

# 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: 9/1/2018-2/28/2019

### Description of Project

The overall goal of this project is to develop a low-cost synthetic procedure to prepare graphene-modified LiFePO<sub>4</sub> (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. But we adjust the schedule slightly according to the project progress and facility availability. For example, we proceed some work in Task 4 and 5 ahead of time. The progress is outlined as follows:

1. Task 2, optimization of LFP/G synthesis procedure is in the final stage, will be completed by March 30, 2019.
  - A series of LFP/G samples have been prepared. Coin cells prepared with these samples have exhibited increased high-rate performance, and specific capacity close to the target value.
  - Minitab statistical software was again utilized to design a series of experiments to optimize the LFP/G synthesis procedure. Experiments were set up as a Taguchi orthogonal array design L<sub>9</sub>(3<sup>4</sup>), testing four factors at three levels each with a total of 9 runs.
2. Task 3, characterization of LFP/G cathode has seen some results.
  - XRD spectra of the LFP/G sample shows expected >99% crystalline purity.
  - XRD spectra of LFP/G samples exhibited sharp peaks corresponding to highly graphitized carbon.
  - Under SEM, LFP/G samples had few carbon agglomerations, indicating the humic acid has formed thin, even coatings on the LFP particles.
3. Task 4, Pilot scale production has proven to be scalable based on optimized extraction procedures at lab scale.
  - A few kilogram humic acid was prepared at pilot scale using equipment for an adjacent project.
  - Separating liquids and solids via filtering was the single-most time consuming step. We have developed a novel staged and pressure-driven filtering that can mitigate this problem and the technology could have broad application in other similar fields.
4. Task 5, electrochemical performance testing is currently being conducted on cell prepared with our LFP/G cathode material.
  - Cells have exhibited good rate performance at high discharge rates, compared with commercial LFP/C.

- The specific capacity of the cathode material is currently around 148 mAh/g. This is expected to increase to 150 or more upon the completion of Task 2.

**Deliverables**

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

- Synthesis of high crystalline purity (>99%) LFP/G with graphene evenly distributed on the surface of LFP particles.

To be achieved soon:

- 15% specific capacity (150 mAh/g) improvement over commercial products (120-125 mAh/g) and better cycle life;

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.

<b>EXPENDITURES FOR THIS REPORTING PERIOD ONLY</b>				
<b>Project Expense</b>	<b>NDIC</b>	<b>REP Recipient</b>	<b>Other Sponsor</b>	<b>Total</b>
Salary & Fringes	31888.26		52,136	84,024.26
Supplies	7700.14		8,000	15,700.14
Fees	8767.45		9,000	17,767.45
Indirects	18888.42		17,284	36,172.42
<b>Total</b>	<b>67319.77</b>		<b>86,420</b>	<b>153,664.27</b>

<b>CUMULATIVE EXPENDITURES</b>				
<b>Project Expense</b>	<b>NDIC</b>	<b>REP Recipient</b>	<b>Other Sponsor</b>	<b>Total</b>
Salary & Fringes	68559.07		82,248	150,807.07
Supplies	10979.59		17,000	27,979.59
Fees	8868.45		11,000	19,868.45
Indirects	34508.49		17,284	51,792.49
<b>Total</b>	<b>122,991.10</b>		<b>127,532</b>	<b>250,523.10</b>