Idaho Incubation Fund Program

Final 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 final report is as follows:

1. Provide a summary of overall project accomplishments to include goals/milestones met, any barriers encountered, and how the barriers were overcome:

During the course of the past year, we reverse engineered six commercial products, developed three new ecofriendly products, and created a standardized method to evaluate cleanser efficacy.

Reverse engineering: The goal of reverse engineering was to analyze commercially available eco-friendly cleaning products to benchmark market standards for green chemistry. A quantitative and qualitative approach was employed to separate, identify, and determine the role of each chemical in standard detergent formulations. We developed high performance liquid chromatography (HPLC) separation methods, and used evaporative light scattering detection (ELSD). We accomplished the component separation by HPLC, and subsequent characterization by nuclear magnetic resonance spectroscopy (NMR) for 4 eco-friendly products and 2 non-eco-friendly products. HPLC was used to isolated and collect chelating agents, surfactants, and miscellaneous chemicals for each of the six products. A combination of NMR spectra, obtained from HPLC separated components, and material safety data sheets (MSDS) listing raw materials for each product was used to confirm reverse engineering results. All raw materials appearing on the MSDS were verified by our reverse engineering method. The barriers to this process were high. We began without an ELSD and used chromatography columns that matched the polarity of the surfactants in the products. At the start of the project, we were unable to see the raw materials we hoped to separate, and the majority of product components did not elute from our C18 silica columns. HERC funds in combination with financial support provided by BHS Specialty Chemical Product (hereafter BHS), allowed purchase of the ELSD and two robust general purpose columns for reverse engineering. The HPLC instrumentation is still limited in its ability to separate closely related surfactants, inorganic salts, and polymers. The

cleaning products that are of interest to BHS are complex formulations that require sequential component separation, specialized HPLC columns for surfactants, a charged aerosol detection (CAD) detector, and a modern HPLC. BHS has purchased and is using a front-end ion exchange separation process along with an ion chromatography system; essential separation methods that are currently complementing our HPLC and NMR analyses. We have also attempted, to varying degrees of success, thin layer chromatography, fractional distillation, solvent extraction, and column chromatography.

Reformulation: We proposed to formulate three new products, an acid cleaner, alkaline cleaner, and a chlorinated-alkaline cleaner, devoid of phosphorus and nonylphenol ethoxylate (NPE). Non-eco-friendly BHS products, fitting each afore listed chemical category, provided targets for reformulation. BHS used technical information from reverse engineering, the scientific literature, and supplier recommendations to replace phosphorus and/or NPE from each product formulation with eco-friendly substitutes. Each reformulated eco-friendly product is in the process of being commercialized by BHS.

Efficacy testing: We developed a standardized, general efficacy testing method for this project using pellets containing canola oil, whey protein, diatomatious earth, and water. The pellets were exposed to eco-friendly, non-eco-friendly, commercial, and reformulated products, shaken for 2 min, followed by mass difference measurement of the initial and final pellet, as a measure of product efficacy. A product that dissolved a greater mass of pellet into solution, as compared to another product, was considered a better cleaner. We were able to successfully estimate the comparable efficacy of cleansers, and noted that eco-friendly products were reformulated with comparable cleaning efficacy for all 3 cleaner types, e.g. acid, alkaline, chlorinated-alkaline. In one case the reformulated eco-friendly product improved cleaning efficacy compared to the original non-ecofriendly commercial product. All efficacy tests were performed five-fold; standard deviation and standard error measures were used to validate test results.

2. Describe the current state of the technology and related product/service:

We used an outdated, 1990's vintage HPLC with a newer ELSD for this project. The UniPoint HPLC software functions on a Microsoft Windows 95 operating system and has not been supported by Gilson for close to a decade. As a result, peak resolution and definitive separation of multiple surfactant systems were

difficult if not impossible to achieve. By HPLC, we were able to separate chelating agents/sequestrants and miscellaneous chemicals from the surfactants in cleaners. We have a state of the art NMR facility that was used for characterization of the separated components, which provided identification of many components from HPLC. Unfortunately, the complexity of sanitation products still presents a challenge in complete characterization and identification of mixture components.

3. List the number of faculty and student participants as a result of funding:

To achieve the success that we have on this project required the dedicated effort of one faculty member, a postdoc, a graduate student, and three undergraduate students. There were a number of other individuals that also contributed considerable effort to this project. Included in this later category are the BHS R&D and Process Manager as well as the BSU NMR Facility Manager.

