

Department of Agriculture, Trade and Consumer Protection
Division of Agricultural Development
Agricultural Development & Diversification Program (ADD)
Grant Project Final Report

Contract Number: 22060

Grant Project Title: **Robust modified soybean hull development**

Amount of Funding Awarded: **\$37,500**

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1. Background and Original Intent of the grant:

CleanWater Solutions, LLC of Eau Claire and Sturgeon Bay is a development stage company in business to advance a green technology to produce water treatment media from a limited use agricultural byproduct feedstock (soybean hulls) for removal of heavy metals (i.e. lead, copper, zinc and nickel) from wastewater and stormwater runoff. Modified soybean hulls are a product with capability to adsorb and retain heavy metal cations from wastewater streams, allowing customers to remove the pollutants to trace levels below limits required by the EPA and state regulatory agencies.

Modified soybean hulls are manufactured from this low-cost, renewable, biomaterial feedstock through CleanWater Solutions' proprietary technology to chemically attach the adsorbing functionality directly onto the fiber of the hull. Modified hulls have a Cation Exchange Capacity (CEC) significantly greater than other available products. Development began in January 2006 with \$286,000 invested to date through grants (including this \$37,500 ADD project) and the founders. The intent when applying for this grant was to allow focus on the process development parameters to generate a robust process for modified hull flakes, and further allow other scarce resources of the company for focus on pilot study and application development.

The objective of this grant project was primarily to optimize the process of chemical modification of soybean hull feedstock. Optimization implied a maximization of the capacity of the end use product to attract and hold heavy metals, defined as the Cation Exchange Capacity (CEC). Furthermore, optimization implied minimization of feedstock, chemical and energy inputs to make the product at scale volumes beyond experimental and small pilot, approaching large pilot and pre-production scale volume. The focus of this project was on one important aspect of the production, with a target objective to consistently produce CEC values of 300 meq/100g or higher in the flake form, in batches large enough to provide pilot scale product to support the commercialization development aspect of the business.

CleanWater Solutions LLC intends to acquire soybean hull feedstock from instate suppliers as much as possible. With limited crush facility capacity in the state, this may or may not be practical at the outset. At high volume, the product line will show a value-add use of soybean hulls that will benefit all soybean growers. The volume of production at which this product will impact the market forces affecting planting decisions and affecting supply and demand is undetermined. It is expected that for the foreseeable future and allowing for significant company growth the needs will be met from existing supply without adversely affecting use as an agricultural feed supplement. Conversely, as soybean hull supply grows to support biodiesel refineries, this product will help consume the surplus.

The upside potential for the product is huge. Safe and clean water supplies in this world are finite, and pressure is being felt in many areas of the United States and world to improve the performance of treatment systems. Some estimate future investment in water infrastructure in the United States in the trillions of dollars. This product has the potential to fit into these future treatment systems. Added significance lies in the fact that several alternative and competitive treatment products are produced from non-renewable feedstocks.

2. Steps to reach goal.

This project funding facilitated the expansion of a series of experiments designed to develop the process parameters for soybean hull modification. Prior to the project beginning, CleanWater had completed experiments X-1 through X-20. Since August 2007, when this project began, X-21 through X-30 have been completed. Each experiment produced at least four new sets of data. The parameters involved in the experimentation included mixing of hulls with water and chemicals, drying parameters, chemical reaction parameters, and excess chemical rinsing (washing).

The ability to focus additional development effort on the modification process was fruitful. It allowed other resources of the company to be assigned to industrial pilot testing, and providing modified hull flake to various pellet and bead formation process to support the pilot testing.

By November the modification process experiments and replications led to the conclusion that CleanWater Solutions had an understanding of three methods to consistently and repeatably produce pilot production volumes of modified hull flakes with CEC values in the range of 300-316 meq/100g, exceeding the objective (i.e. robust process). The next step was to focus on optimizing the amount of chemical and water required in the process as feedstock variables, and after that perform a first test to determine the feasibility to recover and reuse the excess chemical from the wash water. Determination of the high CEC value product validates the capability of the product, while the next steps are focused on developing a more favorable cost model for the business plan.

