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November 17, 2017

Ms. Karlene Fine
Executive Director
North Dakota Industrial Commission
State Capitol – Fourteenth Floor
600 East Boulevard Avenue
Bismarck, ND 58505

Dear Ms. Fine:

Subject: University of North Dakota proposal entitled "Preparation of Graphene-Modified $LiFePO_4$ Cathode for Li-Ion Battery"

Enclosed please find two copies of the subject proposal entitled "Preparation of Graphene-Modified $LiFePO_4$ Cathode for Li-Ion Battery" which is being submitted to the NDIC Renewable Energy Council. Using Phase I/II funding provided under the Research ND program, the team successfully demonstrated they can consistently produce a low cost cathode material with a specific capacity of 120 mAh/g. This proposed work will further improve the performance of lithium iron phosphate batteries currently marketed by Clean Republic using graphene produced from North Dakota Leonardite, with a performance goal of 150 mAh/g. The target market is the energy storage to support renewable energy generation. Successful completion of this project will greatly expand the market of our commercial partner, Clean Republic, who currently produces small battery packs at its Grand Forks location. Enclosed is the $100 application fee.

If you have any questions, please contact me by telephone at (701) 777- 6791 or by e-mail at xiaodong.hou@und.edu.

Sincerely,

Xiaodong Hou
Principal Investigator
Institute for Energy Studies

Barry Milavetz
Associate Vice President for Research &
Economic Development

Renewable Energy Program

North Dakota Industrial Commission

Application

Project Title:

Preparation of Graphene-Modified $LiFePO_4$ Cathode for Li-Ion Battery

Applicant: University of North Dakota

Principal Investigator: Xiaodong Hou

Date of Application: November 17, 2017

Amount of Request: $238,866

Total Amount of Proposed Project: $486,238

Duration of Project: 2 years

Point of Contact (POC): Xiaodong Hou

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POC Address: 2844 Campus road, Stop 8153
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ABSTRACT

Objective:

Starting from May 2015 a Grand Forks-based private company Clean Republic (CR) sponsored the University of North Dakota (UND) Institute for Energy Studies (IES) to launch a collaborative research effort to develop its own lithium iron phosphate (LFP) cathode material for Li-ion batteries (LIBs) to overcome a severe quality inconsistency issue with commercial LFP. Under a recent award from the North Dakota Department of Commerce (Venture Grant II), the project team has reached their goal of producing low cost and high consistent LFP cathode with a specific capacity of 120-130 mAh/g (comparable to commercial cathodes but at much less cost). The success of the existing project will help Clean Republic differentiate their LFP product from their competitors in terms of quality consistency and cost. Based on the positive results of the project, to hold the leading position in this competitive industry, our team is proposing to further improve the technology to prepare a more advanced product - graphene-modified lithium iron phosphate cathode materials (LFP/G) at pilot scale (10 tons/year). *LFP/G has advantages over conventional LFP in many aspects including energy density, rate capability, cycling stability and low-temperature performance*. The target specific capacity of the LFP/G is 150 mAh/g, another 15% improvement over our current technology. The primary target market of this LFP/G cathode materials is Clean Republic's own need on its main products - 'Dakota Lithium' packs and 'HillTopper' E-bikes. The long-rang goal is to produce LFP/G at large scale and its target market is the large battery packs for energy storage to support intermittent renewable energy generation.

Expected Results:

The outcome is high-performance (150 mAh/g) and low-cost LFP/G cathode materials for Li-ion batteries and associated preparation processes. If successful, it would technically and economically transform the current technology of scalable synthesis of high performance LFP cathode. Technically, LFP/G LIBs displayed remarkably improved high energy density, good cycling stability, rate capability, and low-temperature performance over conventional LFP LIBs. Economically, the estimated cost of our LFP/G is about $11,000 per ton, in contrast to the estimated price of $36,000 by directly adding commercial graphene to LFP. A $9,000 per ton profit is estimated at the projected market price of $20,000 per ton (see **Appendix B** for details). This new LFP/G cathode production alone will generate revenue of **$2.4 million/year and 12 full time employees by 2021**, and **$8 million/year and over 40 jobs by 2023** for our commercial partner, Clean Republic, in Grand

Forks, North Dakota. In addition, given 83 tons ($1.66 million) of LFP/G used in its own LIB packs products by 2023, Clean Republic will expand its market share and generate up to **$8 million revenue from its LIB packs. This would offer 20 additional new jobs** in Grand Forks, North Dakota. *This project will result in production capability of high performance LFP/G cathode for supply to the LFP market as well as to Clean Republic's own products that can provide up to 60 new jobs in North Dakota by 2023. This project will also form the foundation for subsequent larger-scale production that could be used for energy storage applications such as for wind/solar power.*

Duration:
Two years (Suggested: Jan 1, 2018 – Dec 31, 2019)

Total Project Cost:
$238,366 requested from NDIC and $486,238 for total project cost. Clean Republic LLC is providing the matching funds to support the project in the amount of $247,873. However, Clean Republic is also currently pursuing external private investment in the company that, if it comes to fruition, will allow an additional $168,000 in matching funds for the proposed project. An alternate budget plan is provided as an appendix to this application (**Appendix F**), in the event that the additional funding is available. It is important to note, however, that the following proposal has been developed based on the total funding of $486,238, and that all project objectives can be obtained given that amount of funding. The additional funding, if available, would be used for equipment upgrades and additional product analysis.

Participants:
University of North Dakota Institute for Energy Studies and Clean Republic LLC

PROJECT DESCRIPTION

Objectives:
The overall goal of this project is to develop a low-cost synthetic procedure to prepare LFP/G for LIBs at pilot scale (10 tons/year). The ultimate goal is to produce the LFP/G at large scale and its target market is the large battery packs for energy storage to support renewable energy generation. *The anticipated specific capacity of 150 mAh/g represents a 15% improvement over current technology. Rate capability, low temperature and cycling stability of the LFP/G are also expected to be improved simultaneously.*

Methodology:
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) the extracted humic acid is then mixed with a Li source and $FePO_4$ to in situ prepare LFP/G via a novel modified carbothermal reduction reaction that has been well-established from previous development efforts by UND IES and Clean Republic. ***The detailed description of the proposed technology is confidential and attached as an appendix (Appendix A). Such information shall be used or disclosed only for the review and evaluation purpose.*** Such an in situ synthesized LFP/G has several advantages over other methods involving adding external graphene into LFP powder. These are summarized below:

Excellent dispersion of graphene: A critical problem with adding external graphene to a cathode is the reagglomeration of graphene that will not only block the diffusion of Li-ion, resulting in a heavy polarization and higher irreversible capacity, but also create extreme difficulty in coating the electrode composite on a current collector. A reagglomeration inhibitor like amorphous carbon must be added to the composite.[1] The reagglomeration problem will not occur in our approach, as the graphene is in situ synthesized on the surface of LFP particles by reducing the homogeneously coated humic acid.

Complete and uniform surface modification: As shown in **Figure 3**, pure graphene or reduced graphene oxide (rGO) is composed of dominant carbon atoms with very few functional groups. Because of the hydrophobic feature of graphene, only "dot-on-plane" contact can happen upon mixing with LFP particles. This poor contact disables graphene to fabricate an effective conductive network within the LFP particles, resulting in limited improvement in electrochemical performance.[2] On the contrary, in our proposed technology, $FePO_4$ particles are previously surface-modified by humic acid. The rich functional groups of humic acid can ensure a complete and uniform surface modification on the polar $FePO_4$ particles.

Improved Li-ion diffusion within the composite: Despite the fact the graphene can significantly improve the electronic conductivity, Wei et al.[3] shows a partial graphene wrapping LFP delivered better electrochemical performance than a full and tight graphene-wrapped LFP. The reason is a partial graphene wrapping can keep a balance between electron transportation and ion diffusion within the composite, while full graphene wrapping isolates LFP particles from the electrolyte and, thus, inhibits Li-ion diffusion to some extent, resulting in large charge transfer resistance. Their approach to obtain the partial graphene wrapping is to add a second carbon source like sucrose or glucose. Alternatively, to overcome this challenge, recent research[4] began to prepare graphene sheets with pores to surface-modify LFP particles. Those pores can shorten the distances of Li-ion diffusion, thereby quickening the Li-ion transportation during the charge–discharge process, ultimately achieving the improvement of Li-ion diffusion and electron conductivity simultaneously. Featured by complexity of its chemical structure, humic acid will generate a naturally porous coating instead of a full and tight wrapping on the LFP particles, resulting in improved electron conductivity and Li-ion diffusion simultaneously.[5] The improvement of Li-ion diffusion will result in better charging and low-temperature performance on LIBs.

R&D Plan

Task 1. Optimization of Humic Acid Extraction Procedure: In this task, we will optimize our procedure to extract high-purity and high molecular weight humic acid from leonardite economically. ***The detailed description of this task is confidential and attached as an appendix (Appendix A).***

Task 2. Optimization of LFP/G Synthetic Procedure. With the backbone of the procedure established in our previous development efforts, this task focuses on optimizing process parameters to yield high content of graphene and thus improve the specific capacity of the cathode materials to ≥150 mAh/g. We will focus on the following parameters: precursors feeding ratio, solid content in the slurry, preheating temperature and time, and sintering temperature and time. The software Minitab will help optimize and investigate the interaction between the parameters. A Taguchi orthogonal array design $L8(2^7)$ with 6 factors at 2 levels and a total of 8 runs will be implemented. A second run of design of experiments will be run to further optimize levels of major factors. The ratio of graphene to disordered carbon (I_G/I_D on Raman Spectra) and the specific capacity of the cathode will be used as the evaluation standard.

Task 3. Characterization of LFP/G Cathode. In this task, a series of analytical techniques will be applied to confirm: 1) graphene is really in situ-formed on the surface of LFP particles, and 2) the LFP/G powder meets the physical requirements of a LIB cathode with the details as follows:

- Graphene will be confirmed by a Raman spectrometer at UND. The morphology of graphene on the LFP particles will be observed under a high-resolution transmission electron microscope (HRTEM) and/or field emission scanning electron microscope (FEG-650, FEI, USA) at IES. Crystal purity will be measured by a Smartlab XRD (Rigaku, Japan) available at IES. Carbon content will be tested by a TOC (total organic carbon) analyzer (Shimadzu, Japan) at IES. Tap density will be measured following the ASTM B527-15, with the apparatus available at IES. Trace impurities of LFP/G powder will be measured by a Supermini 200 XRF (Rigaku, Japan) at IES. Particle size distribution of LFP/G powder will be measured by a Marvin ZS90 zeta Sizer at UND.

Task 4. Pilot scale production testing. This task is to evaluate the reproducibility of the optimized procedure at pilot scale. The scale of the LFP/G production will be gradually increased from 20g, 1 kg, to 30kg per batch, and at each scale multiple (>3) production batches will be repeated to evaluate the quality consistency. Based on the feeding ratio of humic acid for LFP/G production (≤10%), the equipment for lab-scale humic acid extraction can produce sufficient amount for pilot scale production of LFP/G.

Task 5. Electrochemical Performance Testing of LFP/G Cathode. This task is to confirm that the LFP/G has a better electrochemical performance than its commercial LFP counterparts with the details as follows:

- Electrochemical performance testing will be conducted on a Neware CT-3008 battery testing system (Neware Technology Limited) available at Clean Republic. CR2032 coin-type cells (for lab-scale sample) and full size 18650 or 26650 cells (for pilot-scale sample) will be prepared by Clean Republic. The equipment for the full size cells fabrication needs to be purchased, and all the other equipment are currently available at Clean Republic. Current densities and specific capacities will be calculated based on the mass of the target compound of the electrode. Cycle life will be evaluated by using a three-electrode system (ECC-REF, EL-Cell GmbH, Germany) for cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) tests available at Clean Republic.

Anticipated Results:

- High-purity (>99%) and metal-free humic acid extracted from ND leonardite
- High crystalline purity (>99%) LFP/G with graphene evenly distributed on the surface of LFP particles
- Specific capacity 150 mAh/g, a 15% improvement over commercial ones (120–125 mAh/g)
- 60% reduction in cost of LFP/G compared to existing methods of adding external graphene into LFP
- Successful upscale of the technology to a pilot scale (up to 30 kg/batch)

Facilities & Resources:

The facilities, equipment and resources for the proposed work are all available (see appendix for details).

Techniques to Be Used, Their Availability and Capability:

The idea to improve battery performance of LFP by graphene is not new, but *the existing research on the preparation of LFP/G normally requires the synthesis of graphene in advance, which severely inhibits practical applications of those processes. This is because of the prohibitively high price of reduced graphene oxide (rGO), a necessary material for those processes, at up to $50,000/kg.*[6-9] *Our unique in-situ synthetic technology takes advantage of an abundant and low-cost raw material (North Dakota Leonardite), combined with a simple manufacturing process (competing technologies require multiple steps that add complexity/cost), to produce LFP/G cathode materials with 15% more capacity than those on the market today, even at a lower cost.* Our procedure has at least three technical advantages over methods involving externally adding a commercial graphene into LFP powder:

- It allows for excellent dispersion of graphene in LFP that overcomes the critical reagglomeration problem of graphene;
- It provides a complete and uniform surface modification on LFP particles as compared to a "dot-on-plane"-type surface modification using conventional methods;

- It improves Li-ion diffusion within the composite cathode in a similar mechanism to a porous graphene-modified LFP, which is crucial to fast charge/discharge and low-temperature performance of the LIBs.

The availability of our technology and its current state are demonstrated in the following:

1) *Preliminary Experiments – Humic Acid Extraction:* Several potential routes to extract high-purity humic acid from leonardite have been tried and compared. As shown in **Table 1**, the obtained humic acids have much lower ash content than the raw materials, and the main residual impurities are iron and sulfur. Further purification will be executed to remove those impurities as detailed in the methodology section (see the **Appendix A**). For instance, only by using a lixiviating agent via a non-optimized procedure, we are able to reduce the iron (the major unwanted impurity) content to 0.2%. More importantly, *using the humic acid as feedstock, we have successfully prepared LFP powder of the desired olivine crystal structure as shown in* ***Figure 1***, and the only trace insulating impurity Li_3PO_4 can be avoided by precisely controlling the stoichiometry of starting materials. This preliminary result strongly indicates the feasibility of our proposed technology, although the formation of graphene needs to be confirmed and the process hasn't been optimized yet.

Table 1. The Chemical Composition of Leonardite and Extracted Humic Acid by Different Methods

Composition (dry basis %)	***Raw Leonardite***	***HA by Alkali***	***HA by Solvent***
Na	0.15	–	–
Mg	0.71	0.03	0.02
Al	1.58	0.04	0.04
Si	2.11	0.11	0.08
S	1.04	0.46	0.46
K	0.18	–	0.04
Ca	0.80	0.02	0.04
Fe	2.55	0.75 **0.20***	1.20
Ba	0.19	–	–
Ash Content	24.50	2.39	2.15
Organic Matter	75.50	97.61	97.85

*After treated with a lixiviating agent

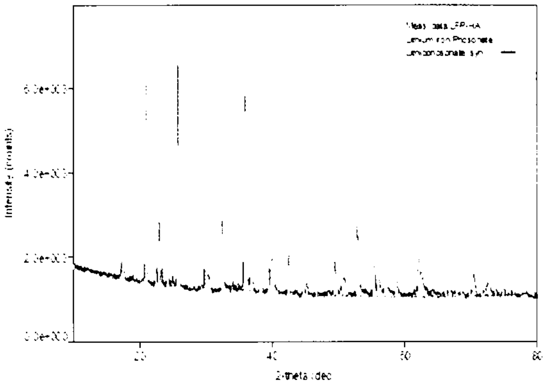

Figure 1. X-ray diffractogram of our LFP/G. The measured data(red) matches well with the standard LFP data (green) of the desired olivine structure.

2) *An Existing LFP Cathode Development Project:* An existing project sponsored by North Dakota Department of Commerce to improve the quality consistency of the LFP/C (C stands for non-graphited amorphous carbon) cathode is close to its Phase II goal – pilot-scale production of 10 tons/year. An optimized synthetic procedure has been finalized and tested, and its reproducibility has been proven at one kilogram-scale as at 20 gram-scale in terms of two key performance metrics: 1) high purity and quality consistency of LFP/C powder and 2) high specific capacity and cycling consistency of the LFP/C powder. **Figure 2** (left) shows our LFP/C has an expected olivine crystal structure (triphylite) with a purity >99.5% by Rietveld analysis and excellent reproducibility demonstrated by the consistent XRD diffractograms of multiple samples. The graph (right) shows our latest LFP/C has a specific capacity of 130 mAh/g, which is among the top levels of commercial products such as Phostech's. In addition, the specific capacity of our LFP keeps consistent after 100 cycles (even slightly increased after 75 cycles), in contrast to a 13% and 24% decrease for Phostech LFP (used by Clean Republic now) and a nano LFP (used by Clean Republic LLC in the past), respectively. All the above data are based on non-graphited amorphous carbon-modified LFP. Considering the excellent properties of graphene, it is reasonable to claim the overall battery performance of proposed LFP/G LIBs will be improved

significantly. Therefore, we are confident setting objectives of specific capacity at 150 mAh/g, which is only a 15% improvement on the current state.

