North Dakota Renewable Energy Program
Status Report

Recipient: Xiaodong Hou University of North Dakota/Institute for Energy Studies
Contract Number: R-035-044
Report for time period of: 3/1/2018-8/31/2018

Description of Project
Please provide a brief description of the project:

The overall goal of this project is to develop a low-cost synthetic procedure to prepare graphene-modified LiFePO4 (LFP/G) as a high-performance cathode material for LIBs at pilot scale (10 tons/year). To fulfill this goal, a two-step procedure is proposed: 1) humic acid is extracted and purified from low-rank ND coal or leonardite and 2) using the extracted humic acid as a key feedstock to in situ prepare LFP/G via a novel modified carbothermal reduction reaction.

Project Tasks
Please describe the progress on all project tasks achieved during the reporting period:

Five tasks have been defined to fulfill the above goals. In the past six months, the project is focused on the task 1 as planned in the time lines. The progress is outlined as follows:

1. The task 1 is completed and two humic acid extraction procedures were optimized and compared with the two key objectives of the highest yield and purity.
2. In the first approach—alkali extraction, a Taguchi orthogonal array design L9(34) with 4 factors at 3 levels and a total of 9 runs was implemented. An optimized procedure is determined with a balance between the purity and yield.
3. A solvent extraction procedure is also studied for comparison. A similar Taguchi orthogonal array design L9(3^4) with 4 factors at 3 levels and a total of 9 runs was implemented. An optimized procedure is determined with a balance between the purity and yield.
4. With all factors considered, the alkali extraction technique is superior to the solvent one and will be used for the rest of the project. The basic extraction technique is far superior in terms of percent yield, producing nearly 140% of the maximum amount of humic acid that can be obtained through solvent extraction. The solvent extraction does have a slight edge over the basic extraction method in terms of overall purity. However, the alkali extraction is again superior when it comes to reducing iron content, with a projected reduction to below 1%, while the solvent extraction method results in a final iron content of 1.24%.
5. To further reduce the iron content down to target value of 0.5% or less, a second purification step is used to treat the alkali extracted raw humic acid, now we can reduce the iron content down to only 0.2%. This primary result indicated that a simple alkali extraction plus a second purification treatment could produce the satisfactory humic acid for LFP/G preparation.
6. With the purified humic acid by Task 1 as a key feedstock, we just start the Task 2. This task and Task 3 will be focused on the optimization of the synthetical procedure of LFP/G.

Deliverables
Please describe the progress on project deliverables, as stated in your contract, achieved during the reporting period:

The major deliverable during this period is the optimized extraction and purification procedure that yields high purity (ash <1%) and low iron content of (0.2%) humic acid derived from leonardite. This deliverable is achieved on time as described above.

Expenditures

Please provide a breakdown of expenditures. Include all sources of match. Provide supporting documentation as a separate attachment.

EXPENDITURES FOR THIS REPORTING PERIOD ONLY				
Project Expense	**NDIC**	**REP Recipient**	**Other Sponsor**	**Total**
Salary & Fringes	36,670.81		30,112	66,782.81
Supplies	3,279.45		9,000	12,279.45
Fees	101.00		2,000	2,101.00
Indirects	15,620.07			15,620.07
Total	55,671.33		41,112	96,783.33

CUMULATIVE EXPENDITURES				
Project Expense	**NDIC**	**REP Recipient**	**Other Sponsor**	**Total**
Salary & Fringes	36,670.81		30,112	66,782.81
Supplies	3,279.45		9,000	12,279.45
Fees	101.00		2,000	2,101.00
Indirects	15,620.07			15,620.07
Total	55,671.33		41,112	96,783.33