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THE UNIVERSITY OF NORTH DAKOTA

CO-PRODUCTION OF MULTIPLE HIGH VALUE SOLID PRODUCTS FROM LIGNITE COALS

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1. ABSTRACT

The University of North Dakota (UND), led by the principal investigator Dr. Michael Mann, in collaboration with Clean Republic LLC, Microbeam Technologies, Inc, and Barr Engineering Co. is proposing to develop a synergic process that co-produce multiple high-value solid products from North Dakota lignite coals, through integration of several successful projects performed at UND the Institute for Energy Studies (IES). The objectives of this proposed project include: 1) Build on current efforts to produce a high-value ore containing >90% REE and other critical elements, leaving a clean lignite by-product; 2) Utilize the clean lignite by-product as a feed stock for extracting humic acid from lignite; 3) Develop two innovative processes to prepare advanced anode materials for Li-ion Batteries (LIBs) from the lignite-derived humic acid, and 4) Determine the chemical and physical properties of the lignite extraction residue for use as a char or activated carbon. The expected technical and economic impacts are following: 1) it will significantly reduce the overall production cost, and maximize the low-rank coal value, meanwhile reduce environmental impacts by limiting waste streams; 2) it will greatly advance all the three sub-processes that this proposed process is built on towards the commercialization: REE extraction from North Dakota lignite coals, Valley City State University steam plant upgrade coupling activated carbon production with steam production, and NDIC/Clean Republic sponsored projects to develop advanced electrode materials for LIBs. This project directly targets the goal of the current FOA -- developing technologies that use domestic U.S. raw coal to produce other marketable solid products or feedstocks for making other high-value solid products. The proposed technology provides a possible solution to the key challenges facing the coal industry in a carbon-constrained world and is closely relevant to the Office of Fossil Energy's mission and technology areas.

2. PROJECT SUMMARY

The University of North Dakota (UND) Institute for Energy Studies (IES) is teaming with Microbeam Technologies Incorporated (MTI), and Barr Engineering Co. to develop high-value materials, including rare earth element (REE, lanthanide series + scandium and yttrium) concentrates, battery anode materials,

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and enhanced fuel and char materials from North Dakota lignite coal feedstocks. The project will be conducted with funding from the U.S. Department of Energy, North American Coal Corporation, BNI Energy and cost-share support from Clean Republic.

North Dakota lignite coal has been discovered with REE levels as high as anything ever reported previously for U.S. coals. In lignite coal, the REE are weakly bound as organic complexes, rather than in mineral forms that are typical of higher-rank coals. These organic associations permit simple dilute acid leaching directly from the lignite. The technology is much less complex than most REE mineral processing methods, potentially offering significant cost savings. Also, the leaching process is also a coal beneficiation process, offering value-added opportunities for the upgraded lignite byproduct. During previous work, REE oxide concentrates of over 85% purity were produced from lignites with over 650 ppm REE with favorable economic projections.

Additionally, lignite coal contains a considerable quantity of organic acids, including humic and fulvic acids, which are excellent precursors for next generation battery anode materials. Carbon-based battery materials have been shown to be highly effective in use, but may suffer from impurities related to the carbon source and high cost of synthetic carbon sources. Beneficiated coal from the REE processing contains significant value for these applications, primarily due to removal of organically-bound ash constituents, allowing for increased purity and reduced battery poisons when produced from coal. These battery materials produced from coal have the potential of reducing total battery costs.

The resultant material generated from multiple extractions from the coal, first ash and then highly oxygen-functionalized organic acids, is expected to produce char-like materials with properties akin to high-value products, such as metallurgical coke or high-grade activated carbon. This project will evaluate potential applications for these valuable properties to determine the most beneficial markets.

The project will largely be conducted at the lab-bench scale, with a resultant total process TRL of 4, and represents an alternative path for use of lignite coal for high-value products. This includes development of domestic sources of REE, significantly reducing battery costs and potential supply opportunities, and

development of carbons and fuels from low-rank coal with similar properties to higher-rank coals. The proposed work will lay strong foundations for subsequent continuous process integration efforts at the pilot scale, and further commercialization of the technology.

3. Project Description

Unique properties of lignite coal enable cost-effective methods of generating high-value solid carbon and mineral products. These properties include high organic association content of valuable mineral matter, including the REE essential in energy systems, defense technology and a number of consumer goods, and high organic acid content, enabling efficient generation of high-purity carbon products and fertilizers. These advantages, in addition to the vast lignite reserves available in North Dakota, enable unique opportunities for high-value carbon and mineral product production from lignite. The proposed technology is capable of making disruptive impacts in domestic and global REE, battery, and char material markets in large part to the synergistic process enhancements and substantial lignite reserves found in ND.

3.1 Overall Project Goal

The overall objective of this proposed work is to develop and demonstrate an economic and synergistic process for production of high-value solid products from North Dakota lignite coals, including recovery of rare earth elements (REE), production of high-quality, low-cost battery anode materials, and high-value char-related products from carbon residuals.