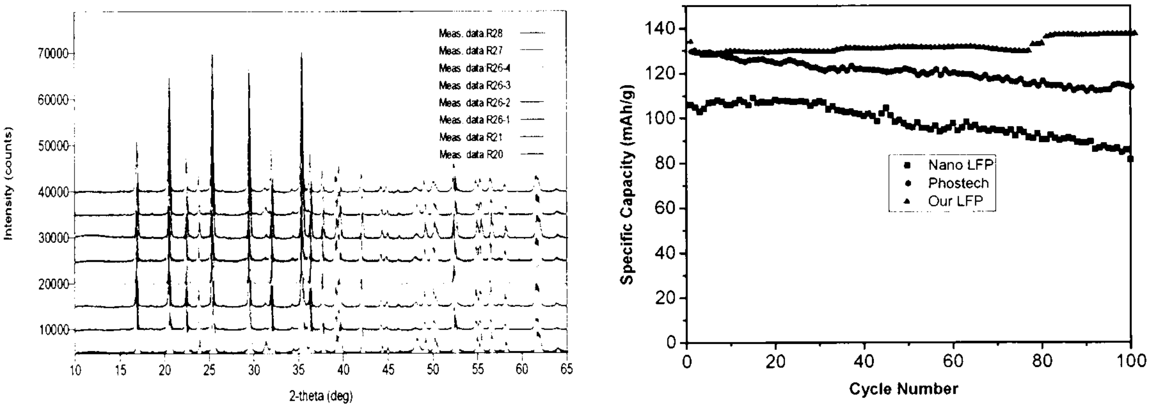

Figure 2. Left: XRD profiles of LFP/C samples from different runs and within the same run. Right: Specific capacity and cycling consistency comparison of our LFP/C and two commercial ones.

3) <u>The difference between the existing and the proposed project</u>

- We are in the final stage of our ND Department of Commerce grant. The results from this grant are successfully demonstrating that we can indeed economically produce consistent, high quality LFP/C cathode material, the original goal of the proposed effort.
- The final aspects of the existing project that are in progress include high purity (>99%) LFP powder with well-controlled particle size distribution (D_{50}<2 um and D_{90} <10um), better than proposed battery performance (130 mAh/g vs 120 mAh/g proposed), the ability to reproduce the same high quality of LFP at one kilogram-scale as at 20 gram-scale, only one-step away to the proposed final goal of pilot scale (10-kilogram level), and establishment of our own capacity to assemble and test coin-type cells beyond the proposed scope of work.
- These results in themselves will help Clean Republic differentiate their product from their competitors. In fact, the exciting results from the existing project has attracted strong interests from Aifloo AB, a private investment firm (see attached support letter). The work proposed to the Renewable Energy Council will make another step-change improvement in their battery technology through the addition of graphene.
- The goal of the proposed project is to significantly improve the battery performance of LFP LIBs by preparing LFP/G cathode, including high energy density, good cycling stability, rate capability, and low-temperature performance.
- The proposed scope of work and project objectives are completely different than the existing project. There is no duplication of effort/funding, but rather complementary projects.

Environmental and Economic Impacts while Project is Underway:

We don't anticipate any environmental impacts when the project is underway. The method we used to extract humic acid from leonardite is recommended by the International Humic Substance Society(IHSS) and EPA. Only very dilute alkaline, acid and/or a small amount of recyclable organic solvent acetone is involved. The modified carbothermal reduction reaction we develop doesn't generate any environmental hazards. All existing UND procedures will be followed regarding waste disposal.

An important advantage of the proposed project is its significant economic impacts on our commercial partner while project is underway. Even pilot scale LFP production of 30 kg/day or 10 tons/year can generate substantial benefits to Clean Republic, as it can be directly used on its two main products—Dakota Lithium packs and E-bikes. Both are seeing quick growth in the next few years. Clean Republic's own sales channels already created a demand for more than 20 tons of LFP powder in 2017, and that demand has been growing by 20%–30% per year since the company started in 2009.

Ultimate Technological and Economic Impacts:
This new LFP/G cathode production alone will generate revenue of $2.4 million/year and 12 full time jobs by 2021, and $8 million/year and over 40 jobs by 2023 in Grand Forks, North Dakota. In addition, given 83 tons ($1.66 million) of LFP/G will be used in its own LIB packs product - Dakota Lithium by 2023, Clean Republic will enlarge its market share in the United States and generate up to $8 million revenue from Dakota Lithium. This would offer 20 additional new jobs in Grand Forks, North Dakota by 2023. These data were validated by a detailed market analysis and Clean Republic's business plan as attached. The successful commercialization of this LFP/G will also have broader technological and economic impacts:

- A high-performance and low-cost cathode material and associated Li-ion batteries will meet the rapidly increasing demand in transportation technology and energy storage technology, among others;
- The high-performance cathode material and associated Li-ion battery packs made in USA will promote innovative products and technologies powered by Li-ion batteries;
- This project will inspire new research ideas and facilitate existing research projects on efficient utilization of the abundant low-rank coal and related resources in the United States. For instance, the University of North Dakota Institute for Energy Studies (IES) is currently conducting a DOE-awarded project on extraction of rare-earth elements (REEs) from lignite coal in which the carbonaceous residue after extraction can be used as even better feedstock than raw coal for this project; therefore, these two projects complement and reinforce each other. *In addition, although it is not the final target of this project, the high purity of humic acid itself is a high value-added product with broad applications such as nutritional supplement for human health[10], which is why the our Leonardite supplier ND Leonardite Production LLC is so excited about this project as well (See the support letter).*

Why the Project is Needed:
$LiFePO_4$ is a specifically important cathode material because of its intrinsic crystal structure (orthorhombic olivine) and chemical stability that leads to excellent safety and superior long cycle life, but its rate capability and low-temperature performance still need to be improved. Since its discovery in 2004,[11-12] graphene has been considered an ideal material to make composite electrodes to improve the overall performance of LFP because of its high charge carrier mobility (200,000 $cm^2V^{-1}s^{-1}$),[13] high theoretical surface area (2630 m^2g^{-1}),[14] a broad electrochemical window, and other remarkable properties.[15-17] Recent reports have demonstrated that graphene-modified LFP cathode (LFP/G)-based LIBs displayed remarkably improved electron transportation, which is quite significant to achieve high energy density, good cycling stability, rate capability, and low-temperature performance. For instance, using electrochemical exfoliation technology, Hu et al.[18] prepared a few-layer graphene-coated LFP cathode that delivers a capacity of 208 $mAhg^{-1}$, which is beyond the theoretical capacity 170 $mAhg^{-1}$. The excess capacity, according to their explanation, is attributed to the ultra-high capacity of the graphene flakes (>2000 $mAhg^{-1}$), which was revealed by the layer-to-layer distance change during the charge and discharge process. Luo et al.[19] prepared a graphene-encapsulated LFP nanocomposite through a self-assembly approach, and the resulting LFP/G cathode demonstrated excellent cycling stability: the capacity retention of 70% at 50 C rate and less than 8.6% capacity loss after 950 cycles at 10 C. Ma et al.[20] in situ synthesized LFP/G nanoplates using ferrocene as the catalyst during the thermal treatment. Surprisingly,

most of the exposing facets are the (010) crystal face that is beneficial to the fast lithium ion diffusion along this direction, resulting in superior electrochemical performances at high rates (100 C) and low temperature (−20°C) because of the fast electron transport through the highly graphitic carbon layer and quick lithium ion diffusion through the thin nanoplates. *The few in-situ synthetic technologies for LFP/G either need advanced equipment and expensive starting materials or complicated catalytic systems and strict reaction conditions, and thus none of them are possible for mass production. While the most existing research on the preparation of LFP/G requires the synthesis of graphene in advance, which severely inhibits its practical applications, as cost-effective production of graphene at large scale is still a big challenge. Therefore, a new technology for LFG/G synthesis that overcomes all these challenges is highly desired.*

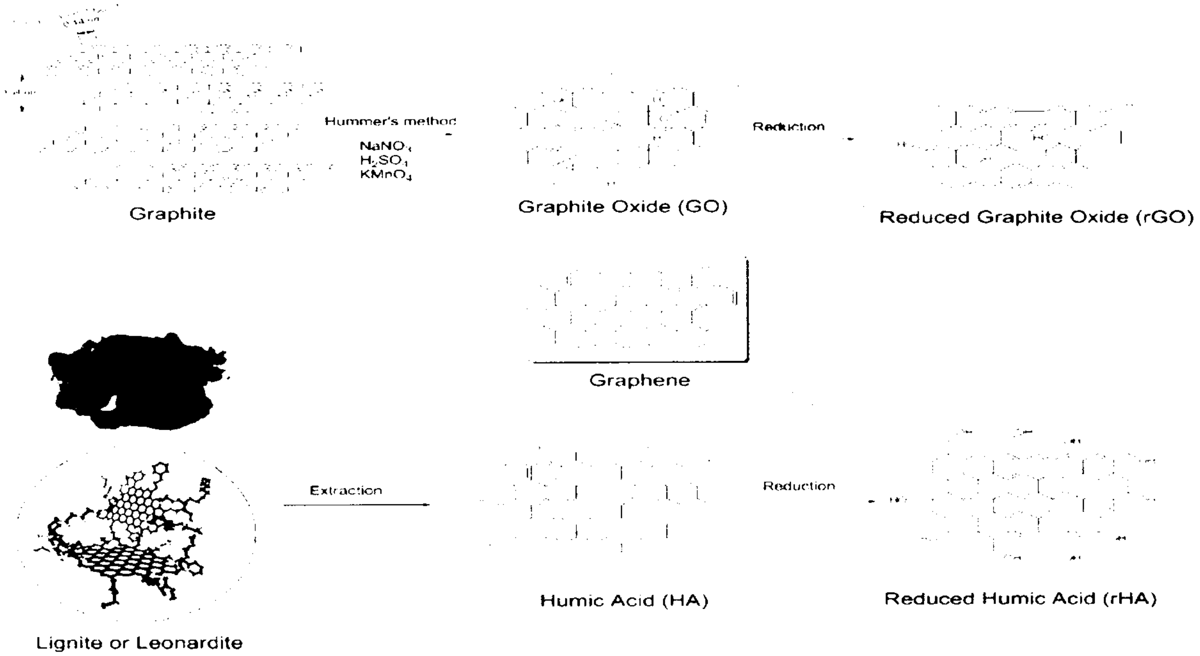

Figure 3. Scheme of two different routes for graphene preparation.

Currently, mainstream efforts to scalable, synthesized graphene use graphite as raw materials by a chemical exfoliation and reduction method via a classic[21] or modified Hummers' approach: graphite → graphite oxide → reduced graphene oxide (rGO),[22] as shown in **Figure 3**. Apart from the harsh reaction conditions and hazardous environmental impact, the rapidly increasing price of graphite are driving researchers to seek alternative raw materials and low-cost procedures for scalable synthesis of graphene. A promising alternative is coal: one of the most abundant natural carbon resources. Humic acid is the major organic component of coal. Chemically, humic acid is not a single molecule but a complex mixture of thousands of organic compounds with a wide range of molecular weight from several hundred to millions. The dominant carboxyl and phenolate groups enable humic acid to form complexes with common cations such as Mg^{2+}, Ca^{2+}, Fe^{2+}, and Fe^{3+} to create humic colloids.[23] Humic acids are commonly used as a fertilizer or soil supplement in agriculture and less commonly as a human nutritional supplement.[10] Among all of the sources of humic acid, coal, and in particular low-rank coal like lignite or naturally oxidized lignite (leonardite), is the most abundant and commercialized source. Despite the occurrence association and structure similarity, leonardite and lignite are significantly different in oxygen and ash content, and very importantly, leonardite has much higher humic acid than lignite. North Dakota leonardite (named after A.G. Leonard, the first director of the North Dakota

Geological Survey) has the highest humic acid (up to 86% dry and ash-free basis) content among all sources worldwide.[24]

Recently, Beall et al.[25-27] extracted humic acid (HA) from lignite or lignite oxide (leonardite) and used this extract instead of graphite as a starting material to produce graphene via a reduction step, as shown in **Figure 3**. Their results show that *HA extracted from leonardite matches the characteristics of graphite oxide but is much less expensive than graphite oxide produced via Hummers' method. More importantly, the reduced humic acid (rHA) is also comparable to rGO in terms of chemical composition (C/O ratio and I_G/I_D ratio) and physical properties such as electrical resistance and visible light transmission.* The rHA demonstrated some potential applications such as nanofiller for polymers[27] and humidity sensor.[28] Unfortunately, to prepare the rHA, an expensive catalyst ruthenium/H_2 is adopted as well as harsh reaction conditions in a Parr reactor at high temperature and pressure, and then an extra step is needed to remove the catalyst. Compared to its counterpart rGO from graphite, these many steps will trade off the cost advantage of rHA in raw materials. As a consequence, the overall performance price ratio of their rHA has negligible advantages over rGO from graphite. Therefore, a low-cost synthetic procedure for high performance graphene modified LFP is highly desirable.

STANDARDS OF SUCCESS

The success of this project will be evaluated by following measurable deliverables:

- Development of the procedure that can produce high-purity (>99%) and metal-free humic acid from leonardite;
- Synthesis of high crystalline purity (>99%) LFP/G with graphene evenly distributed on the surface of LFP particles;
- 15% specific capacity (150 mAh/g) improvement over commercial products (120–125 mAh/g) and better cycle life;
- The production cost of LFP/G lowered by 60% compared to methods involving adding external graphene into LFP;
- Ability to successfully demonstrate pilot scale production (up to 30 kg/day)
- 20 new jobs within Clean Republic to expand its current product line and 30 new jobs for the production and sale of the new cathode material by 2023.

BACKGROUND/QUALIFICIATIONS

This project will capitalize on UND IES expertise in two main areas: i) its unique, deep understanding of the geology and geochemistry of North Dakota leonardite, lignite; ii) its chemical engineering and process development expertise, and specifically the experience and knowledge specific to the proposed technology. Dr. Xiaodong Hou will serve as the principal investigator for the project. Dr. Hou is a material chemist with over 12 years' experience in synthesis and characterization of advanced functional materials. He has published over 20 peer-viewed publications in the field of chemistry materials and holds five patents. He is also serving as the principal investigator for the above-mentioned LFP development project. Dr. Michael Mann will serve as project director. As Executive Director of the UND IES, he is responsible for coordination of all projects within IES. Dr. Mann will work with the project team to ensure all personnel, equipment, and other resources are available to conduct the project efficiently. Dr. Mann has over 35 years of experience in the energy field, with much of his activity having focused on system integration. He has over 10 years' experience in the development of fuel cell technology.

This project also draws on the experience at Clean Republic in clean energy technology, especially in LIBs, and the successful commercialization record of its trademark products Dakota Lithium and "HillTopper" E-bike. As a cofounder of Clean Republic, Dr. Yong Hou will serve as co-principal investigator for this project. Dr. Yong Hou holds Ph.D. and M.S. degrees in systems engineering and has over 15 years of experience in the lithium battery industry. In particular, he developed at least fourteen battery cells and packs products that have been applied successfully in the marketplace, which includes a bicycle battery being applied in Clean Republic's flagship product – Hilltopper; a square shape, polymer cells, and relevant packs being applied in Neosonic's golf cars, a LFP pack for Rapala's ice fishing auger, a universe LIB pack named 'Dakota Lithium', etc. Dr. Yong Hou is serving as a Co-PI on the ongoing project of developing LFP. He also has several peer-viewed publications in the field of renewable energy systems and batteries. Mr. Michael Shope, cofounder and CEO of Clean Republic SODO LLC, will serve as a business advisor. Mr. Shope holds a Bachelor's degree in Aeronautics with an emphasis in Commercial Aviation (track in Entrepreneurship) from UND. He has extensive experience in product development, marketing, and management. Mr. Shope was honored as "SBA Regional Young Entrepreneur of the Year 2011" and guides the administrative, legal, and structural development of the business. Clean Republic was the winner of an "Innovate ND Venture Competition" in 2010.

Dr. Mo, CEO/Owner of Boston Global Technologies LLC and CEO of Guangxi Nowphene Energy Storage Science and Technology Ltd., will serve as a technical consultant pro bono on this project, in exchange for the priority of being the supplier of the key starting material $FePO_4$. Dr. Mo is a leading expert in the synthesis of $FePO_4$ and LFP cathode battery active materials and holds over ten patents in this field. Guangxi Nowphene Energy Storage Science and Technology Ltd. is the top manufacturer and market-share leader of battery-grade $FePO_4$ in the world with annual production capacity over 10,000 tons.

MANAGEMENT

Dr. Xiaodong Hou will serve as the project PI and the contact person for UND and will be responsible for coordination of project activities. Dr. Michael Mann will serve as Co-PI and will work with the project team to ensure all personnel, equipment, and other resource are available to conduct the project efficiently. Dr. Yong Hou will serve as a Co-PI and contact person for Clean Republic. Dr. Yong Hou will be responsible for evaluation of LFP/graphene battery performance and coordinating communication with the UND team, technical consultant Dr. Mo, and other participants. Mr. Michael Shope will serve as a business advisor. Once awarded, the following items will be addressed throughout the duration of the project: 1) Monitor project scope, schedule, cost, and risk; 2) Update project plans periodically to reflect changes in scope, schedule, cost/risk; 3) Provide quarterly technical reports, participate in meetings, and present at conferences as required.

