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Value-added agriculture by-products: Isolation of cellulose by thermal-mechanical extrusion methods

BACKGROUND

In the United States, 62% of the population own companion animals, and each year consumers are spending billions of dollars on their pets. In 2008, the American Veterinary Medical Association estimated that 25% of the nation's pets are overweight or obese. Increasing obesity has led manufacturers to develop foods using ingredients that will aid weight control, e.g., fiber addition.

Cellulose is a common fiber additive used to dilute calorie content and aid in restricting energy intake. It is a polysaccharide consisting of glucose units linked together by β-1,4 bonds. Cellulose is indigestible to common companion animals in contrast to a α- bonded glucose, which is easily digested. This fiber is part of a biomass called lignocellulose, which is comprised of cellulose, hemicellulose and lignin.

The pet food industry is relying on the wood pulp industry as a cellulose source. Wood cellulose can be derived from trees such as bamboo and spruce (Stern, 2009). The price of wood cellulose is continuously rising due to the demand of the paper industry. Wood is composed of 20-30% lignin and 70-80% cellulose, making it an ideal substrate to concentrate high amounts of cellulose (Forstall, 2002).

There is an opportunity to add value to underutilized agriculture by-products for their cellulose content if isolation techniques are perfected. Extrusion processing parameters can affect how efficiently cellulose can be extracted and isolated from different feed by-products. Previous research has found that increased thermal energy has a greater impact on the fibers versus increased mechanical energy. The objectives of this research were to determine the relationship between lignocellulosic degradation, extruder in-barrel temperature and screw speed of the extruder.

MATERIALS AND METHODS

The goal of this experiment was to increase thermal energy to aid in the degradation of the fibrous materials found in soybean hulls. Soybean hulls were extruded using four different extrusion-processing parameters: high and low screw speed and high and low in-barrel temperatures. The moisture content of soybean hull was determined to be 9.1%. Composition of the substrates post extrusion and chemical treatment were determined by standard AOAC methods for neutral detergent fiber (NDF), acid detergent fiber (ADF), acid detergent lignin (ADL), crude fiber and dry matter.

Soybean hulls were processed through a Wenger TX-52 pilot scale twin-screw co-rotating extruder with screw diameters of 52 mm, L/D ratio of 16:1, equipped with a low-shear screw profile (one-inch spacer, two 5.3 mm diameter circular cross-section die). Moisture level was targeted to 50%, and two in-barrel temperature levels (high [HT] and low [LT]) and two screw speeds (250 [LS] and 350 [HS] rpm) were used. The feed rate of raw material was 50 kg/h. Water flow to the preconditioner was maintained at 16 kg/h and steam flow was maintained at 13 kg/h. Water flow in the extruder was maintained at 12 kg/h with no steam inclusion.

Extruded substrates were analyzed using a scanning electron microscope (SEM). This microscopy uses electrons to form an image of the individual particles, which allows one to visually determine if degradation of the fibers has occurred during the extrusion process and (or) before a prospective chemical treatment has been applied.

Chemical treatment: Post-extrusion each extrudate was dried at 55 °C for 96 hours. Following drying, 40g of substrate was placed in a larger soxhlet, with 180 ml of toluene and 90 mL of ethanol added to the soxhlet (2:1 v/v). Each sample was treated for six hours, then allowed to air dry under the fume hood for approximately 12 hours.

Following air drying, 15g of sample was placed in a 500mL Erlenmeyer flask. A 2% hydrogen peroxide mixture was added to the samples. Samples were treated for 16 hours at 45°C in an agitating water bath. Samples were filtered and washed with 250 mL deionized water, followed by 50mL 80% ethanol and 250mL deionized water. Filtered samples were dried at 60 °C for 16 hours and then analyzed for fiber composition.

RESULTS AND DISCUSSION

The concentration of cellulose did not appear to be affected by extruder conditions; however, the content of hemicellulose decreased concurrent with an increase in lignin at each process condition when compared to the raw soybean hulls (Table 1). This result would suggest that hemicellulose is susceptible to degradation by mechanical processes such as extrusion.

Table 1: Proportion of lignocellulosic components, unextruded vs. extruded

From unextruded raw soybean hulls and extruded soybean hulls. Extruder conditions were low temperature (LT), high temperature (HT), low speed (LS), high speed (HS).

	Raw soybean hull	LT-LS	LT-HS	HT-LS
Cellulose (%)	43.99	46.35	43.39	44.53
Hemicellulose (%)	17.8	13.11	12.25	13.66
Lignin (%)	2.45	4.02	8.38	5.36

Following chemical treatment, we observed an increase in the cellulose content for each treatment with the largest concentration in the HT-LS treatment (Table 2). The scanning electron micrographs would further support the notion that the process has had some effect on the fibrous construct of the soybean hulls.

Table 2: Proportion of lignocellulosic components after chemical treatment

From extruded soybean hulls chemically treated with a 2:1 toluene ethanol mixture. Extruder conditions were low temperature (LT), high temperature (HT), low speed (LS), high speed (HS).

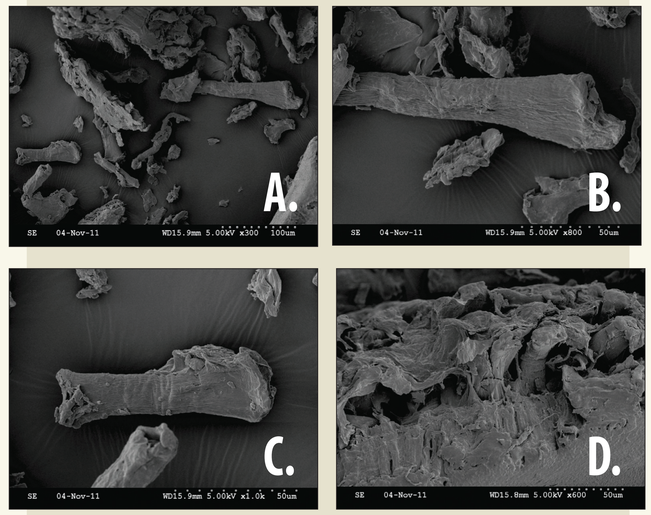
	LT-LS	HT-LS	HT-HS
Cellulose (%)	53.46	57.21	52.07
Hemicellulose (%)	20.32	16.16	23.18
Lignin (%)	6.76	4.86	4.35
Crude Fiber (%)	45.6	45.88	45.32

Conclusions: We concluded that high temperature with lower screw speed visually appeared to degrade the fiber components greater based on the cellulose concentration coupled with the SEM images (Figures A-D), wherein chemical treatment post-extrusion increased cellulose concentration.

Implications: As hypothesized, increased thermal energy from high in-barrel temperature and low mechanical energy input lead to greater cellulose isolation post chemical treatment. The extrusion process did not diminish the lignocellulosic material; rather, it may have weakened or perturbed the strong bonds and cellular integrity of the soybean hull structure. This holds promise for more thorough penetration of chemical additives used to concentrate cellulose in agricultural byproducts.

Figures A-D: SEM images of extruded soybean hulls

Scanning electron micrograph images of soybean hulls, extruded at high temperature with low screw speed.



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