4. What are the potential economic benefits:

Within the next two months, BHS will scale for production and commercialize the three new eco-friendly cleaning products formulated during the past year. These products will provide alternatives for current BHS customers in Idaho that are concerned with limiting phosphorous discharge to below EPA mandated levels. Idaho manufactures are faced with increased usage fees and fines resultant from excessive phosphorous discharge into municipal water treatment facilities and riverine systems. The new eco-friendly BHS products will not only help reduce the overall phosphate load in the environment, but provide an overall cost savings to Idaho manufactures managing phosphate discharge. BHS currently sells ~$400,000 in cleaning and sanitation product to a customer-base that represents ~$5,800,000 in annual consumption, just within the state of Idaho. The scope and value of the eco-friendly product offering by BHS will be extended by the addition of the new products resultant from this HERC award. BHS is able to offer more product options manufactured locally. To the Idaho business customer, the availability of essential sanitation products manufactured within the region corresponds to a transportation cost savings.

5. Description future plans for project continuation or expansion:

We plan to optimize current separation techniques to better characterize product components. This may include methods such as two-dimensional chromatography, including size exclusion and/or ion chromatography to reduce the complexity of product mixtures and increase capability for characterization of the components. We also plan to continue reformulation of eco-friendly cleaning products and test these products using the standard efficacy testing procedure developed in our laboratory. In order to assess eco-friendly product formulations, based on their product-type (ie. acid cleaner, alkaline cleaner, and/or chlorinated-alkaline cleaner), we plan to modify the current pellet constituents to contain components specific to these cleaner types and then conduct the efficacy testing using a similar procedure as stated previously. In addition, we plan to analyze the chemical composition of efficacy test residues, both aqueous and solid portions, to obtain valuable information about the functionality, health, and environmental concerns in the cleaning process. This type of analysis relies heavily on separation and characterization techniques and will require expansion of instrumentation capabilities. To continue the project, we will seek funding through any and all means possible.

6. Please provide a final expenditure report (attached) and include any comments here:

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

8. Any other pertinent information:

FINAL EXPENDITURE REPORT

A. FACULTY AND STAFF		
Name/Title	$ Amount Requested	Actual $ Spent
Owen McDougal, Associate Professor	6,800.00	6,800.00

B. VISITING PROFESSORS		
Name/Title	$ Amount Requested	Actual $ Spent

C. POST DOCTORAL ASSOCIATES/OTHER PROFESSIONALS		
Name/Title	$ Amount Requested	Actual $ Spent

D. GRADUATE/UNDERGRADUATE STUDENTS		
Name/Title	$ Amount Requested	Actual $ Spent
Emily Drussel, Graduate Student	22,000.00	19,500.00
Petr Malek, Undergraduate Student	4,000.00	1,000.00

E. FRINGE BENEFITS		
Rate of Fringe (%)	$ Amount Requested	Actual $ Spent
Average fringe requested 11% - actual spent 8%	3,600.00	2,144.22
PERSONNEL SUBTOTAL:	**36,400.00**	29,444.22

F. EQUIPMENT: (List each item with a cost in excess of $1000)		
Item/Description	$ Amount Requested	Actual $ Spent
1.Gison HPLC ELSD (preparative evaporative light scattering detector)	0	8,899.38
2.		
3.		
4.		
EQUIPMENT SUBTOTAL:		

G. TRAVEL		
Description	$ Amount Requested	Actual $ Spent
1.		
2.		
3		
TRAVEL SUBTOTAL:		

H. PARTICIPANT SUPPORT COSTS:		
Description	$ Amount Requested	Actual $ Spent
1.		
2.		
3		
PARTICIPANT SUPPORT COSTS SUBTOTAL:		

I. OTHER DIRECT COSTS:			
Description		$ Amount Requested	Actual $ Spent
1.materials and supplies		5,000.00	3,082.08
2.graduate student tuition and fees		8,200.00	8,178.00
3.			
OTHER DIRECT COSTS SUBTOTAL:		13,200.00	11,260.08
TOTAL COSTS (Add Subtotals):		49,600.00	49,603.68
TOTAL AMOUNT REQUESTED:			49,600.00
TOTAL AMOUNT SPENT:			49,603.68