TIMETABLE

This project has a duration for 2 years tentatively starting on Jan. 1, 2018 with the timetable and milestones as below:

Table 2. The proposed timeline of the project

No.	*Task*	*Q1*	*Q2*	*Q3*	*Q4*	*Q5*	*Q6*	*Q7*	*Q8*
Task 1	*Optimization of Humic Acid Extraction Procedure*	■	■						
Task 2	*Optimization of LFP/G Synthetic Procedure*			■	■				
Task 3	*Characterization of LFP/G Cathode*			■	■	■	■		
Task 4	*Pilot Scale Production Testing*					■	■	■	
Task 5	*Electrochemical Performance Testing of LFP/G Cathode*				■	■	■	■	■

Table 3. The milestones of the project

[redacted]	[redacted]	[redacted]
1	Achieve high purity (>99%), low ash content (<1%) and iron-free humic acid at 20g scale	5/30/2018
2	**Determination of the optimized humic acid extraction and purification procedure**	**6/30/2018**
3	Confirmation of the successful in-situ synthesis of LFP/G at 20g scale	9/31/2018
4	**Achieve specific capacity 150 mAh/g on coin-type cells**	**11/30/2018**
5	Determination of the optimized LFP/G synthetic procedure at 20g scale	12/31/2018
6	**Completion of pilot scale testing at one-kilogram scale**	**4/30/2019**
7	Completion of pilot scale testing at ten-kilogram scale	8/31/2019
8	**Completion of battery test on full-size cells**	**12/15/1900**
9	Completion of the project and final report	12/31/2019

BUDGET

The amount of $238,366 is requested from the NDIC Renewable Energy Program for the proposed project. The total match from the private company Clean Republic is $247,873, or 51% of total project cost. As detailed in the Abstract section of this document, an alternate budget plan is provided as an appendix in the event that Clean Republic is able to support an additional $168,000 in matching funds for the proposed project.

Personnel salary estimates are based on the scope of work. The labor rate used for specific personnel is based on their current salary rate. Average labor rates are used for general labor categories. The time, in months, each person will work on this 2-year project is: Xiaodong Hou – 6; one graduate student – 12; two undergraduates – 6 each; Yong Hou – 6; Michael Shope – 4; and one technician – 8. Michael Mann will be spending a part of his academic time towards the project but will not be tracking and reporting his effort as cost share.

Fringe benefits are estimated for proposal purposes only, on award implementation, only the true cost of each individual's fringe benefit plan will be charged to the project. Fringe benefits are estimated at a rate of 46% of total salary for PI at UND and 35% for Clean Republic personnel based on historical averages.

Table 4. The budget for the proposed project

Project Associated Expense	**NDIC's Share**	**Applicant's Share (Cash)**	**Applicant's Share (In-Kind)**	**Other Project Sponsor's Share**
Direct Cost-Personnel Salaries				
Michael Mann				
Xiaodong Hou	$ 29,652			
Graduate Research Assistant	**$ 21,600**			
Undergraduate Research Assistant	$ 14,400			
Yong Hou				**$ 43,800**
Michael Shope				$ 37,600
Technician				**$ 38,080**
Direct Cost-Fringe benefits	$ 13,640			$ 41,818
Direct Cost-Travel				$ 9,000
Direct Cost-Materials and Supplies	$ 23,000			$ 10,000
Direct Cost-Analysis and Test	**$ 35,000**			**$ 18,000**
Direct Cost-Equipment	$ 37,000			
Indirect Cost	**$ 64,074**			**$ 49,575**
Total	$ 238,366			$ 247,873
Total cost	**$ 486,238**			
NDIC Share	$ 0.49			

Dr. Yong Hou will travel to attend the Annual International Lithium Battery conference. $3,000 funding will cover the conference registration cost, the flight ticket based on the current rate, and all the other traveling expense. Michael Shope and Dr. Yong Hou will have international travel to China to meet with the technical consultant and cell manufacturer partner with a cost of $3000 each. The total expense of travel by Clean Republic is $3,000*2+$3,000*1=$9,000.

Materials and supplies are estimated at $23,000 in total with breakdown as follows: raw materials and chemicals $16,000; stock gas bottles $2,500; sample containers $1,500; lab consumables $2,000; and office supplies $1,000. Clean Republic will offer $10, 000 to cover a part of the cost for raw materials and chemicals at pilot scale production.

The total analysis and test cost is $53,000 (=$35,000 + $18,000). This estimate is generated by using the standard equipment and instrument fees based on 100 test specimens. The actual rates for each equipment and instrument will be charged to the project. The breakdown is as follows: X-ray Diffractometer $8,000, Scanning Electron Microscope $5,000, TOC analyzer $3,000, X-ray Fluorescence Spectrometer $4,000, Atomic Absorption Spectrometer $3,000, Raman Spectrometer $5,000, High Resolution TEM $4,000, and DSL particle size analyzer $3,000. Clean Republic will offer $18,000 to cover all cell assembly materials and supplies ($6,000), and battery performance testing ($12,000) for both coin-type cells and full size cells.

To meet the demand of making full-size (18650 and 26650) LFP cells, the project team intends to set up a line of R&D scale cylindrical cell fabrication. The necessary equipment includes electrode sheet preparation, cell assembly, case sealing and battery testing. **Table 5** displays the list of necessary equipment and the estimate of budget required.

The indirect cost rate included in this proposal is the federally approved rate 39% for the University of North Dakota, and 25% for Clean Republic calculated based on its annual overhead cost in the previous 3 years. Indirect cost is calculated based on the Modified Total Direct Cost (MTDC), which includes all direct costs minus equipment items > $5,000 and subcontracts in excess of the first $25,000.

Table 5. The cost estimate for the major equipment to be purchased in the proposed project

Item	*Cost*		*Justification*	*Comment*
Atmosphere furnace	$	-	Sinter cathode and anode materials	existing at UND lab
Planetary ball mill	$	-	Grind and homogenize the sintered materials	existing at UND lab
Vacuum mixer	$	3,000	Mix slurry	
Coater with drying oven	$	10,000	Coat slurry onto current collector and dry it	
Rolling press	$	5,000	Compress electrode to proper thickness	
Slitting machine	$	2,000	Slit electrode to proper size	
Ultrasonic metal welder	$	4,000	Weld tap/layer of current collector	
Vacuum oven	$	2,000	Dry electrode under vacuum	
Welding machine	$	3,000	Weld anode, separator and cathode to cylinder	
Grooving machine	$	3,000	Groove the case to fix the cell core	
Glove box	$	-	Inject electrolyte and seal cell cases	existing at UND lab
Electrolyte dispenser	$	500	Inject electrolyte into cell cases	
Degassing chamber	$	3,000	Disperse and degas electrolyte	
Crimping machine	$	1,500	Crimp cell in glove box after electrolyte injection	
Battery analyzer	$	-	Test electrochemical performance of battery cell	existing at CR
Total	$	**37,000**		

* Data resource: MTI Corporation[29]

CONFIDENTIAL INFORMATION

The confidential information is attached as an appendix. Such information shall be used or disclosed only for the review and evaluation purpose.

PATENTS/RIGHTS TO TECHNICAL DATA

The technology developed in the previous collaborative research funded by Clean Republic and the North Dakota Department of Commerce, and the additional new technology being developed in the proposed work will be co-owned by Clean Republic and UND. A detailed intellectual property agreement between Clean Republic and UND is already available on an ongoing collaborative project. Only minor modification is needed to generate a new one upon the notification of the award recommendation. In certain cases, our unique understanding that we would gain from our synthesizing and testing efforts will lead to new procedure design/operation for which we will file domestic and foreign patent applications as necessary.

APPENDICES

APPENDIX A:

CONFIDENTIAL INFORMATION

We are proposing an in situ synthetic technology that integrates the reduction of humic acid to graphene (or called reduced humic acid [rHA]) into the synthesis of LFP cathode materials via a modified carbothermal reduction route (CTR) as shown in **Figure A1**. Specifically, *humic acid extracted from leonardite is mixed with two pulverized precursors $FePO_4$ and LiOH in water, and then the resulting slurry is introduced into a furnace with atmosphere control. The slurry is first dried at 70-105°C under reduced pressure and then heated at 300-350°C in the furnace for a certain period, followed by a sintering step at 600°–800°C for 4–12 h under a reducing atmosphere Ar/H_2.* The simplicity, controllability and reproducibility of our unique slurry-heating technology has been well proven in our current project funded by North Dakota Department of Commerce. LFP sample performance shown previously in **Figure 2** are made through this technology. The key components of our technology are summarized as follows.

Extraction of Humic Acid: A key to the success of the proposed technology is extracting high purity and high molecular weight humic acid from leonardite. Humic acid extracted by conventional approaches, such as alkali extraction, always contains a small amount of ash, in particular metal impurities, that is not a problem for its common application as a fertilizer or soil supplement, but can cause a deadly problem to a Li-ion battery — resulting in failure of the battery. Therefore, the ash content and metal impurities need be minimized. Besides high purity, high molecular weight of the extracted humic acid is preferable because of two reasons. First, it can generate a larger size of graphene. The basic structure of humic acid is polyaromatic rings with a variety of substitutional groups such as predominantly carbonyl groups and hydroxyl groups,[10] as the idealized chemical structure of humic acid shown in the **Figure A1**. Therefore, a reasonable assumption is that the higher the molecular weight of humic acid, the more the polyaromatic rings and, thus, the large size of the resulting graphene after reduction. Second, it facilitates the homogenization of all the precursors in the slurry. Inhomogeneous mixing of the starting materials is an intrinsic disadvantage of the classic CTR route because it will cause an uneven surface modification of LFP particles, resulting in a quality inconsistency of LFP and, ultimately, the quality inconsistency of the batteries. To solve this inhomogeneity problem, in the ongoing project we innovatively disperse all of the precursors in water to form a homogeneous slurry using a polymeric dispersant. The polymeric dispersant can significantly reduce the polarity of water solutions and has excellent wetting and film-forming properties.[30] All of these properties make complete and uniform surface modification on LFP particles possible. The high molecular weight of humic acid will function as an even better polymeric dispersant than its low molecular weight counterpart.

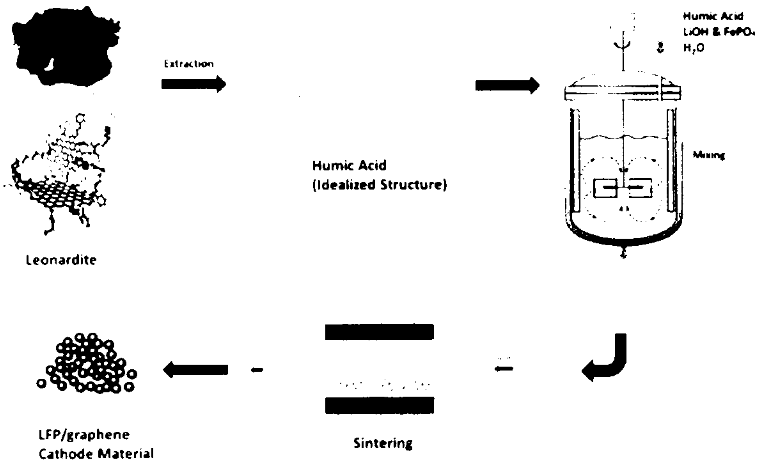

Figure A1. The proposed in situ synthetic procedure for preparation of graphene-modified lithium iron phosphate (LFP/G) cathode

Functions of Humic Acid: Besides the aforementioned function as a polymer dispersant, the more important function of humic acid is as the starting material for synthesis of graphene. The dual function of humic acid makes additional carbon sources like glucose unnecessary, which is common in most approaches for synthesis of LFP/G and thus simplifies the reaction system.

LiOH: As a carefully chosen lithium source, LiOH has a much higher solubility in water (12.8 g/100 mL at 20°C) than the conventional lithium source lithium carbonate (Li_2CO_3) (1.29 g/100mL at 25°C). Meanwhile LiOH solution is also a moderate base ($pK_b=-0.04$ $LiOH(aq) = Li^+ + OH^-$) in which humic acid can be completely dissolved. Therefore, all of the starting materials (Lithium source and humic acid) but $FePO_4$ will be dissolved in water, which ensures that a thorough interaction among all of the reactant molecules is achieved, and the homogeneity of the resulting slurry is comparable to solution-phase routes and much better than the conventional CTR route. Meanwhile, the only insoluble specie, $FePO_4$, is intentionally designed to keep its crystal structure and particle morphology to ensure the quality of LFP. As for the production cost, *the average market price of battery grade Li_2CO_3 and LiOH are \$10,910/t and \$11,280/t[31], respectively, which are quite close. However, choosing LiOH as lithium source is more energy-efficient as it avoids the energy-consuming step of grinding lithium carbonate to achieve the equivalent homogeneity of slurry.*

Slurry sintering: The slurry from the mixer is directly loaded to the furnace for drying at 70-105°C, followed by heating at elevated temperature (300-350°C) and the final sintering at 600°–800°C. This process avoids separate drying equipment, thus lowering manufacture cost. This radically innovative method benefits from using water as the mixing media that is otherwise impossible in the conventional CTR route. *The high-molecular weight of humic acid working as a dispersant in water can ensure the mobility of slurry for homogeneous mixing and, meanwhile, avoid the agglomeration of the precursor particles during the removal of water in the drying step. This concept has already been proven in our experiment at one-kilogram scale as 20-gram scale, as shown in **Figure 1**.* It is worthy of mention that we did not apply a conventional spray-drying technology to dry the slurry, as the too large secondary particle size of the obtained powder does not meet the requirements of cell manufacturers. In practice, therefore, a redispersing step is usually needed for the spraying-dried powder. Sintering is critical as it determines if the proposed two reactions: reduction of humic acid to graphene and synthesis of LFP will happen simultaneously. Powell and Beall[26] studied the reduction of humic acid to graphene in a series of experiments using chemical reductants as well as thermal reduction with H_2. They found thermal reduction is more effective. More importantly, *the temperature window suitable for thermal reduction is wide, from 500° to 900°C, which overlaps well with the one for synthesis of LFP via the CTR route, normally around 700°C. Our preliminary results strongly support this hypothesis.*

R&D Plan

Task 1. Optimization of Humic Acid Extraction Procedure: In this task, we will optimize our procedure to extract high-purity and high molecular weight humic acid from leonardite economically. Vermeer A. et al reported a humic acid extraction procedure to successfully obtain ash-free and trace metals lower than detection limit of ICP (Inductively Coupled Plasma) measurement[32]. Our method is based on this report but with significant modification, as their procedure is too time-consuming (over two weeks) and uses raw humic acid instead of coal or similar as the feedstock. As shown in **Figure A2**, the ground raw leonardite first is pretreated with dilute hydrochloric acid (HCl). This acid-leached leonardite is actually the product produced from an ongoing DOE-awarded project (DE-FE0027006) on extraction of REE from low-rank coals and thus could be beneficially integrated to reduce cost dramatically. The acid-leached leonardite (or raw leonardite) is then extracted either by an alkaline solution (Approach 1) or an organic solvent (Approach 2) to obtain the humate. The resulting humate is then treated with a mixed dilute HCl and hydrofluoric acid (HF) to yield the raw humic acid with ash content reduced significantly. The raw humic acid is then re-dissolved in a dilute alkali solution (i.e. ammonia or KOH) and treated with a lixiviating agent (i.e. Ethylenediaminetetraacetic acid EDTA) to further remove metal impurities. By adjusting the pH value of the solution a series of fractions of HA with different molecular weights are separated. In case the purity of the fractionated HA fails to meet the requirement, ion exchange resin (IER) will be used to further remove traces of metallic cations to achieve high purity of humic acid.

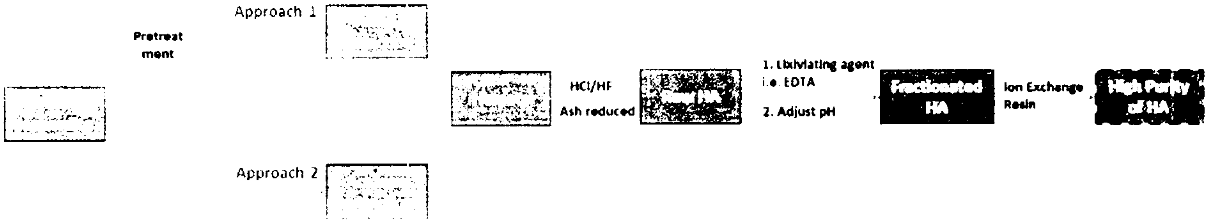

Figure A2. The procedure for extracting humic acid from

The HA extraction procedure will be systemically investigated with a focus on the following parameters:

- *Dissolution and extraction.* Two approaches will be tested and compared. In approach 1, an alkaline solution will be used to dissolve leonardite. Parameters including type of base (ammonia, potassium hydroxide [KOH]), concentration (0.1N – 0.5N), leonardite/solution ratio, and extraction time will be systematically studied with each parameter at three levels. In approach 2, a common organic solvent acetone mixed with dilute HCl will be used to dissolve and extract leonardite. Removing acetone via evaporation yields the humic acid, and the acetone can be recovered and reused. Parameters including acetone/acid ratio, liquid/solid ratio, temperature and extraction time will be optimized. The two critical indexes, purity and yield will be used to evaluate the extraction process. A software Minitab will be used to optimize the four parameters and investigate the interaction between them. A Taguchi orthogonal array design $L9(3^4)$ with 4 factors at 3 levels and a total of 9 runs will be implemented. A second run of design of experiments is contingent on the purity obtained by the first run to further optimize each factor.
- *Purification and fractionation.* While the pH decreases by slow addition of dilute hydrochloric acid, different fractions of humic acid will precipitate out in order of molecular weight. This method is similar to the widely used dissolution–precipitate approach for purification and classification of synthetic polymers. It is low-cost and easy to operate, as the only factor to control is the pH value. 3-5 values in the entire applicable pH range will be chosen to collect the precipitated humic acid. The effect of the humic acid of different molecular weight fraction on the formation of graphene and the final electrochemical performance of LFP/G will be compared.
- *Ion exchange resin.* In case the ion exchange resin is needed to further purify the fractioned humic acid, the humic acid will be re-dissolved in a diluted alkali solution (i.e. 0.1N KOH) and passed through two consecutive OH-form and H-form ion-exchange resin columns, following a slightly modified procedure reported before[33]. The resulting purified humic acid can be directly used for the synthesis of LFP in the form of a solution without drying.