The most significant challenge arrived in December when it became clear the product's ability to function as desired in the pellet and bead form (this is a development objective outside the scope of the ADD grant project) would require significantly more development for industrial use applications. The company made a choice to focus on an alternative application in the emerging stormwater treatment market. This decision required a specific study to get feedback about the new market application. While not directly related, the effort of this ADD project was scaled back for a couple months to conserve funds for the final testing to support the new overall company product development objective.

The final tests under this ADD grant addressed the reduced chemical and water inputs, and supported part of the membrane pilot test study, and produced a supply of modified soybean hulls for use in application development activities in the immediate future.

Overall, the course of this project exceeded expectations for CleanWater Solutions. The ability to apply a focused effort to this aspect of the overall product development plan to develop robust process parameters and gather significant cost model data is very important. Results of this project are supporting the most significant accomplishment of the company to date. CleanWater Solutions would not choose to handle this portion of the development any differently.

3. Accomplishments

The accomplishments from this project are as follows:

- A. **Modification Process** – The time and temperature parameters for consistently and repeatably producing a CEC value over 300 in the flake form are documented.
 - i. In the course determining this process, three distinct manufacturing methods were defined to produce this result in varying quantities, including lab scale, small batch, pilot scale and production volume.
 - ii. Design criteria were established for pilot scale and high volume production equipment, with capital outlay and operating cost estimates generated.
- B. **Process Chemical reduction** – Several tests were performed and replicated reducing the amount of feed chemical (the single most expensive input to the process) and water.
 - i. Using two of the three methods identified in A(i), the result of reducing the major chemical feedstock resulted in a reduction of CEC values in the 2-3 up to 15%. By one method in pilot scale volume the value was 260-275, while a small test by another method yielded 294. This indicates at least a 50% overall cost reduction potential to produce the product. Given the superior capacity of modified soybean hulls to other alternative products indicates there is significant room to take advantage of this finding.
 - ii. Reducing the water content did not yield the same result. Fortunately, the added energy cost to produce this product at this level is not as significant as the chemical cost. What is significant is a better understanding of the recipe.

- C. **Chemical Recovery and Reuse** – There are two steps to this process. First, unwanted reaction byproducts must be removed from the wash water without removing the chemical. Second, the chemical concentration must be increased to a level acceptable for recycling. The initial indication of the pilot study using present day membrane technology is that it is feasible to recover a significant volume of excess chemical for reuse. Much more extensive testing will be required, but initial estimates are that another double digit (10%+) can potentially be removed from the product cost model

4. Conclusions

Several positive conclusions resulted from this project:

- A. The process parameters are defined to consistently produce a high CEC value (over 300) by a variety of methods that can be developed to a manufacturing process.
- B. Feedstock chemicals can be reduced without a corresponding reduction in CEC, enhancing the manufacturing cost model.
- C. It is feasible to recover the excess reaction chemical to further improve the efficiency of the process and enhance the cost model.
- D. Finally, pilot testing and other activity supported by this project indicate that in the flake form this product has the potential to be a potent, quick-acting adsorbent for at least four heavy metal constituents found in typical stormwater runoff streams. However, additional development is required before effective marketing of a product in the industrial wastewater market will be practical.

5. Future work

There remains significant work in several areas to move forward. Based at least in part on the results of this project, the company will begin undergoing limited marketing and application development for the product in the flake form for specific stormwater treatment regimens. The manufacturing process to provide product needed to support this activity is understood at this date.

Additional focus will be on the market study and the specifics of marketing the product, including pricing and distribution channels.

6. Additional Resources to commercialize this enterprise

CleanWater Solutions LLC estimates a \$250,000 investment needed to build the process equipment required to produce volumes sufficient to support application development and initial market penetration, including marketing and working capital. Within a few years, a minimum of an additional \$650,000 will be needed to take the venture to cash flow positive status.

7. Ag Industry use of results

The results of this project will allow the Agricultural Industry to tout another value-add use of an agricultural byproduct in a green technology application. CleanWater Solutions LLC will be interested in collaborative efforts if the agricultural industry has marketing ideas.