

Publications:

R. A. Walker, B. Johnson, E. J. Quin, P. L. Dawson, and A. A Ogale*, "Thermal Processing of Animal Coproduct-based Proteins", abstract in EcoComp 2005, 3rd International Conference on Eco-Composites, June 20-21, 2005, Royal Institute of Technology, Stockholm, Sweden

Future Work:

The proposed research is directed at the use protein by-products for nonfood applications. Such bio-based materials may serve as substitutes for petroleum-based synthetic polymers in environmentally-beneficial, geostructural applications. Specifically, the extrusion and molding of animal by-product proteins will be investigated to develop geostructural sheets for applications such as reinforcements of temporary roads on soft/weak soils and for oil spill containment. A thick foundation of this protein sheet can help in increasing the load bearing strength of weak subsoils. The layer is permeable to water so that it would not permanently retain a pool of water. Another, potential application is for oil spill containment near drilling and construction sites. These operations are temporary but oil seepage into the ground can contaminate the water table. The use of a thick layer of geomembrane created from these biopolymers may be explored to prevent the oil seepage, and the oil spill can be directed and collected in a sump near the construction zone. The eventual biodegradation of these materials would eliminate the problem of removal of the temporary geomembrane after the excavation is completed.

The thermal degradation characteristics of various proteins and their fractions will be determined systematically by differential scanning calorimetry (DSC) using a Perkin Elmer Pyris 1. DSC measurements will determine the "pseudo-melting" and glass transition temperatures of the various materials. Thermogravimetric analysis will be conducted in a Perkin Elmer TGA to measures the weight loss as a function of the sample temperature. The water vapor permeability (WVP) tests on the films/laminates will be performed following the standard procedure outlined in the ASTM E 96-95 (ASTM 1995). The data of weight loss versus time will be analyzed to yield the water vapor permeability values normalized with respect to the specimen thickness. In addition, water solubility test will be performed to determine hygrothermal stability in neutral, acidic, and basic media.

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