According to our preliminary experiments, a maximum of 10% humic acid is needed for producing LFP/G. Our preliminary experiments show the yield of humic acid is about 50%-60% of raw leonardite. With a yield 50%, the estimated cost for producing 100 kg of humic acid for a ton of LFP/G is about \$500 with the breakdown specified in **Table A1**. An optimized synthetic procedure for LFP/G could use humic acid even less than 10%. In addition, the above calculation is based on the raw leonardite, but we can use the pretreated leonardite instead of raw leonardite as our starting material, which is actually a byproduct from an ongoing DOE-awarded project on extraction of REE from low-rank coals and thus could be beneficially integrated to reduce cost as well. Because of its low feeding ratio, humic acid only occupies about 5% of the total cost of LFP/G as detailed in **Table A2**, which is not significant compared with the other two precursors $FePO_4$ and Li source.

Table A1. Estimated Cost for Producing 100 kg of Humic Acid for a ton of LFP/G

Item		*Amount*	*Price Per Unit*	*Cost*	*Price source or Justification*
Chemicals	*Leonardite*	200 kg	\$220/ton	\$ 44.00	http://www.leonarditeproducts.com/
	Pottasium hydroxide (KOH)	1.96 kg	\$1089/ton	\$ 2.10	https://www.bulkapothecary.com/
	Hydrochloric acid (HCl)	51.9 L	\$165.50/ton	\$ 10.20	http://www.kemcore.com/
	Hydrofluoric acid (HF)	5.7 L	\$1255/ton	\$ 8.60	http://www.kemcore.com/
	lixiviating agent (EDTA)	2.6 kg	\$2697.35/300 lbs	\$ 26.00	http://www.essentialwholesale.com/
	Ion-exchange Resin (Reusable)	10 kg	\$109.95/52 lbs	\$ 23.40	https://www.uswatersystems.com/
	Acetone(Reusable)	10 gal	\$449/55 gal	\$ 81.64	https://www.dudadiesel.com/
Labor		12 h	\$25/h	\$ 300.00	Based on actual labor hours
Water(reusable)		1500 gal	\$3.32/1000 gal	\$ 4.98	http://www.grandforksgov.com/
Wastewater disposal		1500 gal	\$2.62/1000 gal	\$ 3.93	Grand Forks Wastewater Treatment Plant
			Total	\$ 504.85	

Note:
1. Shipping and tax are not included in the cost
2. Costs for reusable items are equivalent to single-use cost
3. Electricity and other facility use are not included here, but included in LFP production cost as a whole

Table A2. Cost/Profile Structure of Producing 1 ton of LFP/G by the Proposed Technology Compared with the Prices of Commercial LFP in the Current Market

Cost (per ton)	*Our LFP/G (in situ preparation)*	*LFP/G (mix LFP with rGO)*	*Commercial LFP Made-In-China (low grade)*	*Commercial LFP Made-In-USA (high grade)*
$FePO_4$	\$3,610[a]			
Li Source	\$2,651[b]			
Humic Acid	\$505			
Other Additives		\$25,000 [c]		
Labor	\$3,383[d]			
Energy				
Overhead	\$1,015[e]			
Total Cost	\$11,164	\$36,340		
Market Price	\$20,000 (projected)	N/A	\$14,000	\$30,000
Profit	**\$8,839**		\$3390	\$19,390

[a] Given \$3,800/ton cost and requiring 0.956 tons of $FePO_4$ as a starting material, we have \$3,800 at 0.956 = \$3,610.

[b] Given \$11,280/ton cost and requiring 0.235 tons of Li source, we have \$11,280 at 0.235 = \$2,651.

[c] Given \$5,000,000/ton cost and adding 0.50% of graphene, we have \$50,000,000*0.01 = \$25,000. The price of graphene \$5,000,000/ton is the minimum quoted by the major suppliers based on our market investigation.[6-9] All other costs are estimated based on our process, which could be an underestimate because of the simplicity of our proposed technology.

[d] Operation cost, such as labor and energy accounts for 20-50% of raw materials cost normally in powder industry. Given the highest rate of 50%, we have \$(3,610+2,651+505) *0.5=\$3,383 expensed on direct labor cost and energy consumption.

[e] Overhead accounts for 10-30% of operation expense normally. Given maximum 30% here, we have \$3,383*0.3=\$1,015.

APPENDIX B

MARKET ANALYSIS AND COMMERCIALIZATION PLAN

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I. Elevator Pitch

1.1. The Customer

The primary market of the proposed LFP/G cathode materials is to meet Clean Republic's own demand of low-cost and high-performance LFP for its LIB packs that are used in E-bikes and large power tools. Clean Republic has already supplied thousands of custom battery packs per year in the U.S. market. Based on first-hand sales figures (**Figure A3**), the average monthly unit of Dakota Lithium pack sold by Clean Republic is 110 in 2015, 271 in 2016 and 463 per month in 2017 respectively. Sales have also seen a large increase as of October 2017 to an excess of 1000 per month. Combining battery packs used for its own E-bike products and for industrial users, Clean Republic has already generated a demand for more than 20 tons of LFP powder in 2017. Given its continued annual growth rate of 27%, which the company has had since it started in 2009, Clean Republic will consume at least 83 tons/year of high-quality LFP/G powder by 2023. Internal demand alone will consume 20% of total LFP/G production the company produces by 2023, which will contribute $1.6 million in revenue.

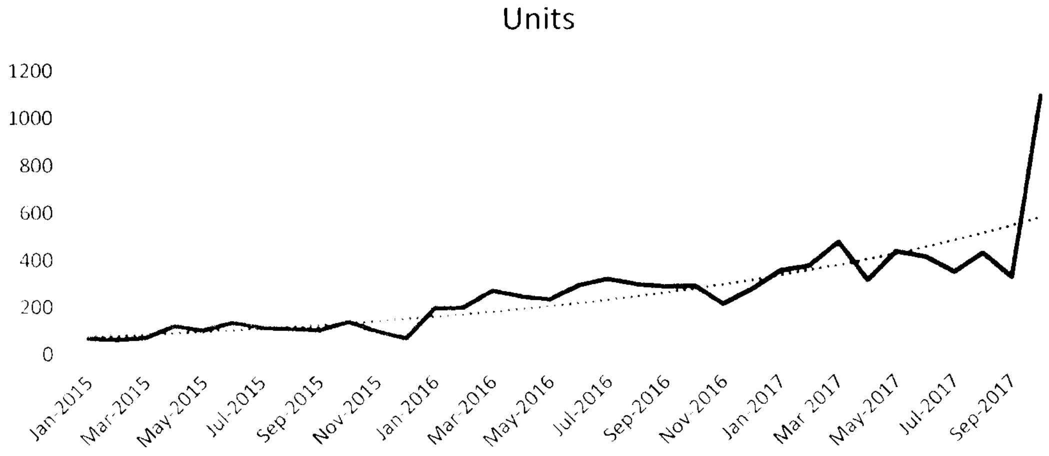

Figure A3. Dakota Lithium battery unit sales by month since 2015. Note the recent large increase in sales volume

There are other large demands for LFP materials in the United States besides Clean Republic's own need: 1) electric vehicles (EVs) and hybrid electric vehicles (HEVs), accounting for a total market share of about 62%, the EV and HEV segment dominated the market for LFP during 2015. 2) Energy storage. LFP batteries come as the most preferred choice not only for alternative energy storage systems, but for large-grid systems in load-leveling and central backup applications. According to the statistics on the website of Department of Energy, the announced 'load-leveling' projects alone raised the demand of LFP by 192 tons in 2016.[34] Benefited by a unique synthetic procedure and competitive production cost, this project will offer Clean Republic a great opportunity to sell 80% of the LFP/G materials it makes to other LIB producers in the United States. The amount will be 320 tons by 2022, which is about $6.4 million in revenue.

1.1 The Value Proposition

There are no commercially available LFP/G cathodes on the global market. The mainstream LFP materials are still showing nothing near the reported performance of LFP/G. For instance, the specific capacity of the top-quality LFP currently is 120–125 mAh/g, while that of LFP/G is close to the theoretical value of 170 mAh/g. The proposed project will allow offering the market a unique LFP material with higher energy density, higher rate capability, and much better low-temperature performance, compared to global competitors. The estimated cost of Clean Republic's LFP/G is about $11,000 per ton, in contrast with the estimated price of about $36,000 by directly adding commercial graphene to LFP. Our production cost would even be lower than that of low-grade LFP products ($14,000/t made in China) and of high-grade LFP made in the United States ($30,000/t). About $9,000 profit per ton will be expected at the projected market price of $20,000/t from this project. More importantly, Clean Republic already has 8 years of experience networking with the entire supply chain of LIBs, from LFP suppliers and cell manufacturers to end users of LIBs, which is nearly impossible for other start-ups to build in a short time.

1.2 The Innovation

Existing research on the preparation of LFP/G normally requires the synthesis of graphene in advance, which severely inhibits practical applications of those processes. Reduced graphene oxide (rGO), a necessary material for those processes, can cost up to $50,000/kg, prohibitive for battery applications. This innovation aims to develop a unique, low-cost synthetic procedure to prepare LFP/G cathode materials. It proposes a two-step procedure: 1) humic acid is extracted and purified from leonardite or low-rank coals from North Dakota, and 2) the extracted humic acid is mixed with a Li source and $FePO_4$ to prepare LFP/G in situ via a modified carbothermal reduction route (CRT). Our process cleverly takes advantage of an abundant and low-cost raw material, combined with a simple manufacturing process (competitors take multiple steps), to produce an LFP battery with 15% more capacity than those on the market today, even at a lower cost.

North Dakota has abundant, cheap, and the highest-quality leonardite worldwide. UND IES is currently conducting a DOE-awarded project for the extraction of REEs from lignite (including leonardite), in which the carbonaceous residue after extraction can be used as a source for the humic acid at a dramatically reduced cost compared to other conventional sources.

II. The Commercial Opportunity

2.1 Broader Societal Need

In the past decades, increasing concerns on environmental impacts and supply shortage of fossil fuels stimulated the urgent demands of clean, sustainable energy technology. Among all of the clean energy technologies, lithium-ion rechargeable batteries (LIBs) are arguably the most promising power system and energy storage devices, which have wide applications from driving EVs to powering consumer electronics such as cell phones, cameras, and laptops. Since the successful commercialization of $LiCoO_2$ as a cathode material for LIBs by Sony Inc. in 1991,[35] numerous efforts have been made to continuously improve the performance of LIBs to meet various applications.[36] In fact, the battery industry is making incremental capacity gains of 8%–10% a year.[37] However, LIB technology is still far away from full maturity, and many challenges remain in environmentally benign manufacturing of low-cost, high-performance, high-safety LIBs, especially for large-scale applications such as transportation technology like EVs and energy storage technology.

2.2 Market and Addressable Market for the Innovation

Promoted by the rapidly increasing demand in power batteries for large applications such as EVs, PHEVs (plug-in HEVs), and energy storage devices, the global demand and production capacity of cathode materials for LIBs have skyrocketed in recent years, and this growth trend will remain at least until 2026 with a compound annual growth rate of 11.6%~12.5% as forecasted by multiple leading advisory institutes worldwide.[38-40] According to "Global Outlook of the Lithium Iron Phosphate Battery Market," a market study report published by TechNavio, the global lithium iron phosphate battery market will grow impressively at more than 20% during 2015–2020.[41] In 2014, about 12,500 tons of LFP powder was sold globally, and China accounts for almost 75% of the total because of the Chinese government's great support for new electric vehicles. Along with the dramatically increasing demand impelled by the EV industry in the Asia Pacific area and growing applications in the energy storage industry in North America and Europe, annual global demand of LFP is expected to be more than 30,000 tons in 2016–2020.[42] Meanwhile, global production capacity is far from meeting the growing market demand. Traditional LFP manufacturers mainly include the U.S.-based A123, Valence, and Canada-based Phostech, which hold slightly dated but mature mass production technology and supply first-grade, quality products. However, the total production capacity of Valence, Phostech, and A123 is only about 1,000 tons annually. In short, a huge gap exists between the demand and supply of LFP, especially the top-quality materials, in North America and worldwide.

(1) Meet Clean Republic LLC's Own Need

The primary goal of the proposed project is to develop LFP that meets Clean Republic's own need for its two main products—E-bikes and LIB packs. Both are seeing quick growth in the next few years. Clean Republic's own retail channels already created a demand for more than 20 tons of LFP powder in 2017, and that demand has been growing by 20%–30% per year since the company started in 2009. *E-bikes*, the main products of Clean Republic, are seeing extreme growth in the United States, and all of their supply chains require the high-quality LFP powder materials described in this proposal. *Large power tools* such as ice augers and electric mowers are other target markets. For instance, although exact sales figures are confidential, Clean Republic already supplies tens of thousands of custom battery packs to this market.

(2) Other Potential Markets

There are other huge potential markets besides Clean Republic's own need: *1) EV and HEVs.* A123 Systems, BYD, and Electric Vehicle Power System Technology have been some prominent vendors in the market. Accounting for a total market share of about 62%, the EVs and HEVs segment dominated the LFP market during 2015.[34] *2) Energy Storage.* The application of LFP batteries in energy storage photovoltaic and communication batteries is on the rise, reflecting a huge space for development. Because of their long lifetime, cycle time, and safety, LFP batteries come as the most preferred choice not only of alternative energy storage systems, but also of large grid systems in load-leveling and central backup as well. In the Unites States, 66 electric storage projects with 24,072 kWh capacity in total were announced in 2015 to May 2016, which are using LFP batteries.[34] Those announced load-leveling projects alone raised the demand by 192 tons of LFP cathode material currently in the United States. Those projects indicate that energy storage systems are generating huge demand for LFP battery products domestically and globally.

The market opportunity for Clean Republic's own need is validated based on the first-hand sales figure and analysis of recent demand trends. The potential and broad market opportunity is predicted by multiple leading advisory institutes worldwide as cited above.

2.3 Competition

Currently, there are no commercially available LFP/G cathode materials. The main competition is from the mainstream LFP cathode products on the market. The main LFP suppliers in the world include A123, Valence, Sony, Phostech, and many others in Asia like BYD. Despite the increasingly improved overall performance of the LFP cathode, it is still nothing near the reported performance of LFP/G. For example, the specific capacity of the top-quality LFP on the current market is 120–125 mAh/g, while LFP/G demonstrates a specific capacity close to the theoretical value 170 mAh/g in most reports.

As detailed in **Table A2**, the estimated cost of our LFP/G is about $11,000 per ton, in contrast to the estimated price of $36,000 by directly adding commercial graphene to LFP. This price is lower than that of commercial LFP products, $14,000 made in China and $30,000 made in the United States. A $9,000 per ton profit is estimated at the projected market price of $20,000 per ton.

2.4 Key Risks to Bring Innovation to Market: Commercialization Approach

Several challenges and risks associated with commercialization of the proposed technology have been identified along with our preliminary mitigation strategies as described below.

(1) Increasing the Laboratory Process to Economy Scale

Successful commercialization implies profitability in the long term that requires mass production of LFP/G. Therefore, the primary challenge is to scale up the technology that succeeded at laboratory scale into pilot production scale. Long-term profitability of this proposed technology is possible only by taking advantage of economies of scale and automation to reduce U.S. labor costs.

Clean Republic has already overcome many challenges involved in bringing cutting-edge lithium battery technology to retail customers in a profitable model. As the company has expanded its production assembly lines at the LFP battery pack level, it has already completed many cycles of research and implementation of bulk purchasing of materials and supplies and introducing semiautomatic equipment into previously manual processes.

(2) Gap Between Pilot-Scale Production and Mass Production

For most of the cathode material producers, once the technology of producing LFP powder succeeds at pilot scale, the next major challenge is to persuade the customers (cell manufacturers) to accept their new LFP materials before tons of powder can be sold. The process of trying and testing a new supplier's LFP powder in a customer's own cell manufacturing facility is such a long and painful process that not many LFP vendors can smoothly cross over this challenge and make the political, marketing, and cash flow leaps up to mass production and supply contracts with their first big customers even if their materials are of high quality. Without deep collaboration with real consumers, the gap between pilot-scale production and mass production is huge. Many ventures like this died from not crossing the gap smoothly.

Clean Republic already has 8 years of experience in the entire supply chain of LIBs from LFP cathode material suppliers and cell manufacturers to its end users of custom LIBs and E-bikes. For other start-ups, this industry network building can be nearly impossible, but for Clean Republic, working closely with its LFP suppliers and cell manufacturers on factory floors in China is already a daily routine to ensure the quality of its own battery packs and E-bikes. Clean Republic is very proud of this close cooperation with its business partners and quite confident in persuading them to use its own LFP once it is of equal or even superior quality than that available from current suppliers.

(3) Uncertainty on Market Price

As described previously, the global demand for LFP is rapidly increasing. Therefore, not only are new ventures likely going to enter this LFP market, but the existing big suppliers are extending their production capability. That will increase the risk of a falling market price of LFP material if the supply is much greater than the demand. Alternately, one of the main raw material prices could increase suddenly.

