AGRICULTURAL RESEARCH FOUNDATION
FINAL REPORT
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TITLE: Alkali processing with caustic recovery for enhanced digestibility of roughages

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SUMMARY: The research was aimed at developing a cost-effective alkali processing scheme for the generation of high-quality feedstocks from low-quality roughages. "High quality" roughages are herein defined as those that have substantial energy reserves, in the form of carbohydrates, that are relatively amenable to biological digestion. Wheat straw was used as a representative low-quality roughage; sodium hydroxide was the alkali reagent (caustic). Results from this study are important with respect to current work assessing the feasibility of implementing such a value-added processing scheme. Results from mass balance and digestibility experiments done as part of this project clearly demonstrate the effectiveness of using alkali to enhance the available energy content of straw. These results are promising and argue for continued work in this area. Other experiments demonstrated potential pitfalls in this technology, two in particular. The first is the enzyme inhibitory nature of the soluble phase resulting from alkali treatments. This finding indicates that further soluble phase treatments are likely required before combined processing (soluble and insoluble phases) is feasible. The second potential pitfall relates to the anodic fouling that was observed in experiments involving electrolysis of wheat straw alkali extracts. The nature of these reactions, which are as yet unknown, is likely to be relevant when considering the overall efficiency and optimization of the electrolysis-based alkali recovery process.

OBJECTIVES: The principal objective of the study was to develop an economically viable processing system for the sustainable production of readily digestible, roughage-derived, feedstocks for use in animal product and/or biogas production systems.

PROCEDURES: Wheat straw was obtained from the Hyslop farm (OSU). Aqueous alkali treatments were done using 1-5% sodium hydroxide, at solids concentrations to 10%, and temperatures to 100^{o}C – always with agitation. Following alkali treatments, the resulting slurries were either used "as-is" (combined soluble and insoluble phases) or the solid and liquid phases were separated by filtration for independent analyses. Subsequently, the cellulose fraction of feedstocks were tested for *in vitro* digestibility using commercial cellulase and hemicellulase enzyme preparations. The macrocomponent composition of feedstocks was quantified by standard methods. The impact of alkali-generated byproducts on the activity of cellulolytic enzymes was determined through kinetic experiments. A Hoffman voltameter apparatus was used for electrolysis experiments involving wheat straw alkali extracts and model solutions.

SIGNIFICANT ACCOMPLISHMENTS:

A mass balance analysis of straw components during alkali processing was completed. This phase of the study showed that total carbohydrate recoveries were negatively correlated with the severity of the alkali treatments. The cellulose (i.e., “glucan”) content of the solid phase resulting from such treatments increased with treatment severity. Glucan recovery in the solid phase was high, > 97%, in all cases; this being important as glucan/glucose is the primary energy source in these feedstock and it is of direct relevance to the usefulness of these materials as energy sources for animal and biogas production systems. Increasing amounts of hemicellulose partitioned out of the straw and into the soluble phase as alkali treatment severity increased. The carbohydrates that partition into the soluble phase, including substantial amounts of oliogmers, are theoretically available for recovery. Such recovery systems await development. Alkali treatments, in general, effectively lowered the lignin and ash content of the solids; these components could also be recovered in the soluble phase. The digestibility of a feedstock is expected to be negatively correlated with its lignin content.

An in vitro system for testing feedstock digestibility and digestive enzyme inhibition was developed. This experimental system was necessary in order to characterize the kinetic consequences of alkali treatments. The focus was on cellulose digestion/cellulolytic enzyme inhibition since cellulose is the primary energy source in these low-quality roughages. A sequential two-step enzyme treatment was developed that allowed nearly theoretical recovery of solid phase cellulose (~99%) from optimally treated straw. This is important because these *in vitro* cellulose digestibility results are expected to directly correlate with the available energy content of these feedstocks when used as a ruminant animal feed or when employed as an energy source for biogas production in anaerobic digesters. The highest sugar/energy yields from feedstock digestion were obtained in experiments which focused solely on the insoluble phase resulting from alkali treatment of the straw, *i.e.* the soluble phase had been filtered off prior to insoluble phase digestion. Sugar/energy yields, and rates of sugar production, from treated feedstocks for which the soluble and insoluble phases remained combined were not as high. Experiments revealed the soluble phase contained compounds that are inhibitory to cellulolytic enzymes. This finding is critical in that these components must be removed and/or modified for optimum use of the soluble phase components in biological systems. Initial experiments aimed at characterizing the inhibitory compounds and developing simple approaches for their removal were completed – findings from those experiments will be used to direct more detailed qualitative analyses of these compounds.

A laboratory scale system was developed to assess the efficiency of anode performance during electrolysis of wheat straw alkali extracts. Anode performance is relevant to the overall economics of alkali recovery (alkali recovery being an electrolysis-based process). Initial experiments lead us to surmise that anode efficiency could be a problem in the alkali recovery process due to the presence of alkali-generated compounds that appear to interact at the anode (based on reduced oxygen production during electrolysis). The analytical system employed for these experiments allows rapid determination of the extent to which non-oxygen reactions are occurring at the anode based on the volumetric ratio of anode- and cathode-generated gases.

BENEFITS & IMPACT: The work completed in this project contributed to our producing a relatively complete mass balance accounting of the carbohydrate fraction of straw during alkali processing. This is critical information since carbohydrate is the principle component of these roughages and it is the organic component of greatest bioavailability. The work also allowed us to document the functional properties of byproducts generated as a result of alkali processing. The impact of the combined information is that it allows improved techno-economic assessments of the feasibility of alkali processing. Such assessments, in turn, may be used to determine the merits of research projects aimed at upgrading the value of low-quality roughages through alkali processing.

ADDITIONAL FUNDING RECEIVED DURING PROJECT TERM: None. However, the results from this project have provided supporting data for a proposal that is currently under consideration for funding; that proposal, if funded, would extend our work aimed at upgrading low-quality roughages through alkali processing.

FUTURE FUNDING POSSIBILITIES: United States Department of Agriculture NIFA program, Oregon BEST, and yet-to-be identified private entities dealing in roughage utilization.