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: 03/01/2019-08/31/2019

Description of Project
The overall goal of this project is to develop a low-cost synthetic procedure to prepare graphene-modified $LiFePO_4$ (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 2 and part of the task 3 as planned in the time lines. The equipment that was planned for task 4 has arrived. The equipment is being used by multiple projects and we will run our process soon. The progress is outlined as follows:

1. Task 2, optimization of LFP/G synthesis procedure is in the final stage, will be completed by October 15, 2019.
   - Minitab statistical software was utilized to design a series of experiments to optimize the LFP/G synthesis procedure. Experiments were set up as a Taguchi orthogonal array design L9(3^4), testing four factors at three levels each with a total of 9 runs.
   - Coin cells prepared with LFP/G DOE have exhibited high-rate performance, and substantial cycle life.
   - LFP/G synthesis procedure has been optimized on the general parameters. General parameters include sinter temperature, sinter time, and preheat temperature. The final parameter, percent humic acid is currently being optimized and will be completed soon.

2. Task 3, characterization of LFP/G cathode results:
   - XRD spectra of LFP/G samples continues to show >99% crystalline purity.
   - The carbon content of the LFP/G samples measured by TOC analyzer is slightly higher than the expectation. An optimal carbon content will be achieved by adjusting the humic acid feeding ratio.
   - Tap density of the LFP/G samples are comparable to commercial ones.
   - SEM imaging finds the LFP/G has well-formed primary particles.
   - Raman spectroscopy to be run on LFP/G samples to verify graphene formation.

3. Task 4, Pilot scale production has proven to be scalable based on optimized extraction procedures at lab scale.
   - Samples from the 0.5 kg humic acid sample have been purified and cells prepared using the precursor have exhibited high rate capacity and stable cycling.
   - Equipment is now available for the pressure-driven filtering step. Equipment is being used by multiple groups at IES and will be used soon for Humic Acid preparation.

4. Task 5, electrochemical performance testing is currently being conducted on cell prepared with our LFP/G cathode material.

- Cells continue to exhibit good rate performance at high discharge rates, compared with commercial LFP/C.
- The specific capacity of cathode material remains at 148mAh/g. It is expected to increase past 150 upon further optimization of LFP/G synthesis procedure.

Deliverables

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

- Optimization of general parameters for LFP/G synthesis.
- Pilot scale humic acid (0.5 kg), after purification steps, shown to be viable for high-performance batteries.

To be achieved soon:

- 15% specific capacity (150 mAh/g) improvement over commercial products (120-125 mAh/g) and better cycle life.
- 1 kg pilot scale production of humic acid from leonardite using new equipment. This task is to be done by October 31.

Deliverables achieved during the previous period:

- Development of the procedure that can produce high-purity (>99%) and metal-free humic acid from leonardite. We have achieved the high purity (ash <1%) and low iron content of (0.2%) humic acid derived from leonardite.

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	53,037.28		36,096	89,133.28
Supplies	6539.92		8,000	8,539.92
Fees	8767.45		9,000	33,904.45
Indirects	26,723.17		17,284	42,531.17
Total	95,067.82		86,420	174,108.82

CUMULATIVE EXPENDITURES				
Project Expense	**NDIC**	**REP Recipient**	**Other Sponsor**	**Total**
Salary & Fringes	84,925.54		118,344.00	203,269.54
Supplies	14,240.06		19,000.00	33,240.06
Fees	17,786.10		36,137.00	53,923.10
Indirects	45611.59		33,092.00	78,703.59
Total	162563.29		206,573.00	369,136.29