To mitigate the risks in bringing our proposed technology to market, we have developed a conservative commercialization approach: Clean Republic will control the schedule of capacity expansion to make sure that at least 20% of the material product is being used for Clean Republic's own cell production and will end up being packaged and sold to customers through its own retail and wholesale channels. The vertically integrated business model already operated by Clean Republic can remain protected from even large shifts in market prices at various supply chain levels because of the stability and profit margin already commanded by its retail sales to end users.

(4) Uncertainty in Price of Lithium Resource

The growing demand for lithium ion batteries worldwide causes the concern on uncertainty in price of lithium resource. However, assume the market price of LiOH doubled than $11,280/t that this proposal quoted in 2017, that means the total cost would raise to $13,815/t, Clean Republic may still have $6,100 profit per ton. A 40% margin presents the project is still very profitable when price of lithium resource even doubled than current market price.

2.5 Resources Needed for Commercialization

The facility and equipment for the pilot production of proposed LFP/G are either available or ordered during the existing LFP project. See **Appendix D** for details. Clean Republic already has access to the suppliers and industry advisors necessary to acquire the particular equipment and design more efficient processes to scale the results confirmed by the work outlined in this proposal profitably. In addition, the UND IES' chemical and process engineering experiences will facilitate upscaling the proposed technology from laboratory scale to pilot plant.

2.6 Key Technical Challenges and Risks in Bringing the Innovation to Market

(1) High-Purity, Low-Ash Content, and Iron-Free Humic Acid

Metallic impurities in particular iron is well-known to have an adverse effect on the battery performance of LFP cathode materials.[43] A prerequisite for this proposed technology to succeed is to obtain low ash content and iron-free humic acid. Fortunately, there are successful examples in the literature in which high purity and metal-free humic acid has been extracted (See reference 27: Vermeer, A et al, *Langmuir* **1998**, 14 (10), 2810-2819). The existence of such examples shows that our objective is achievable. Our objective is set for an ash content <0.5% and iron <0.1%, and we are able to reduce iron content to 0.2% (see **Table 1**) without optimizing the procedure. Therefore, it is safe to claim that with further purification via optimized procedure we can produce high purity humic acid. Our plan to overcome this technical challenge is detailed in the methodology section of the proposal.

(2) In Situ Formation of Graphene on LFP Particles

Although humic acid has been successfully reduced to graphene independently in literature, in situ formation of graphene in the synthesis of LFP could be a technical challenge. The reaction conditions must be well controlled to ensure both reactions (reduction of humic acid to graphene and synthesis of LFP) proceed simultaneously. The rationale of our proposed technology is discussed in the methodology section of the proposal. Exploring the strategies to mitigate this risk as the process is scaled up will be one main tasks (Task 2) of the proposed project.

III. Business Plan

The ultimate objective of this proposed project is to set up a pilot plant for making LFP/G material at Clean Republic's facility at 5515 University Avenue, Grand Forks, North Dakota. With support from the North Dakota Industrial Commission, this project will result in production and sale of 320 tons of LFP/G material by 2023. Based on the average growth rate of business since the company started in 2009, at least 20% of the powder product will be used by Clean Republic itself, which will not only strengthen its competition in the market by utilizing the high-performance, low-cost materials, but generate more new jobs locally. **Table A3** briefly describes our vision for the company and the expected impact from the LFP/G production over the next 5 years. The LFP/G powder production alone will generate revenue of $2.4 million/year by 2021 and may reach $8 million annual revenue by 2023. The current ratio of revenue and full-time employee numbers in Clean Republic is about $200K:1. Based on the ratio, and by conservative estimates, this new LFP/G product alone will generate 12 full time jobs by 2021 and over 40 jobs by 2023 in Grand Forks, North Dakota, which includes production operators, sales staff, engineers, and administrative staff.

In addition, the market value addition ratio between cathode material and the battery packs made out of them in the LIB industry is roughly 1:6. Given 83 tons ($1.66 million) of high-performance, low-cost LFP/G cathode materials will be used in its own LIB pack products by 2023, ***Clean Republic will have the chance to enlarge its market share in the United States and generate up to $8 million USD revenue from its LIB pack products. This would offer another 20 jobs in Grand Forks, North Dakota (not listed in the table below).***

Table A3. Vision for Clean Republic and Expected Impact for 5 years

Year	***Operation***	***LFP Powder Production***	***Projected Demand from CR***	***Revenue from LFP Powder***	***New Jobs Generated by LFP***
2017	Funded by ND research (Venture Grant Phase I/II grant), Clean Republic and UND IES are collaborating on an existing project to develop their own LFP/C cathode material. By the end of 2017, the Phase II goal is to achieve pilot-scale production.	LFP/C: lab scale	25 tons	0	
2018	Operating of the pilot plant with a capacity of 10 tons/y of LFP/C This Proposed Project at lab scale: Preparation of Graphene-Modified $LiFePO_4$ Cathode for LIB	LFP/C: 10 tons/y LFP/G: lab scale	32 tons	$200,000	1
2019	Enlarging LFP/C production facility from pilot plant to mass production plant Preparation of Graphene-Modified $LiFePO_4$ Cathode for LIB in Pilot Plant Meeting Clean Republic's own demand of LFP powder and, at the same time, selling small amounts of powder to others	LFP/C: 40 tons/y LFP/G: 10 tons/y	40 tons	$1 million	4
2020	Transition from LFP/C to LFP/G Enlarge the preparation of LFP/G from lab pilot scale to mass production plant Meeting Clean Republic's own demand of LFP powder and, at the same time, selling almost half of powder to others	LFP/G: 80 tons/y	45 tons	$1.6 million	8
2021	Supplying commercial LFP/G to Clean Republic and other customers in North America	LFP/G: 120 tons/y	52 tons	$2.4 million	12
2022	Supplying commercial LFP/G to Clean Republic and other customers in North America	LFP/G: 200 tons/y	66 tons	$4 million	20
2023	Supplying commercial LFP/G to Clean Republic and other customers in North America	LFP/G: 400 tons/y	83 tons	$8 million	40

APPENDIX C

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APPENDIX D

FACILITIES, EQUIPMENT AND OTHER RESOURCES

EQUIPMENT:

Field Emission Scanning Electron Microscopes (FE-SEM)
This FEI Quanta 650 FEG SEM is located at Material Characterization lab (MCL) of UND. The Field emission SEM capable of obtaining high-resolution data from almost any sample materials. The instrument is operable in both high and low vacuum modes. The x-ray microanalysis system consists of an energy dispersive Bruker QUANTAX 200 x-ray detector. The system is equipped with backscattered and secondary electron imaging. The backscattered imaging allows for discerning materials based on atomic number. The presence of higher atomic number materials increases the brightness and allows for easy identification and subsequent analysis. The instrument is able to achieve 1-3 nm resolution. The imaging software package allows for performing analysis of mineral association with coal and other minerals.

Hitachi SEM 3400N with an Energy Dispersive System (SEM/EDS)
The Hitachi 3400N SEM is also located at Material Characterization lab (MCL) of UND and equipped with backscattered and secondary electron detectors for imaging and is automated with energy dispersive x-ray detectors for chemical composition analysis. The system can perform computer controlled scanning electron microscopy (CCSEM) of particles to determine the size, composition (major, minor, trace elements), and mineral typing. The system is also equipped to provides microstructure analysis, and elemental composition information on association of minerals with coal particles.

X-ray Fluorescence Spectrometers (XRF)
Located at Material Characterization lab (MCL) of UND, this Rigaku Supermini 200 XRF is a wavelength dispersive bench-top XRF able to provide low ppm detection limits for major, minor, and trace elements. The instrument is equipped with a 12 sample auto-sampler and can analyze either solids or liquids. The software allows rapid analysis of known and unknown samples. The system provides the ability to perform quantitative analysis and qualitative survey scans to identify the presence of elements.

X-ray Diffraction (XRD)
Located at Material Characterization lab (MCL) of UND, the Rigaku SmartLab is a fully automated XRD that utilizes cross-beam optics (CBO) enabling fast and easy changing of the incident X-rays by substituting selection slits. The instrument can operate in either Bragg-Brentano or parallel beam focusing methods. The flexible design allows for analysis of samples ranging from loose powder to large pieces of sample. The instrument is equipped with both a scintillation acquisition. A Cu $K\alpha_1$ system with a monochromator is also available for high intensity measurements. The system is equipped with a CCD camera for imaging of specific areas on a sample and has a variety of stages allowing analysis of a wide array of sample types and applications. Once the x-ray diffraction pattern is obtained it is analyzed to determine the crystalline phases present. The system can also be used to perform quantitative XRD analysis.

Raman Spectrometer
This Thermo Scientifics' Nicolet NXR 9650 FT-Raman spectrometer is located at chemical engineering department of UND college of engineering and mines. It is capable of producing high-quality Raman measurements using high-speed scanning, sample mapping, screening applications, and kinetic analysis. This Raman Spectrometer can measure organic and inorganic samples using a spectral range down to 100 cm^{-1} which is advantageous for

inorganic samples where bands are typically observed at low frequencies. The confirmation of graphene will be performed by the Raman Spectrometer.

High Resolution of Transmission Electron Microscope (HR-TEM)
This JEOL JEM-2100 multipurpose analytical high-resolution transmission electron microscope system is located in Electron Microscopy Center of North Dakota State University, which is only 70 miles away from UND. Being a member under North Dakota university system (NDUS) with NDSU, UND users have an access to this equipment with a favorable price. HRTEM is a powerful tool to study properties of materials on the atomic scale, such as semiconductors, metals, nanoparticles and sp^2-bonded carbon (e.g., graphene, C nanotubes). At present, the highest point resolution realized in phase contrast TEM is around 0.050 nm.

Zetasizer nano ZS90 (Malvern Instruments Ltd, UK)
This equipment is available at Chemistry Department of UND. It is a Dynamic Light Scattering (DSL) system for the measurement of particle size and molecular size at a 90 degree scattering angle using Dynamic Light Scattering, also with the ability to measure zeta potential and electrophoretic mobility using Laser Doppler Microelectrophoresis, and molecular weight using Static Light Scattering. Using 90 degree scattering optics, it allows for size measurement from 0.3nm (diameter) to 5 microns as well as molecular weight measurement down to 9,800Da.

Carbon Analyzer TOC SSM 5000A analyzer (Shimadzu, Japan)
This equipment is located at Environmental Analytical and Research Laboratory (EARL) of UND. Total carbon (TC), inorganic carbon (IC), total organic carbon (TOC) analyses via Shimadzu TOC Analyzer and SSM-5000A Solid Sample Module in both aqueous and solid samples as well as total nitrogen analyses via TNM-1 Unit in aqueous samples. The carbon content of LFP/G will be tested by this carbon analyzer.

Sample Preparation
To take advantage of the above equipment, UND has a fully-equipped sample preparation lab, with all of the necessary capabilities for the sample preparation requirements contained in the proposed this project. Available equipment includes a Mixer/Mill 8000 M (SPEX, USA), a LaboPol-21 polisher (Sturers Inc.), a X-press sample presser (SPEX, USA), a K-1 flux (SPEX, USA), a shatter box (SPEX, USA) and a Micronizing mill (McCrone).

Neware BTS-3000n Battery Analyzer
Neware's BTS-300n is capable of cycling 8 channels of coin cell or cylinder cell in its' clamps and adjustable cell holders. Capable of charging and discharging from 3mA to 6A up to 5V. Software package allows for custom test settings and automatically builds charge-discharge curves.

MBraun LABstar MB10 Glove Box
Glove box workstation capable of automatically maintaining a clean atmosphere of inert gas with less than 1ppm O_2 and H_2O. The workstation was purchased in 2010, then refurbished and certified by an MBraun technician in 2017. Scrubs O_2 and H_2O with a single column, regenerable catalytic bed of copper catalyst and mole sieve. Capable of maintaining atmospheres of nitrogen, argon and helium. An atmosphere of ultra-pure argon is used for lithium-ion battery cell assembly, to prevent the reaction of N_2 with the pure lithium counter electrodes.

Kejia KJ-A1200-27L Atmosphere Furnace
Kejia's atmosphere furnace can reach 1200°C with 1100°C working temperature with a 35-step programmable temperature controller. Chamber size is 27 liters. Holds a vacuum down to -85 KPa_{gauge} and pressure up to 80 KPa_{gauge}. The chamber can be filled with any desired atmosphere after applying vacuum and filling cycles to achieve

a pure working atmosphere. Uses one-way inlet and outlet ports to allow for excess pressure relief without contaminating the atmosphere.

JSDL-Q-3 Jet Milling system

This system consists of a jet mill, an air compressor, a cold dryer, a filter, and a gas storage tank. This jet mill can pulverize the raw materials with feed particle size less than 1 mm to ultra-fine powder with adjustable particle size (D_{50}=0.2-2 um and D_{90}=5 um). The working pressure of the jet is 0.8-1 MPa with air consumption 3 m^3/min, and the production throughput is 3-5 Kg/h. The working temperature is slightly higher than ambient temperature by about 5-10 degrees. All surfaces with contacting feeding/outputting materials is iron-free and free of air dust pollution. The equipment is a special superfine crushing equipment, the product is pure and pollution-free.

FACILITIES

Battery Test Center

This center is located at Clean Republic LLC, 5515 university Ave, Grand Forks. It has the capability to test a verity of batteries from small size CR2032 coin-type cells, to full size 26650 cells, and up to 48V battery packs. It houses a Neware BTS-3000n Battery Analyzer for coin-type cell and a HYNN-BP600A battery testing system with the capabilities including battery charge/discharge characteristic test, battery capacity test, battery cycle life test, battery filled/discharge efficacy test, and battery standard dynamic test.

Materials Characterization Lab (UND)

The MCL was established to support UND research and educational activities, to support industry research and sample analysis needs, and to serve as a regional satellite lab. The laboratory is supported by experienced technicians and analytical chemists and has a vast array of analytical equipment and capabilities, including SEM-EDAX, XRF, XRD, and TGA. The MCL will be used to examine the OC particle morphology and composition and to determine particle-size distribution.

Institute for Energy Studies Lab

This lab is located on the second floor of the newly built Collaborative Energy Complex of UND College of Engineering and Mines. It houses a standard chemical fume hood, a walk-in fume hood, a MBraun LABstar MB10 Glove Box, a Kejia KJ-A1200-27L Atmosphere Furnace, and ancillary equipment to support research projects.

Environmental Analytical Research Laboratory (EARL)

UND College of Engineering and Mines. EARL is located on the 3rd floor of Leonard Hall at the UND campus and houses some advanced analytical instruments including an DIONEX DX-120 Ion Chromatograph (IC), an SOLAAR M6 Atomic Absorption Spectrometer (AAS), a SHIMADZU Total Organic Carbon Analyzer (TOC), a LECO SC-432DR Total Sulfur Analyzer and ancillary equipment to support teaching, scientific research and engineering design projects.

OTHER RESOURCES:

UND has fully-equipped laboratories and larger bench and pilot-scale demonstration areas. Wet chemistry laboratories will be utilized in the proposed work to conduct some of the characterization work in Task 4. UND also has a fully-equipped mechanical and electrical fabrication shop, with a full list of capabilities that include welding and machining as well as mechanical and electrical installation services. The shop is staffed by experienced personnel with the training and availability to perform the necessary work as requested. UND's office areas are equipped with all of the necessary software and computing requirements to complete the scope of work.

APPENDIX E

RESUMES OF KEY PERSONNEL

Xiaodong Hou, Ph. D

Institute for Energy Studies,
College of Engineering & Mines, University of North Dakota
Upson II 16, 243 Centennial Drive, Grand Forks, ND58202-8153
701-777-6791 (O) 701-739-9058 (C) xiaodong.hou@und.edu

EDUCATION

2009 **Ph.D.** **Polymer Chemistry and Physics**, Shanghai Jiao Tong University/Shanghai Institute of Organic Chemistry (CAS), Shanghai, China. Dissertation: Studies on the Chemistry and Physics of a New PS-*b*-PLA block copolymer/Nanocrystal Hybrid Material

2005 **M.S.** **Chemical Engineering**, Shaanxi University of Science and Technology Shaanxi, China. Thesis: Preparation and Properties of Composite Material of Cellulose and Collagen Recovered from Chrome-containing Leather Waste

2002 **B.S.** **Chemical Engineering**, Shaanxi University of Science and Technology Shaanxi, China

WORKING EXPERIENCE

2014.10-present **Senior Researcher /Analytical Chemist** Institute for Energy Studies (IES), College of Engineering and Mines, University of North Dakota, Grand Forks, ND.
Research direction: development of advanced energetic materials (eg. cathode materials for Li-ion battery) using diverse chemistry approaches: organic chemistry, polymer chemistry and inorganic chemistry

2013-2014.09 **Lab Director** and **Analytical Chemist** for EARL, College of Engineering and Mines, University of North Dakota, Grand Forks, ND

2010-2013 **Postdoctoral Research Associate**, Chemistry Department, University of North Dakota, Grand Forks, ND.

