

Idaho Incubation Fund Program

Quarterly Progress Report Form

Proposal No. IF12-005
Name: Owen McDougal, Ph.D.
Name of Institution: Boise State University
Project Title: Academic Liaison with Industry: from Big D Little r to Big D Big R is Good for Idaho

Information to be reported in your progress report is as follows:

1. Provide a summary of project goals/milestones for the period just completed, accomplishments for the period just completed, and plans and goals for the coming quarter:

Project goals/milestones: The goals for this quarter were to: 1) characterize the components of products A and B using a combination of Raman spectroscopy, infrared (IR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, X-ray diffraction (XRD), and mass spectrometry (MS); 2) optimize solvent extraction techniques that are complementary to high performance liquid chromatography (HPLC) separation; 3) begin with product C for separation and component characterization; 4) evaluate the characteristics of market standard raw materials that may be used in the reformulation of eco-friendly industrial products for commercialization.

Accomplishments: HPLC separation methods were evaluated and used to characterize the retention times for surfactants and polymers common to commercial cleansers. The functionality of the newly installed evaporative light scattering (ELS) detector to identify surfactants and polymers by HPLC was verified. A commercial product, product C, was evaluated by solvent extraction and the functional component was identified as poly dimethylsiloxane. Analysis of components of four "eco-friendly" commercial products has lead to the identification of molecules significant to cleanser functionality. Eco-friendly product D contains sodium dodecylbenzene sulfonate as the principle surfactant used for cleaning.

Plans and goals: Plans to continue our work on reverse engineering of the commercial products will aid in the determination of the chemical components for standard cleanser formulations. This will include further development of HPLC methodologies, as well as characterization of the components using IR spectroscopy, Raman spectroscopy, NMR, and mass spectrometry. The spectroscopic data will be compiled into a spectral database of raw material components (surfactants, polymers, chelators, etc.) that can be used for subsequent reformulation of new products. The database will consist of components from both non-eco-friendly products and their eco-friendly product equivalents. This characterization process will allow informed decisions during reformulation.

Simultaneously, we plan to install a method for efficacy testing in the laboratory to compare existing commercial products to the newly created eco-friendly formulations. We are utilizing methods and approaches established in the literature to compare cleaning capabilities. The apparatus will be designed to mimic the bath-substrate-flow device found in the literature, which consists of a washing solution in a temperature controlled bath, a substrate coated with standard soil packed in a column, and a peristaltic pump.^{1,2} These items will be connected with silicone tubing to allow for complete circulation of the washing solution. Samples will be collected from the washing bath and assayed for total fat content in the washing bath as a means to determine wash efficiency. We then plan to move forward with product development, production, and sales of these new products.

(1) Jurado, E.; Bravo, V.; Nunez-Olea, J.; Bailon, R.; Altmajer-Vaz, D.; Garcia-Roman, M.; Fernandez-Arteaga, A. Enzyme-based Detergent Formulas for Fatty Soils and Hard Surfaces in a Continuous-Flow Device. *J. Surfactants Deterg.* **2006**, 9, 83-90.

(2) Jurado-Alameda, E.; Garcia-Roman, M.; Altmajer-Vaz, D.; Jimenez-Perez, L. Assessment of the use of Ozone for Cleaning Fatty Soils in the Food Industry. *J. Food Eng.* **2012**, 110, 44-52.

2. Provide a summary of budget expenditures for the period just completed:

\$4360.71 for graduate student salary, \$4089 in graduate student fees, and \$263 in recharge center fees to use the Center for Materials Characterization on the BSU campus.

3. List patents, copyrights, plant variety protection certificates received or pending:

None this quarter.

4. List invention disclosures, patent, copyright and PVP applications filed, technology licenses/options signed, start-up businesses created, and industry involvement:

We continue to meet weekly with industry representatives from BHS Specialty Chemical Products to review data, coordinate work effort, and discuss technical aspects of the project.

5. Include funding burn rate:

The burn rate for the first three quarters amounts to 79% of available funds being expended. At the end of the first quarter, 29% of the total funds had been expended with an additional 28% being expended in the second quarter, and an additional 21% in the third quarter.

6. Any other pertinent information:

HPLC methods developed in the lab led to the separation of two raw materials common to industrial cleaners, one an anionic surfactant and the other a nonionic polymer. Figure 1 shows the HPLC separation of the surfactant and polymer as recorded by use of the evaporative light scattering detector.

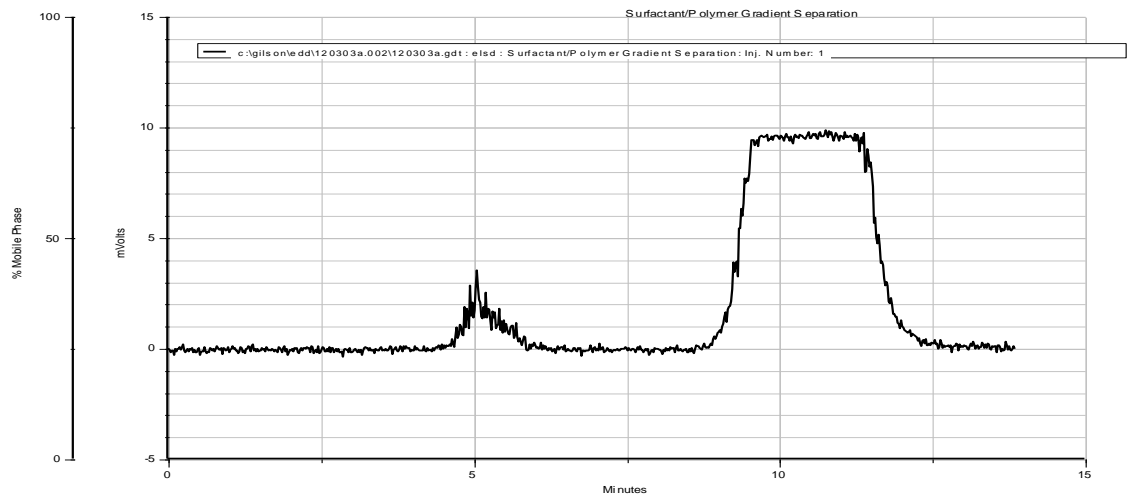


Figure 1. HPLC elution of a mixture of anionic surfactant (t=5 min.) and nonionic polymer (t=12 min.). A solvent gradient ranging from 10-90% acetonitrile was run through a C-18 reversed phase column with dimensions of 4.6 x 150 mm, 5 μ m, and 300 Å pore size.

HPLC separation methods are complex and time consuming for surfactants and polymers. While this process has presented significant challenges, results are being obtained toward the success of this project.