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 goal for this quarter was to identify optimal separation methods for surfactants and polymers present in complex mixtures common to commercial cleansers and detergents followed by the implementation of these methods for the separation and characterization of product contents. Common separation protocols utilize high performance liquid chromatography (HPLC) or liquid-liquid separations. HPLC recognition of polymers and surfactants can be done with ultraviolet-visible (UV-Vis) detection only if a chromophore is present in the material. For our purposes, it was necessary to purchase, install, and validate functionality of an evaporative light scattering (ELS) detector that allows identification of components, by HPLC, that do not contain a chromophore. The goal was to demonstrate proof of competency in component separation of two commercial detergents that we refer to as product A and product B.

Accomplishments: An ELS detector was purchased, installed, and tested for functionality. The HPLC technician that installed the ELS detector provided an additional day of training due to the complexity of the materials that require detection for the current project. A range of raw materials commonly used in the formulation of commercial cleansers and detergents were analyzed by HPLC using a combination of UV-Vis and ELS detection. The detectors have been set up in parallel to identify any and all components separated by the HPLC. The retention times for industrial standards consisting of surfactants, polymers, and chelatants were recorded. In addition, preliminary separation of product A and product B has occurred.

Plans and goals: For the next quarter, we plan to do the following: 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 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.

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

In the second quarter, $4342 was expended in student salaries, $30 in fringe benefits, $592 for supplies, and $8,899 in capital equipment (the ELS detector for the HPLC).

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:

None this quarter.

5. Include funding burn rate:

The burn rate for the first two quarters amounts to 57% 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.

6. Any other pertinent information:

A common separation of nonionic surfactants with benzene typically yields two layers: nonionic surfactants separated into the oil (benzene) layer and the remainder of the mixture should be soluble in the aqueous phase. When product B was separated with benzene, the result was three layers: oil layer containing the majority of the components of product B, as detected by proton (^{1}H) NMR spectroscopy, aqueous layer (not yet characterized), and a third, smaller layer, believed to have a mixture of surfactants (not yet characterized).

Separation of both products, A and B, by HPLC yielded one species in each chromatogram as shown below in Figures 1 and 2. In product A, there is a chromophore that is detected by UV-Vis at wavelengths of 214 and 254 nm. The component is likewise recognized by the ELS detector. Product B lacks a chromophore, which does not show up by UV-Vis, but is observable using the ELS detector.

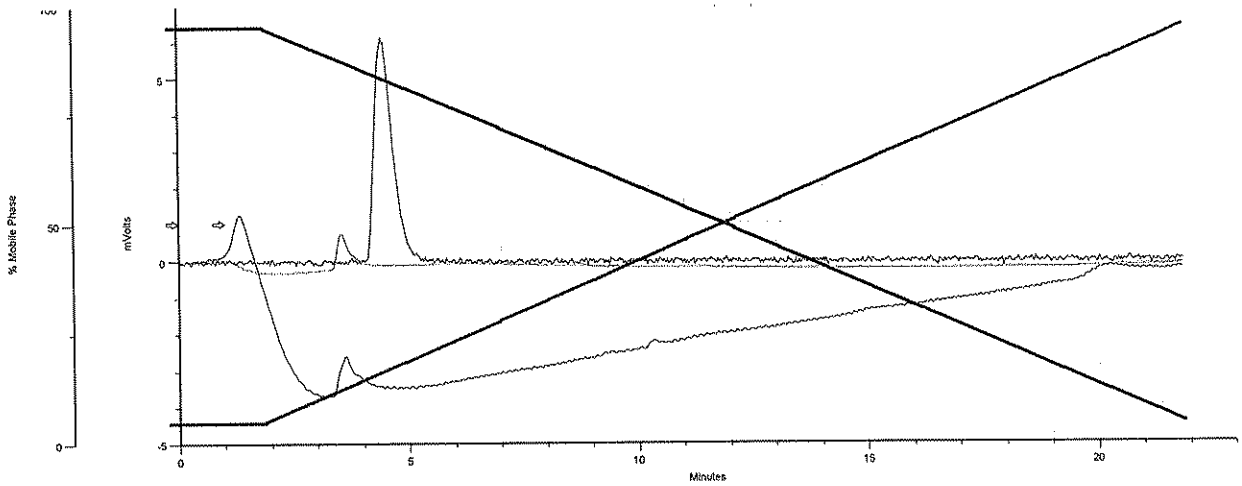

Figure 1. Gradient elution of product A. A C-18 reversed phase column with dimensions of 4.6x150 mm, 5 µm, and 300 Å pore size was used in a 20 minute gradient of 5% acetonitrile (0.1% trifluoroacetic acid (v/v)) to 95% acetonitrile (0.1% trifluoroacetic acid (v/v)). The separation is detected by 254 nm (green), 214 nm (red), and ELS (purple).

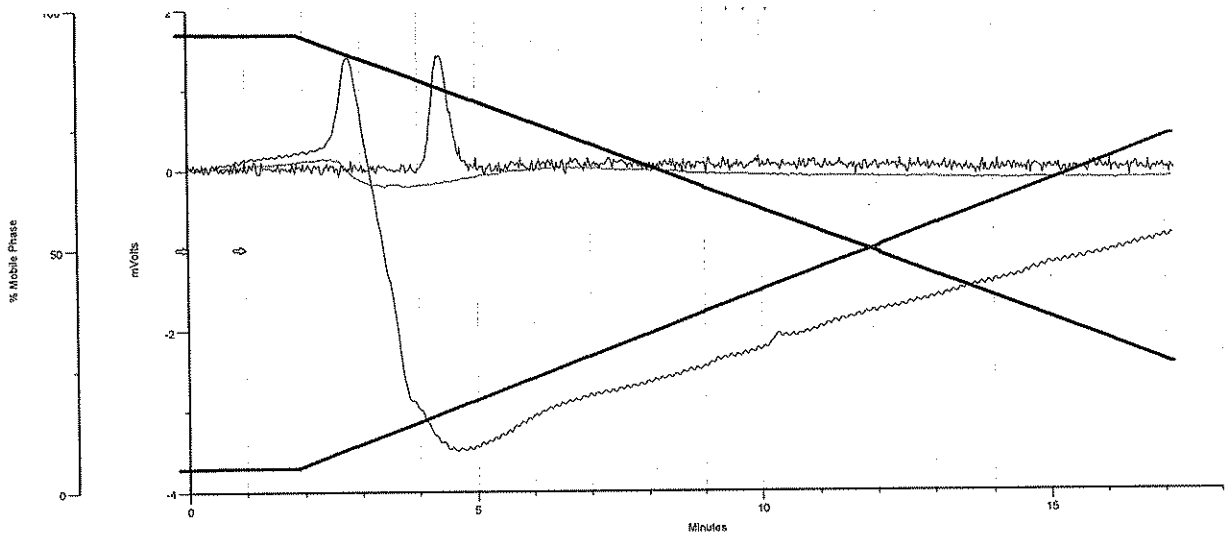

Figure 2. Gradient elution of product B. A C-18 reversed phase column with dimensions of 4.6x150 mm, 5 µm, and 300 Å pore size was used in a 20 minute gradient of 5% acetonitrile (0.1% trifluoroacetic acid (v/v)) to 95% acetonitrile (0.1% trifluoroacetic acid (v/v)). The separation is not detected by 254 nm (green) but is detected by 214 nm (red) and ELS (purple).

The separated components from these analyses are currently being characterized. In addition, gradient conditions are being modified to enhance separation and improve resolution.