- Synthesis and Characterization of covalently bonded hierarchical organic-polymeric nanomaterials as advanced energetic materials

2009 **Intern/Application Chemist**, Shimadzu Corporation, Shanghai, China

- Perform material characterization service using abroad instruments such as DSC, FT-IR, UV, AA and ICP

RESEARCH INTERESTS

- Development of Cathode materials for high performance Li-ion battery
- Synthesis and Characterization of Inorganic/Organic hybrid materials, and functional polymer materials for advanced energy application
- Preparation and application of hierarchical organic/polymeric nanomaterials such as 1D and 2D nano-architectures by supramolecular self-assembly and polymerization.
- Synthesis and biomedical application of polymers-modified semiconductor nanoparticles

RESEARCH PROJECTS ACCOMPLISHED

- "A Low Cost and Reproducible Synthetic Procedure for Mass Production of Lithium Iron Phosphate Cathode Materials for Lithium Ion Batteries", Agency: NDIC, Venture Grant Phase I/II, PI, Amount: $148, 105, Total Award Period Covered: 1/1/2017-12/31/2017
- "Rare Earth Element Recovery from North Dakota Coal Related Feedstocks", Agency: DOE, key

participant, PI: Seven Benson, Amount: $937,000, Total Award Period Covered: 03/01/2016 – 08/31/2017

- "Williston Basin Advanced Core and Well Log Consortium" Agency: Hess Corp., Hunt Oil, Murex Petroleum, Whiting Petroleum, and NDGS. Amount: $500,000, Key participant, PI: Peng Pei, Total Award Period Covered: 10/01/2014 - 9/30/2019
- "Production and Characterization of CO_2 Capture-Based Polyurea Bulk Polymers and Coatings" Agency: National Science Foundation (NSF), Key participant, PI: Dr. Qianli Chu, Amount: $126,052, Total Award Period Covered: 10/01/2013 - 9/30/2016
- "Novel Polymeric Architectures from Monomers with Multiple Reactive Centers" Agency: American Chemical Society (ACS) and Petroleum Research Foundation (PRF), Key participant, PI: Dr. Qianli Chu, Amount: $100,000, Total Award Period Covered: 01/01/2013 - 8/31/2015
- "Construction of Nano-structures Using Symmetric Molecules as Building Blocks" Agency: ND EPSCoR SMS Program, Key participant, PI: Dr. Qianli Chu, Amount: $50,000, Total Award Period Covered: 10/01/2013 - 8/15/2014
- "Construction of Nano-structures Using Symmetric Molecules as Building Blocks" Agency: ND EPSCoR SMS Program, Key participant, PI: Dr. Qianli Chu, Amount: $27,566 Total Award Period Covered: 01/01/2013 - 8/15/2013
- "Construction of Novel Organic Nanotubes with Potential Green Energy Applications" Agency: ND EPSCoR AURA Program, co-advisor, PI: Dr. Qianli Chu, Amount: $7,975, Total Award Period Covered: 05/16/2013 - 11/29/2013
- "Design, Synthesis and Self-Assembling Behaviors of Nanocrystal-Bearing New Functional Biocompatible Polymer" Agency: National Natural Science Foundation of China (No. 20574087/B040101): Key participant, PI: Dr. Amin Cao, Amount: RMB260,000, Total Award Period Covered: 2005.12~2009.6
- "Research and Development of Engineering Sizing Agent for Carbon Fiber" Agency: Chinese Department of Defense: Key participant, PI: Dr. Amin Cao, Amount: RMB1,000,000, Total Award Period Covered: 2008.5~2009.6
- "Preparation of Adsorbent for Removing Low Density Lipoprotein from Plasma" (No. 0452nm050). Key participant, PI: Dr. Amin Cao, Amount: RMB700,000, Agency: Shanghai Nanotechnology Promotion Center 2005.10~2007.3
- "Preparation and Application of Composite Material by Natural Macromolecules Collagen and Cellulose" (No: 02JS41). Key participant, PI: Drs. Zhijie Wang & Chuanbo Zhang, Amount: RMB300,000, Agency: Department of Education of Shaan Xi Province: 2001.12~2005.4

EXPERIENCE AS AN INVITED INDEPENDENT REVIEWER

- *Macromolecules (1 time)*
- *Journal of Applied Polymer Science (20⁺ times)*
- *Macromolecular reaction and engineering (4 times)*
- *CrystEngComm. (4 times)*
- *Macromolecular materials and engineering (3 times)*
- *Polymer for Advanced Technologies (1 time)*
- *Journal of Polymer Research (1 time)*
- *Carbohydrate Polymer (2 time)*

PUBLICATIONS

Peer reviewed journal articles:

1. **Xiaodong Hou**, Jonathan Butz, Jiao Chen, Zijun D. Wang, Julia X. Zhao, Tiffany Shiu, and Qianli Rick Chu*. Low Molecular Weight Organogelators Derived from Three-Fold Symmetric Tricarbamates. Tetrahedron Letters, **2017**, *58*, 101-105.
2. Peng Pei*, Kegang Lingb, **Xiaodong Hou**, Stephan Nordengc, Scott Johnson. Brittleness investigation of producing units in Three Forks and Bakken formations, Williston basin. *Journal of Natural Gas Science and Engineering,* **2016**, *32, 512-520.*
3. Wang, Z.; Randazzo, K.; **Hou, X.**; Simpson, J.; Struppe, J.; Ugrinov, A.; Kastern, B.; Wysocki, E.; Chu, Q. R. *Macromolecules,* **2015**, 48, 2894-2900.
4. **Xiaodong Hou*,** Tao Zhang, and Amin Cao. Preparation of New Amphiphlic Microporous Non-woven Polymeric Adsorbents for Selective Removal of Low-Density Lipoprotein from Plasma. *Journal of Biomedical Material Research: Part B,* **2015**, *103*, 52-61.
5. Chen, J., Wu, X., **Hou, X.**, Su, X., Chu, Q., Fahruddin, N., & Zhao, J. X. Shape-tunable hollow silica nanomaterials based on a soft-templating method and their application as a drug carrier. *ACS Appl. Mater. Interfaces*, **2014**, *6*, 21921-21930.
6. Zijun Wang, Joseph Lee, Casey Oian, **Xiaodong Hou**, Zhihan Wang, Angel Ugrinov, Rajiv K. Singh, Erin Wysocki and Qianli R. Chu. An unsaturated hydrogen bonded network generated from three-fold symmetric carbamates. *CrystEngComm*, **2014**, *16*, 7176-7179.
7. **Xiaodong Hou,** Qiaobo Li, and Amin Cao. Solvent Annealing-Induced Microphase-Separation of Polystyrene-b-Polylactide Block Copolymer Aimed at Preparation of Ordered Nanoparticles/Block Copolymer Hybrid Thin Film. *Journal of Polymer Research*, **2014**, *21:*491.
8. **Xiaodong Hou,** Qiaobo Li, Lin Jia, Yang Li, Yingdan Zhu and Amin Cao. Synthesis and Microphase-Separation of Polystyrene-b-Polylactide Block Copolymers Aimed at Preparation of Ordered Nanoparticles/Block Copolymer Hybrid Materials. *Polymer International,* **2014**, 63:1159-1167.
9. **Xiaodong Hou**, Zhihan Wang, Joseph Lee, Erin Wysocki, Casey Oian, and Qianli R. Chu. Synthesis of Polymeric Ladders from Plant-Derived Starting Materials. *Chem. Comm.*, **2014,** *50*, 1218-1220.
10. **Xiaodong Hou**, Zhihan Wang, Molly Overby, Angel Ugrinov, Casey Oian, Rajiv K. Singh, and Qianli 'Rick' Chua. A Flexible Organic Nanosheet Framework that Accommodates and Releases Guest Molecules. *Chem. Comm.*, **2014**, *50*, 5209-5211.
11. **Xiaodong Hou,** Qiaobo Li, Lin Jia, Yang Li, Yingdan Zhu and Amin Cao. In-situ Self-assembly of enantiomeric Diblock Poly(styrene)-*b*-Poly(lactide) Via Stereocomplexation in Non-selective Solvents. *Macromolecular Chemistry and Physics* **2013,** *214*, 1569–1579.
12. **Xiaodong Hou**,Tao zhang, and Amin Cao. A Heparin Modified PP Non-woven Fabric Membrane Adsorbent for Selective Removal of Low Density Lipoprotein from Plasma. *Polymer for Advanced Technologies* **2013,** *24*, 660-667.
13. **Xiaodong Hou,** Matthew Schober, Qianli Chu. A Chiral Nanosheet Connected by Amide Hydrogen Bonds. *Crystal Design & Growth,* **2012**, *12*, 5159–5163.
14. Rajiv Singh, **Xiaodong Hou**, Molly Overby, Matthew Schober, Qianli Chu. Hydrogen Bonded Chiral Sheet Self-assembled from a C_3-Symmetric Tri-carbamate. *CrystEngComm,* **2012**, *14,* 6132.
15. Rajiv Singh, Matthew Schober, **Xiaodong Hou**, Alys Seay, Qianli Chu. Facile and Efficient Synthesis of C_3-symmetric Benzoxazine: A Novel Tri-arm Molecular Scaffold. *Tetrahedron Letters*, **2012**, *53*, 173-175.

16. **Xiaodong Hou,** Young He, Lin Jia, Yingdan Zhu, Ruilong Sheng and Amin Cao. Thermal properties and crystallization behavior of PS-b-PDLA block copolymers. *Acta Polymerica Sinica,* **2010**, *6*, 739-746.
17. **Xiaodong Hou,** Qiaobo Li, Lin Jia, Yang Li, Yingdan Zhu and Amin Cao. New Preparation of Structurally Symmetric, Biodegradable Poly(L-lactide) Disulfides and PLLA–Stabilized, Photoluminescent CdSeQuantum Dots. *Macromolecular Bioscience,* **2009**, *9,* 551-562.
18. **Xiaodong Hou**, Qiaobo Li, Lin Jia, Yang Li, Yingdan Zhu and Amin Cao. Visualization of Spontaneous Stereocomplex Aggregates by Diblock Poly(styrene)-b-Poly(l-lactide)s and Poly(d-lactide)s With New Fluorescent CdSe Quantum Dot Labels. *Journal of Polymer Science: Part B: Polymer Physics,* **2009**, *47*, 1393–1405.
19. Yang Li, Yingdan Zhu, Kejia Xia, Ruilong Sheng, Lin Jia, **Xiaodong Hou**, Yuhong Xu and Amin Cao. Dendritic Poly(l-lysine)-b-Poly(l-lactide)-b-Dendritic Poly(l-lysine) Amphiphilic Gene Delivery Vectors: Roles of PLL Dendritic Generation and Enhanced Transgene Efficacies via Termini Modification. *Biomacromolecules*, **2009**, *10*, 2284.
20. Tao zhang, **Xiaodong Hou** and Amin Cao. Irradiation Graft Copolymerization and Its Application for Preparation of Organic Biomedical Materials. *Chinese Journal of Organic Chemistry*, **2006**, *26*, 1328~1334.
21. **Xiaodong Hou** and Chuanbo Zhang. Methods of Hydrolyzing Collagen and Measuring the State of Hydrolysis. *Leather Science and Engineering*, **2005**, *15*, 35-40.
22. **Xiaodong Hou**, Chuanbo Zhang and Zhijie Wang. Studies on Preparation of Composite Material of Chrome-containing Leather Waste and Cellulose Fiber. *China Leather*, **2004**, *33*, 33-35.
23. **Xiaodong Hou,** Chuanbo Zhang, Xiongyan Xia, and Jiang Wang. The Application of Phosphate Ester Contained Softener in the Composites Composed of Collagen Fibers and Cellulose Fiber. *Paper Chemicals*, **2004**, *16*, 33-36.
24. **Xiaodong Hou**, Chuanbo Zhang, Zhijie Wang and Xiangyang Ma. Study on the Physical Property of Composite Material of Collagen Fiber and Cellulose Fiber. *Journal of Shaanxi University of Science & Technology (Natural Science)*, **2004**, *22*, 106-108.

Patents

1. **Amin Cao, Xiaodong Hou and Tao Zhang.** Preparation and Application of an Adsorbent Membrane for Selectively Removing Lipoprotein from Plasma (China Patent: 200710036406.5)
2. **Amin Cao, Xiaodong Hou and Tao Zhang.** Preparation and Application of an Porous Membrane Binding Heparin on the surface(China Patent: 200710036407.X)
3. **Amin Cao, Xiaodong Hou and Tao Zhang.** Preparation of an Porous Membrane Binding Taurine on the Surface and its Application in Removing Lipoprotein from Plasma (China Patent : 200710041060.8)
4. **Xiaodong Hou, Xiang Cai, Chuanbo Zhang and Zhijie Wang.** An Approach of Preparing Daily-use Paper Towel by Compositing Collagen and Cellulose (China Patent: 200410026132.8)
5. **Xiaodong Hou, Xiang Cai, Chuanbo Zhang and Zhijie Wang.** An Approach to Improve Strength of Paper by Using Collagen as Additive (China Patent: 200410026131.3)

MICHAEL D. MANN, PhD

Chester Fritz Distinguished Professor, Department of Chemical Engineering
Executive Director, Institute for Energy Studies, University of North Dakota

Education and Training

Mayville State University Chemistry, Mathematics B.A., 1979
University of North Dakota Chemical Engineering M.S., 1981
University of North Dakota Business Administration M.B.A., 1987
University of North Dakota Energy Engineering Ph.D., 1997

Research and Professional Experience

2014 –Present: Executive Director, Institute for Energy Studies:
Help realize the Institute for Energy Studies goal of developing UND into a premier "Energy University" that "inspires the creation of new knowledge to enable the development of revolutionary energy technologies, train the next generation of energy experts, and establish advanced industries required to make affordable emissions free energy technologies a reality". Responsibilities include identifying key technical and economic barriers to the development of secure, affordable, and reliable energy production technologies; identifying proposal opportunities and develops new relationships with potential partners; and drawing from resources across campus building teams to deliver the research, education, and outreach required to meet the needs of public and private partners.

2009-14: College of Engineering (Associate Dean 2013-14; Associate Dean for Research 2009-13):
Provide advice and support to the Dean in issues related research and development within the college and support academic affairs. Responsible for the implementation of the college's major research goals, promoting a culture of research in the college, enhancing research opportunities for faculty and students, and providing administrative oversight for proposal submittal and grant accounting.

2008: Interim Dean, UND School of Engineering and Mines:
Responsible for all academic and research activities within SEM. In this role he expanded his leadership experience and broadened his overview of the campus wide talents and opportunities for enhancing UND's reputation as a leader in energy research and education.

1999 – Present: UND Department of Chemical Engineering (Professor, 2006-present; Chair 2005-13; Associate Professor, 1999-2006):
Developed a reputation as an engaging teacher, excellent researcher, and inspirational leader. Awarded UND's highest honor, the Chester Fritz Distinguished Professorship in 2009 in recognition for his accomplishments in research, teaching, and service. Led the Department of Chemical Engineering to UND's top departmental awards for Excellence in Research in 2005 and 2011 and Excellence in Teaching in 2007. Co-founder of the SUstainable eNergy Research, Infrastructure, and Supporting Education (SUNRISE) group in 2004. SUNRISE now has over 30 faculty participants from 12 different departments and 4 North Dakota Universities with over $20 million in research grants.

1981-99: UND Energy & Environmental Research Center (Sr. Research Mgr, Advanced Processes and Technologies 1994-99; Research Mgr, Combustion Systems 1985-94; Research Engineer 1981-85):
Activities evolved from hands on research to the development and marketing of ideas and technology. Involved in a wide range of technology development, including energy production from combustion and gasification, wind, and geothermal resources. Highlights include management of over $15 million in research projects; design, installation, and operation of a 1 MW_{th} CFBC; design, installation, and operation of a 250 lb/hr gasifier; manager for project for the development of small power systems for Alaskan villages; and the development of a small-modular fluid-bed combustion system (0.5 to 5 MW)

PUBLICATIONS (selected from over 150)

1. Dale, N.N.; Mann, M.D.; Salehfar, H.; Dhirde, A.M.; Han, T.; "ac Impedance Study of a Proton Exchange Membrane Fuel Cell Under Various Loading Conditions", Journal of Fuel Cell Science and Technology, 7 (2010) 031010.
2. C.Y. Biaku, N.V. Dale, M.D. Mann, H. Salehfar, A.J. Peters, T. Han; "A semiempirical study of the temperature dependence of the anode charge transfer coefficient of a 6 kW PEM electrolyzer" International Journal of Hydrogen, 22 (2008) 4247-4254
3. A. M. Dhirde; N. V. Dale; H. Salehfar; M. D. Mann; T. Han; "Equivalent Electric Circuit Modeling and Performance Analysis of a PEM Fuel Cell Stack Using Impedance Spectroscopy" IEEE PES Transactions on Energy Conversion, TEC-00258-2009.
4. Dale, N.V.; Mann, M.D.; Salehfar, H.; "Semiemperical model based on thermodynamic principles for determining 6 kw proton exchange membrane electrolyzer characteristics" *Journal of Power Sources*, 2008, 185, pp. 1348-1353.
5. Biaku, C.Y.; Dale, N.V.; Mann, M.D.; Salehfar, H.; Peters, A.J.; Han, T.; "A semi-empirical study of the temperature dependence of the anode charge transfer coefficient of a 6 kw PEM electrolyzer", *International Journal of Hydrogen Energy*, 2008, 33 (16), pp. 4247-4254.
6. Peters, R.R.; Muthumuni, D; Bartel, T.; Salehfar, H.; Mann, M.D.; „Static VAR Compensation of a Fixed Speed Stall Control Wind Turbine during Start-up" Electric Power Systems Research, 80 (2010), 400-405.
7. Karki, S., Mann, M.; Salehfar, H.; "Substitution and Price Effects of Carbon Tax on CO_2 Emission Reduction from Distributed Energy Sources", *Asian Journal of Energy & Environment"*
8. Bandyopahdyay, G.; Bagheri, F.M.; Mann, M.D.; "Reduction of Fossil Fuel Emission in US: A Holistic Approach Towards Policy Formulation", *Energy Policy*; 2007, 35 (2) 950-965.
9. Karki, S; Kulkarni, M.; Mann, M.D.; Salehfar, H.; "Efficiency Improvements through Combined Heat and Power for On-Site Distributed Generation Technologies", *Cogeneration and Distributed Generation Journal*, Vol 22, No 3, 2007, pp 19-34.
10. Sondreal, E.A.; Benson, S.A.; Hurley, J.P.; Mann, M.D.; Pavlish, J.H.; Swanson, M.L.; Weber, G.F.; Zygarlicke, C.J. "Review of Advances in Combustion Technology and Biomass Firing". *Fuel Processing Technology* 2001, 71 (1-3), 7-38.

POSTDOCTORAL SCHOLAR: Eduardo Hernandez Pacheo, 2005, Intel; Motjaba Sadraemli, 2009, University of Tehran, Iran.

PH.D. ADVISEES: Eduardo Hernandez Pacheo, 2004, Intel; Yongxin Zhao, 2005, ARCADIS, Shankar Karki, 2006, Electric Power Research Institute; Kevin Harrison, 2006, National Renewable Energy Laboratory; Rhonda Hill, 2007, Clipper Energy; Gopal Bandyopadhyay, 2007, Pacific Northwest National Laboratory; Philip Hutton, 2009, Energy & Environmental Research Center; Nilesh Dale, 2009, Bhanuaben Patel, 2010; Taehee Han, 2010, Nissan; Christian Biaku, 2010; Ryan Knutson, 2011; Udhe Corp of America; Kirtipal Barse, 2014, UND; Stacy Bjorgaard, 2015 UND. Jivan Thakare, 2016, Energy & Environmental Research Center (I currently advise two Ph.D. students)

M.S. ADVISEES: Jermie Moberg, 2001, Intel; Jason Jacobson, 2001, Trane; Muhammad Amanullah, 2003, University of Minnesota-Duluth; Biplab Mukerjee, 2004, Washington University, St. Louis, Ph.D. program; Kyle Martin, 2004, EERC; Devinder Singh, 2005, Kansas State University, Ph. D. program; Devdutt Shukla, 2005, University of Missouri-Rolla, Ph.D. program; Srinivas Koli, 2005, University of Kentucky, Ph.D program; Joshua Strege, 2005, EERC; Carol Horabik, 2006, University of Minnesota-Duluth; Xi Hong, 2006, Michigan Tech, Ph.D. program; Prasana Sardenia, 2006, Microbeam Technologies; Christopher Kinchin, 2006, National Renewable Energy Laboratory, Prasad Chavan, 2007, Hutchinson Technologies; Taskin Karim, 2007, University of Texas; Marika Nel, 2007, Northwest University, South Africa; Mohammed Hussain, 2007, Kansas State University PhD program; Jason Hrdlicka, 2007, National Renewable Energy Laboratory; Rahul Pandry, 2008, private company in India; Taskin Karim, 2010, University of Houston; Shankar Lande, 2011; Sam Cowart, 2012, West Point Military academy; Kirt Leadbetter, May 2014; Chris Haugen, 2015, Hess Corporation. (I currently advise two MS students)

Yong Hou, Ph.D.

Email:hou@cleanrepublic.com Phone: 218-791-3746

BUSINESS AND WORKING EXPERIENCE

2008-present Co-founder, Vice President of Research
Clean Republic SODO LLC, Grand Forks, North Dakota
- Direct nano-sized LiFePO4 powder research as ideal cathode material for lithium ion battery
- Direct lithium battery packs development and production

2008 - 2012 Adjunct Professor
Department of Technology, University of North Dakota

2007- 2008 Vice President of Product Development
Neosonic Li-Polymer Energy (Zhuhai) Corporation
- Direct the design and development of new Lithium Polymer battery using for light electric vehicles
- Direct the Neosonic business department in mainland China
- Taking charge of ERP system and Testing Laboratory

1995-2002 Founder/General Manager
Shanghai Zhongdian International Company
- Founded and managed company's computer service business into the largest wholesaler of Compaq Computers in eastern China with an annual revenue of $13 million USD and 50 plus employees
- Made the company one of the two contractors of Warehouse Supplier for Compaq (China), and took charge in its parts imp/export in eastern China
- Leaded software product team designing and maintaining 'Management Information System' (MIS) projects for enterprises

1992-1995 Department Manager
Shanghai Branch Company of Chinese Electronics Group

1983-1989 Electronics Engineer
Hunan Puyuan Engineering Machinery Company

EDUCATION BACKGROUND

2002-2007 Ph. D., Management Science and Engineering
University of Shanghai for Science and Technology

1989- 992 Master of Science, Systems Engineering
University of Shanghai for Science and Technology

1979-1983 Bachelor of Science, Electronics Engineering
Hunan University of Art and Science

RESEARCH and PRODUCTS DEVELOPMENT

Ph. D. Dissertation
- A Systemic Optimization Model of Renewable Energy Substitution

Master Program
- Delaminating and Systems Analyzing of Corporation Cost by its Historic Data.

Research Projects Leaded and Participated
- Technical Feasibility Analysis of Distilling Biodiesel from Cultivated Microalgae
- Systemic Analysis of Optimized Reduction of Greenhouse Gases with Renewable Energy (Principle Investigator)

- Development of a Site Assessment Instrument for Small Wind Turbine Development in North Dakota (Co-investigator with Dr. Alex Johnson)
- Redesigning Residential Wind Turbine (RWT) for Rural Areas with Abundant Wind Resources (Principle Investigator, with Dr. Jing Shi)
- Development of Innovative Modular Lithium Iron Phosphate Battery Packs for Energy Storage (Principle Investigator, with Michael Shope)
- Synthesis of ordered, nano-sized spherical LiFePO4 particle with 99% purity and high tap density as ideal cathode for lithium ion battery (Principle Investigator with Michael Shope and Dr. Xiaodong Hou and Dr. Michael Mann)
- Management Information Systems (MIS) of commerce bank's loan management
- Lithium polymer battery packs for golf-cars
- Hill Topper electric conversion kit (with Michael Shope)
- 12V lithium battery module (Dakota Lithium) with standard size and enhanced low temperature performance
- 58V lithium battery pack for ice fishing augers
- 240V lithium battery pack for water pump system driven by solar energy

GRANTS

- Business and Public Administration & Law Funding, PI, $4,835, May, 2011
  Title: **Development of a Site Assessment Instrument for Small Wind Turbine Development in North Dakota**
- Research ND, Venture Grant Phase I/II, $148,105, Co-PI, Dec 2016,
  Title: **A LOW COST AND REPRODUCIBLE SYNTHETIC PROCEDURE FOR MASS PRODUCTION OF LITHIUM IRON PHOSPHATE CATHODE MATERIALS FOR LITHIUM ION BATTERIES**

AWARDS

- **Innovate ND** Venture Competition, North Dakota, 2010
  (Award product: Hill Topper Electric Bike Kit)

PUBLICATIONS

1. Yong Hou and Luke Huang. "**The Present State of and a Suggested Inserting Point for Biodiesel Production Industrialization in China**", 2006 IEEE International Conference on Service Operations and Logistics, and Informatics Proceedings, Page 679-683, ISBN: 1-4244-0318-9 (EI Accession Number: 074610921148), June 2006.
2. Yong Hou and Fuyan Xu. "**The Development of Biodiesel Industrialization**", Commercial Research, ISSN: 1001-148X, October 2006.
3. Jinrong Zhen and Yong Hou. "**Research on an Evaluation Model of Technique Innovation**", 2006 IEEE International Conference on Service Operations and Logistics, and Informatics Proceedings, Page 187-191, ISBN: 1-4244-0318-9, June 2006.
4. Luke Huang and Yong Hou. "**Manufacturing information recognition, analysis, and simulation using Windows based programs**", The 5th Wuhan International Conference on e-Business: Integration and Innovation through Management, Vol. 3, pp1931-1937, May 2006.
5. Zheng Jin-rong, Xu Fu-yuan, Hou Yong, "**The Selection of Stock Options for Venture Firms**", Commercial Research, 2006.18,1-5
6. Yong Hou, Fuyuan Xu and Wei Cheng, "**A Sustainable Growth Model with the Utilization of Renewable-Energy**", 2007 IEEE International Conference on Communications, Services, Knowledge and Engineering, Page 5012-5015, ISBN: 1-4244-1311-7, September 2007.
7. Yong HOU, Fu-yuan XU, Wei CHENG. "**A Microeconomic Model of Optimized Investment Project on the Substitution of Renewable Energy**", Commercial Research, 2008-05.
8. Yong Hou, Fuyuan Xu and Wei Cheng. "**A Sustainable Growth Model Based on the Substitution of Renewable Energy**", Systems Engineering – Theory & Practice, Vol. 28 (9): 67-72, September 2008.

9. S.M. Hanson, A.L Johnson, Yong Hou, M.D. Hellwig (2012). "**Recharging Centers for Disease Control Light Trap Batteries with Solar Panel**": International Journal of Applied Science and Technolog , Vol.2 No.7, September 2012.

10. Yong. Hou, Y. Peng, A.L. Johnson, J. Shi (2012). "**Empirical Analysis of Wind Power Potential at Multiple Heights for North Dakota Wind Observation Sites":** Energy Science and Technology ISSN 1923-8460 [Print] ISSN 1923-8479 [Online] Vol.4 No.1, August 2012.

11. Yong Hou, Jing Shi and Alex Johnson; **Empirical analysis of capital accumulations in constant price and the role of energy consumption on China's economic growth**, International Green Energy Conference, IGEC-2016-1017, May, 2016

12. Weidong Wu, Jun Wu, Jiawei and Yong Hou; **Absorption properties of ionic liquids-CO2 as new working pairs used for absorption refrigeration system powered by low-grade heat energy**, International Green Energy Conference, IGEC-2016-1022, May, 2016

13. Weidong Wu, Yong Hou, Jun Wu and Lin Su; **Predicting phase behavior of CO2 and imidazole ionic liquids as new working pairs in absorption refrigeration system using GC-EOS method**, International Journal of Thermal Sciences, June, 2016

14. Hongbo Zhu, Yan Gao Yan and Yong Hou; **Real-time pricing strategy for smart grid based on Markov decision processes**, Systems Engineering Theory and Practice, Nov. 2017

Michael Shope

1222 20TH Avenue East, Seattle WA 98112
Phone: (218) 779 3136 Email: mike@cleanrepublic.com

Education

- Bachelor of Science Degree in Aeronautics with an emphasis in Commercial Aviation from the University of North Dakota, Track in Entrepreneurship.
  Enrolled: September, 2000. Graduated: December, 2004
- Semester Abroad: American College of Thessaloniki, Greece. 12 credits completed.
  Enrolled: January, 2002 – June, 2002

Experience

- Business founding, marketing and communication. 2004-Present
  - Founded Clean Republic LLC with Dr. Yong Hou. See CleanRepublic.com 2008
  - Authored and edited two business and marketing plan documents with financials.
  - Edited and compiled: LLC registrations, bylaws, and management contracts.
- Commercial Pilot, AMEL, CFII.
- Founded Pilot Friendly Products, LLC in 2003.
  - Negotiated $15,000 seed funding from Center for Innovation.
  - Developed marketing and sales systems, and prepared invoices.
  - Created and administered company e-commerce website.
  - Sold customer base and entire inventory to an industry leader.
- Presentation and instruction experience.
  - Delivered business plan and marketing presentations for clients and customers.
  - Commercial flight instructor, JDO School of Aerospace Sciences 2004-2005
  - Prepared lesson plans and instructed single and multiple students.

Employment Experience

- Product Specialist, Aviation Supplies & Academics, Bellevue, WA
  - Invent, source production for new products. Google "ASA Tri-Lite, Hoodwink."
  - Collect market feedback for product development cycle (over 300 products).
  - Prepare reports on customer service systems for management.
  Employed: October, 2006-Jun 2008
- Non-Profit Manager, Outdoor Education Post, Garfield High School, Seattle, WA
  - Advise over 50 student staff, chair and facilitate community meetings.
  - Provide organizational and leadership reports and advice to the Board of Directors.
  - Oversee $90,000 annual budget.
  Employed: August, 2007- June 2009
- Founder and CEO – Clean Republic LLC
  - Developed new practical retail products for alternative energy industry
  - Invented solutions for electric bike throttle/motor control and lithium ion batteries
  - Manage sales, advertising, and accounting.
  Managing: 2008 - Present

Personal Information and Honors

- Vice President of Delta Tau Delta Fraternity: 50 members, $200,000 annual budget.
  National Top 10 Awarded Chapter. November, 2002 – November, 2003
- Outward Bound leadership academy graduate July, 2003
- President's Honor Roll: University of North Dakota Spring/Fall, 2003
- Dean's List: College of Aerospace Sciences Spring/Fall 2002/2003
- Jack Wright Entrepreneurship Award
- Certified keel boat skipper
- Innovate ND Venture Competition 2010
- SBA Regional Young Entrepreneur of the Year 2011

Youde Mo Ph.D

Boston Global Technologies LLC
40 Alden Place, West Newton, MA 02466
617-505-1393

EDUCATION AND TRAINING

1989.9 – 1992.6: PhD, Institute of Physics, Chinese Academy of Science, China
1986.9 – 1989.6: Graduate Student, Institute of Theoretical Chemistry, Jilin University, China
1979.9 – 1983.6: Undergraduate Student, Guangxi University, China

PROFESSION EXPERIENCE

2014 - present: CEO, Boston Global Technologies LLC, USA
2014.4 - present: CEO, Guangxi Nowphene Energy Storage Technologies Ltd, China
2006.3 - 2012.3: CEO, Guangxi BMB Technologies Ltd, USA
2003.5 – 2005.7: Senor Consultant, State Street Bank, Fiedelity Investment Group, USA
2001.1 – 2003.3: Senor Engineer, MagnSource LLC, USA
1997.8 – 1999.12: Postdoc Scientist, Boston University, USA
1995.12 – 1997.7: Postdoc Scientist, Chicago University and Columbia University, USA
1994.7 – 1995.8: Visiting Scholar, University of York, UK
1992.7 – 1994.6: Research Associate, Institute of Physics, Chinese Academy of Science, China
1983.9 – 1986.8: Lecturer, Guangxi Normal University for Nationality, China

SELECTED PUBLICATIONS

1. **Mo, Y.D.**, Campos, B., Mealy, T.R., Commodore, L., Head, J.F., Dedman, J.R., Seaton, B.A. Interfacial basic cluster in annexin V couples' phospholipid binding and trimer formation on membrane surfaces. J. Biol. Chem 278, 2437-43(2003).
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3. Capila, M.J. Hernaiz, **Y.D. Mo**, T.R. Mealy, B. Campos, J.R. Dedman, R.J. Linhardt, and B.A. Seaton "Annexin-V Heparin Oligosaccharide Complex Suggests Heparan Sulfate-Mediated Assembly on Cell Surfaces." Structure" 9, 57-64(2001).
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5. Campos, M.B., **Mo, Y.D.**, Mealy, T.R., Li, C.W., Swairjo, M.B., Balch, C., Head, J.F., Retzinger, G., Dedman, J.R. and Seaton, B.A. Mutational and crystallographic analyses of interfacial residues in annexin V suggest direct interactions with phospholipid membrane components. Biochemistry 37 (22), 8004 -8010(1998).
6. Fan Hai-fu, Wan Zheng-hua, Li Jian-qi, Fu Zheng-qing, **Mo You-de**, Li Yang, Sha Bing-dong, Cheng Ting-zhu, Li Fang-hua, Zhao Zhong-xian. "Multi-dimensional electron crystallography of Bi-based superconductors" in Book titled "Electron Crystallography" Ed. by D. L. Dorset, S. Hovmöller & X. ZouKluwer Academic Publishes, The Netherlands, 1998, pp. 285-294.
7. X.F. Zheng, Y.X. Gu, C.D. Zheng, **Y.D. Mo**, H.F. Fan, Q. HaoA procedure for ab initio phasing of one-wavelength anomalous scattering data from proteins - combining direct methods with the solvent flattening technique Zeitschrift fur Kristallographic 212,95-98(1997).
8. Xiao-feng Zheng, chao-de Zheng, Yuan-xin Gu, **You-de Mo**, Hai-fu Fan, Q. HaoUse of single isomorphous replacement data of proteins - resolving the phase ambiguity and a new procedure for phase extension. Acta Cryst. D53, 49-55 (1997).

9. Mo **<u>You-de</u>**, Fu Zheng-qing, Fan Hai-fu, Sander Van Smaalen, Erwin J.W. Lam, Paul T. Beurskens Direct methods for incommensurate intergrowth compounds. III. solving the average structure in multidimensional space Acta Cryst. A52, 640-644(1996).
10. Fan Hai-fu, Wan Zheng-hua, **<u>Mo You-de.</u>** Application of direct methods in electron crystallography (lecture at the XVIIth IUCr Meeting) (1996).
11. **<u>Y D Mo</u>**, T Z Cheng, H F Fan, J Q Li, B D Sha, C D Zheng, F H Li and Z X Zhao Structural features of the incommensurate modulation in the Pb-doped Bi-2223 High-Tc phase revealed by direct-method electron diffraction analysis Supercond. Sci. Technol. Vol.5,2 ,69-72(1992).
12. **<u>Y D Mo</u>**, T Z Cheng, H F Fan, J Q Li, B D Sha, C D Zheng, F H Li and Z X Zhao Incommensurate modulation of the Bi-2223 superconducting phase observed by direct-method electron diffraction analysis Phys. Scr., T42, 18-19(1992).
13. **<u>Y.D. Mo</u>**, T.Z. Cheng, H.F. Fan, J.Q. Li, B.D. Sha, C.D. Zheng, F.H. Li, Z.X. ZhaoElectron crystallographic study of incommensurate modulation in the Pb-doped Bi-2223 high Tc superconducting phase (lecture presented by Fan Hai-fu in USA, 1992).
14. **<u>Mo Y.D</u>**., Cheng T.Z., Fan H.F., Li J.Q., Sha B.D., Zheng C.D., Li F.H. & Zhao Z.X., Incommensurate modulation of the Bi-2223 superconducting phase observed by direct-method electron diffraction analysis. Nobel Jubilee Symposium, Gothenburg, Sweden, December 1991. Published in Low Dimensional Properties of Solids, Proceedings of the Nobel Jubilee Symposium. Ed. by Mats Jonson & Tord Claeson. Physica Scripta, The Royal Swedish Academy of Sciences. World Scientific, Singapore 1992, pp. 18-19.

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1. Huang, G.; Mo, B.; Mo, Y. Methods of making low cost electrode active materials for secondary batteries from ilmenite. US 201414485016 20150076750, A1 20140912.
2. Huang, G.; Mo, B.; Mo, Y. Cabinet for high-temperature sintering. CN 2015-20128994 204474330, 20150306.
3. Huang, G.; Mo, B.; Mo, Y. Method for preparing energy battery active electrode material by traditional process of ilmenite. CN 2015-10158529 104934587, 20150407.
4. Huang, G.; Mo, B.; Mo, Y. Methods of making low cost electrode active composite materials for secondary electrochemical batteries. US 201313971252 20140054494, 20130820.
5. Huang, G.; Mo, Y.; Mo, B. Revolving furnace used for preparing lithium iron phosphate battery material. CN 2012-20395706 202808368, 20120810.
6. Huang, G.; Mo, Y.; Mo, B. Reaction apparatus for preparing nanoscale ferric phosphate. CN 2012-20522949 202864927, 20121014.
7. Huang, G.; Mo, Y.; Mo, B. Environment-friendly apparatus for iron powder acidification. CN 2012-20582528 202861393, 20121107.
8. Huang, G.; Mo, Y.; Mo, B. Heating apparatus for reaction kettle. CN 2013-20157192 203140006, 20130401.
9. Huang, G.; Mo, Y.; Mo, B. Manufacturing of active phosphate electrode material for lithium ion battery. CN 2013-10403393 103456954, 20130906.

APPENDIX F

AN ALTERNATE BUDGET PLAN THAT INCLUDES ADDITIONAL INVESTMENT IN CLEAN REPUBLIC

Christer Staaf is the leading investor of Aifloo AB, a Sweden private investment firm. He and his investment partners are currently processing a final step in terms of investing in Clean Republic. Their main interest is in the Dakota Lithium Battery and the LFP powder project. With the joint research work the University of North Dakota and the Clean Republic is doing, Christer's group firmly believes this investment meets their investment criteria on the lookout for game changing technologies. "We see the economic and environmental potential in this technology as being substantial and believe investments in such a company to be a significant opportunity", Christer claimed in his support letter. In consideration of the highly possible $1.5 millions USD investment, Clean Republic will raise its match funds to $457,873 for this research project if it receives the investment from Christer's group.

The raised match funds Clean Republic plans for this research project is $168,000. It will be used in a) increase expense of analysis and test of full size cells (18650/26650) from $18,000 to $36,000, which will allow doubling the test rounds of battery smaples the basic portfolio depicted ; b) purchase an industrial pusher furnace with high accurate atmosphere control. It will cost more than $150,000 and secure the synthesis of powder achieving high productivity and beter quality.

Table A4. An alternative budget plan for the project

Project Associated Expense	NDIC's Share	Applicant's Share (Cash)	Applicant's Share (In-Kind)	Other Project Sponsor's Share
Direct Cost-Personnel Salaries				
Michael Mann				
Xiaodong Hou	$ 29,652			
Graduate Research Assistant	$ 21,600			
Undergraduate Research Assistant	$ 14,400			
Yong Hou				$ 43,800
Michael Shope				$ 37,600
Technician				$ 38,080
Direct Cost-Fringe benefits	$ 13,640			$ 41,818
Direct Cost-Travel				$ 9,000
Direct Cost-Materials and Supplies	$ 23,000			$ 10,000
Direct Cost-Analysis and Test	$ 35,000			$ 18,000
Direct Cost-Equipment	$ 37,000			$ 150,000.00
Indirect Cost	$ 64,074			$ 49,575
Total	$ 238,366			$ 397,873
Total cost	$ 636,238			
NDIC Share	$ 0.37			

With new investment from Christer's group, the alternative budeget plan of this research project is listed in **Table A4**. If this additional investment in Clean Republic comes to fruition, Clean Republic is pleased to offer this additional cost share to the proposed project. However, this additional funding is not yet a certaintly, and thus we must offer the proposed project budget as summarized previously without the additional $168,000. We note, however, that in the likley event that the additional matching funds will be available, we will communicate that with the NDIC and the Renwable Energy Council immediately. If necessary, a more detailed revised project scope of work can be submitted at that time. It should also be noted that the proposed project objectives can be obtained without the additional funding from Clean Republic.

APPENDIX G

SUPPORT LETTERS

November 12, 2018
Dr. Michael Mann
Executive Director, Institute for Energy Studies, University of North Dakota
2844 Campus Road, Stop 8153, Grand Forks, ND58202.

Subject: Letter of Support for proposal entitled "Preparation of Graphene-Modified $LiFePO_4$ Cathode for Li-Ion Battery" submitted to NDIC Renewable Energy Program.

Dear Dr. Mann:
We are excited to team up with the University of North Dakota (UND) Institute for Energy Studies (IES) on this application to continue our collaborative development of low cost, high quality lithium iron phosphate cathode materials. We believe the proposed technology has the opportunity to further improve the performance and lower the cost of lithium ion batteries. Such a high performance lithium ion battery is exceptionally important to energy storage technology to support renewable energy generation in North Dakota. In this collaborative project, work will be conducted by the University of North Dakota (UND) Institute for Energy Studies (IES) and Clean Republic LLC. The scope of the activities is summarized as follows:

Task 1. Optimization of Humic Acid Extraction Procedure: Clean Republic will work with IES team to optimize the extraction procedure to produce high purity (>99%) and metal-free humic acid.

Task 2. Optimization of LFP/G Synthetic Procedure. With the backbone of the procedure established in the ongoing project, this task focuses on optimizing process parameters to yield a high content of graphene and thus improve the specific capacity of the cathode materials to ≥150 mAh/g. LFP/G sample preparation and physical properties testing will be done at UND IES. The following parameters will be optimized: precursors feeding ratio, solid content in the slurry, preheating temperature and time, and sintering temperature and time. The software Minitab will help optimize and investigate the interaction between the parameters. A Taguchi orthogonal array design $L8(2^7)$ with 6 factors at 2 levels and a total of 8 runs will be implemented. A second run of design of experiments will be run to further optimize levels of major factors.

Task 3. Characterization of LFP/G Cathode. In this task, a series of analytical technologies will be applied to confirm 1) the graphene is really in situ-formed on the surface of LFP particles and 2) the LFP/G powder meets the physical requirements of a LIB cathode with the details as follows: Graphene will be confirmed by a Raman spectrometer at UND. The morphology of graphene on the LFP particles will be observed under a high-resolution transmission electron microscope (HRTEM) and/or field emission scanning electron microscope (FEG-650, FEI, USA) at IES. Crystal purity will be measured by a Smartlab XRD (Rigaku, Japan) available at IES. Carbon content will be tested by a TOC (total organic carbon) analyzer (Shimadzu, Japan) at IES. Tap density will be measured following the ASTM B527-15, with the apparatus available at IES. Trace impurities of LFP powder will be measured by a Supermini 200 XRF (Rigaku, Japan) at

IES. Particle size distribution of LFP/G powder will be measured by a Marvin ZS90 zeta Sizer at UND.

Task 4. Pilot scale production testing: this task is to evaluate the reproducibility of the optimized procedure at pilot scale. This task will be conducted by Dr. Yong Hou leading one technician at Clean Republic. The scale of the LFP/G production will be gradually increased from 20g, to 200g, to 2kg, to 20kg per batch, and at each scale multiple (>3) production batches will be repeated to evaluate the quality and consistency. Based on the feeding ratio of humic acid for LFG/G production, the equipment for lab-scale humic acid extraction can produce efficient amount for pilot scale production of LFP/G.

Task 5. Electrochemical Performance Testing of LFP/G Cathode. This task is to confirm that the LFP/G has a better electrochemical performance than its commercial LFP counterparts with the details as follows: CR2032 coin-type cells (for lab-scale sample) and full size 18650 and 26650 cells (for pilot-scale sample) will be prepared and tested by Clean Republic. The equipment for the full size cell fabrication needs to be purchased, and all the other equipment is currently available at Clean Republic. Electrochemical performance testing will be conducted on a Neware CT-3008 battery testing system (Neware Technology Limited, Shenzhen, China) available at Clean Republic. Current densities and specific capacities will be calculated based on the mass of the target compound of the electrode. Cycle life will be evaluated by using a three-electrode system (ECC-REF, EL-Cell GmbH, Germany) for cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) tests available at Clean Republic.

The total cost of the proposed work is $486,238 as summarized in the budget form and Clean Republic will offer a co-share of $247,872 in total. The project duration of 24 months is expected to begin in January 1, 2018. Key personnel on the Clean Republic team for this project include Dr. Yong Hou who will serve as a co-PI and contact person for Clean Republic, and lead one technician on the battery assembly and testing program, and Mr. Michael Shope who will serve as a business advisor and coordinate the overall Clean Republic efforts. We look forward to working with UND IES on this exciting topic. If there are any questions, please do not hesitate to contact us at the letterhead address or at the contact information provided below.

Sincerely,

Michael Shope
CEO of Clean Republic SODO LLC
1222 20TH Avenue East, Seattle WA 98112
Phone: (218) 779 3136
Email: mike@cleanrepublic.com

LEONARDITE
PRODUCTS·LLC

August 2, 2017

Dear Dr. Hou,

I was exciting to learn more about your current work which incorporates humic acid into the production of lithium ion phosphate batteries. As you know, Leonardite is a great source of humic acid, and in fact, we have agriculture product lines that are leonardite based humates.

We are always looking for new markets for our product and see this as a good opportunity. We expect the battery industry to grow rapidly as more intermittent energy resources are brought on line, and would be happy to be able to profit from your developing technology.

We would not have any problem supplying the quantities you suggest for your near term-term market (five to ten tons per year). We would hope that this market grows as your battery technology is developed and fully commercialized.

We will be happy to provide you with samples to use for your research. By working together we can ensure that the material you are testing is fresh and is representative of the products that we sell.

Thank you for the opportunity to work with you on this project and best of luck with the grant application.

Sincerely,

Cherie Harms

Cherie Harms
President

Christer Staaf
Hällby Gårds Road 21
746 93 Bålsta
+46767136312
christer@aifloo.com
Sweden
November 14, 2017

Industrial Commission of North Dakota
Ref: Preparation of Graphene-Modified LiFePO4 Cathode for Li-Ion Battery

To whom it may concern:

Over the last four months I have had the opportunity to conduct an in depth due diligence on the company Clean Republic including its battery sales and research for the purposes of Capital Funding. Having conducted many due diligences over the last 7 years I have been extremely impressed with the professionalism and knowledge expertise's found within Clean Republic.

At the start of November, I had the opportunity to visit and meet the Clean Republic and North Dakota Research staff at their research facility on the campus of the University of North Dakota. I was astonished at the level of knowledge and expertise exhibited within the group and further impressed by the facilities offered. Based on my experience in equity placements it is clear there is exciting opportunities developing with this partnership. The ability to produce lithium battery powder without the need to include Cobalt and Nickel is truly exciting not only from a business prospective but my personal beliefs of obtaining and investing in environmentally friendly products.

Representing a large group of Swedish investors, we are always on the lookout for game changing technologies. With the work the Clear Republic and the University of North Dakota is doing our group firmly believes this investment meets that criteria. We see the economic and environmental potentials in this technology as being substantial and believe investments in such a company to be a significant opportunity. Our long term plan is to scale both powder and battery production and would greatly enjoy keeping these operations in the state of North Dakota.

I support the research Clean Republic and the University of North Dakota is doing and believe there are strong financial and environmental benefits in doing so.

Please contact me directly if you have any questions.

Kind regards,

Christer Staaf

Jemco Power Saver
WEST FARGO, ND
1-701-281-0362
Electric Motor Controls

Attn: Dr. Michael D. Mann
Executive Director, Institute for Energy Studies
University of North Dakota
234 Centennial Drive
Grand Forks, ND 58202

Ref: support the proposal entitled "Preparation of Graphene-Modified LiFePO4 Cathode for Li-Ion Battery" submitted to Renewable Energy Program, North Dakota Industrial Commission

Dear Dr. Mann,

I am glad of hearing that your ongoing research of LFP powder is going very well, and you are proposing further improvement on the technology of preparing high-performance, graphene-modified LFP cathode material (LFP/G) for lithium batteries, which will potentially help Clean Republic, our battery supplier meet our demand on high quality LFP/G lithium batteries with low cost.

One of our agricultural applications is the agricultural drain tile that is installed in fields to help keep the water table levels in farm fields down during rainfall events. We have been working on a developing solar/battery option for this application. Globally, solar pumping has increased in usage, and ourselves, as well as our customers see the potential to this application.

Along with the solar pumping application, we have also been working with on two other applications that use batteries from Clean Republic that are directly related to the drain tile application.

1) A solar/battery powered drain tile pump.
2) A solar/battery powered valve that used as control structure for agricultural drain tile to control water flow in many drain tile discharge configuration.
3) A solar/battery powered charging system and control system for a generator/solar/battery powered drain tile pump.

We have been also working on other applications using Clean Republic, and we are confident that this business model will be an attractive option to prospective customers.

As agricultural applications have increased with the adaption of cut-edge technology, the ability to proactive with field conditions has given farms the ability to increase yield by automating the flow of water below soil surface. The ability to use emerging technology to control water table levels is the future of farming, and we are in an enviable position to have partners like University of North Dakota and Clean Republic to help develop these ideas into application to help farmers in America, and the world.

Thanks for the opportunity to discuss one of our emerging markets with our partnership.
Sincerely,

Josh Johnson
Jemco Power Saver
Drain Tile Manager
Jemco Inc.

Mueller Electronics
801 Clayride Dr.
Wakarusa, IN 46573

31 July 2017

Attn: Dr. Michael D. Mann
Executive Director, Institute for Energy Studies
University of North Dakota
234 Centennial Drive
Grand Forks, ND 58202

SUPPORT FOR UNIVERSITY OF NORTH DAKOTA'S RENEWABLE ENERGY PROGRAM - GRAPHENE-MODIFIED LiFePO4 CATHODE FOR LITHIUM BATIERY RESEARCH

Dear Dr. Mann,

It has come to our attention that your team and Clean Republic are researching a way to produce the raw materials for lithium batteries inside the U.S. in a way that will potentially yield higher quality, higher performance, and potentially even lower cost than those batteries currently available.

As a customer, already happy with Clean Republic's current products, we are happy to present this statement of general support and encouragement for your proposal to the Renewable Energy Program, North Dakota Industrial Commission titled "Preparation of Graphene-Modified LiFePO4 Cathode for Li-ion Battery" that you are currently submitting.

We are currently using Clean Republic's batteries in GPS Correction Stations for providing high accuracy Real Time Kinematic corrections to Agricultural users and it would be an excellent step forward for our U.S. economy if local research institutes and companies like Clean Republic could develop these technologies even further. To have a U.S. manufacturer increasing the quality and performance of these batteries' raw materials, in this field, as your research intends to do, would benefit not only our specific industry, but the national economy as well, since the application of lithium batteries will continue to proliferate in many industries for decades to come.

Best wishes and good luck with your efforts!

Matt Lindsey
System Engineer

CRC
CENTRAL RADIO COMPANY INC
VOSB
cVE

Central Radio Company Inc.
P O Box 6348
1083 W 39th Street
Norfolk, VA 23508

Date: November 16, 2017

Attn: Renewable Energy Program,
North Dakota Industrial
Commission

SUPPORT FOR CLEAN REPUBLIC'S PREPARATION OF GRAPHENE-MODIFIED LiFePO4 CATHODE FOR LI-ION BATTERY RESEARCH PROPOSAL

Dear Michael Shope & Dr. Hou,

It has come to our attention that your team is researching a way to produce the raw materials for lithium batteries inside the U.S. in a way that will potentially yield higher quality, higher performance, and potentially even lower cost than those batteries currently available.

As a customer already happy with your current products, we are happy to present this statement of general support and encouragement for your proposal to North Dakota state titled In-situ Preparation of Graphene-Modified LiFePO Cathode for Li-ion Batteries that you are currently submitting.

We are currently using your batteries in our wireless announcing system, remote board locations in ship yards in support of ship repair for the local ship yards, and it would be an excellent step forward for our U.S. economy if local companies like yours could develop these technologies even further. To have a U.S. manufacturer increasing the quality and performance of these batteries' raw materials, in this field, as your research intends to do, would benefit not only our specific industry, but the national economy as well, since the application of lithium batteries will continue to proliferate in many industries for decades to come.

Best wishes and good luck with your efforts!

Sincerely,

Kelly Dickinson
LMR Manager