3.2 Project Objectives

This project aims to develop a synergic process that co-produces multiple high-value solid products from North Dakota lignite coals, including Rare Earth Elements (REE) and critical Materials (CM), advanced anode materials for Li-ion Batteries (LIBs), clean coal product, char, and fulvic acid. The specific objectives include: 1) Build on current efforts to produce a high-value ore containing >90% REEs, other critical elements, and impurities while producing a clean lignite by-product; 2) Utilize the clean lignite by-product as a feed stock for extracting humic acid (HA) from lignite; 3) Develop a hydrothermal carbonization

process to convert HA to carbon spheres (CS) for advanced anode materials for LIBs; 4) Investigate the feasibility of using HA as a precursor for graphene production to prepare low-cost high-performance anode materials for LIBs; 5) Determine the chemical and physical properties of the extracted product for use as a char or activated carbon; and 6) Evaluate the economic and commercial potential of the technology.

3.3 Scope of Work

Testing is aimed at optimizing the processes including REEs recovery, HA extraction, and AC/Char production based on-going work and developing new processes for fulvic acid recovery and anodes preparation. A major effort will be made to produce high value-added anode materials from HA for LIBs via optimizing the two proposed routes. Existing equipment will be operated in a batch mode. For the new processes such as anode materials preparation, laboratory-scale testing will be conducted at one gram per batch. For the established processes including REEs recovery, HA extraction, and AC production, the bench-scale testing has already been operated with >500-gram batches (~ 1 kg/hr coal throughput).

A   TASKS TO BE PERFORMED

Task 1.0 – Project Management and Planning: This task shall include all work elements required to maintain and revise the Project Management Plan, and to manage and report on activities in accordance with said plan. It shall also include the activities to ensure coordination of the project with DOE/NETL and other project participants. UND will be responsible for completion of this task.

Task 2.0 – Optimization of Ash Reduction and REE & CM Recovery: Primary objectives are generation of low-ash lignite, particularly in the organic fractions used for anode material production, and co-generation of high-grade REE and critical minerals.

Subtask 2.1: Optimization of Ash Removal Methods. Optimization of leaching conditions, particularly with reducing humic and organic ash components, will be developed and may include multi-stage leaching systems to maximize REE recovery potential. Use of mineral acids, organic extractants, and other additional ash-reduction mechanisms, particularly for removal of chelated metal ions, will be investigated.

Subtask 2.2: Determination of the Most Cost-Effective Process. Testing will focus on recovering a high-

grade REE and critical mineral ore. Optimization will focus on economic considerations, and may include processes such as solvent extraction, solid-phase ion exchange resins, and selective precipitations. Investigation into additional product streams, such as low-value ores are considered to bolster economics of the overall process and eliminate possible waste streams.

Subtask 2.3: Bench-Scale Processing at Optimized Conditions. A 50 kg sample for representative testing in subsequent tasks will be prepared using the optimized leaching conditions. This task leverages the unique bench-scale equipment constructed during previous REE extraction projects.

Task 3.0 – Extraction of Humic Acid from Low-ash Lignite: The goal is to extract high-purity HA from the low-ash lignite generated in Task 2 for anode preparation in Task 4, collect the low-ash humins for char production in Task 5, along with the recovery of fulvic acid as a high-value by-product. Due to the similarity of lignite and leonardite and our proprietary technology, the work on this task will be minimal. The primary objective is to maximize extraction HA yield and minimize ash content to <0.5%. As shown in the preliminary data, our purification method of treating with a lixiviating agent can achieve the target sufficiently, so we expect to obtain similar purity and low ash content at the pilot-scale by further optimizing process parameter such as solid/liquid ratio, extraction/treatment time, and agent concentrations. If needed but unlikely, we will use ion exchange resins to further remove trace metallic cations.

Task 4.0 – Development of Advanced Anode Materials from Humic Acid

Subtask 4.1 – Synthesis and Characterization of PCS. An optimal HTC process to produce mono-dispersed and micro-sized PCS using high purity of HA generated in Task 3 will be developed building upon proof-of-concept validation previously illustrated. Key PCS performance metrics - sphere yield, size, and sphere size distribution will be evaluated as a function of i) temperature (150-250 °C), ii) reaction time (1-24h), iii) solid/water ratio (1/20-1/5), iv) surfactant type and v) surfactant concentration (0-1%). The Taguchi orthogonal array will be used to design optimization experiments of these factors and investigate interactions between them. After the optimized HTC process is established, a major effort will investigate the feasibility of incorporating Li-alloy materials (such as Si and Sn) into the PCS. The goal is to produce

core(metal)-shell(carbon) structured composite PCS to further improve the overall battery performance. Metal precursor type and loading will also be optimized. SEM-EDX will be utilized to characterize the sphere size, sphere size distribution, and metal distribution in the PCS.

Subtask 4.2 – Optimization of the Graphitization Process of PCS. The graphitization process will be optimized to identify the best-performance PCS anode. Key performance metrics -graphitization degree, porosity, and spheritic morphology maintain will be evaluated as a function of temperature (700-1800°C), time (0.5-12h), atmosphere (vacuum, N_2 or Ar), and chemical activation agent type (KOH and LiOH) and loading using the experimental design method applied in Subtask 4.1. Sphere size, pore microstructures, and metal distribution will be characterized by SEM-EDX, the graphitization degree will be measured by Raman spectrometer, porosity will be tested by a BET surface area analyzer and tapping density and pressing density will be measured following the relevant ASTM approaches B527 and B962, respectively.

Subtask 4.3 – Synthesis and Characterization of pSi. The MR reaction of silica will be systematically investigated to optimize silica particle size (400–2000 nm), ramp rate (~5°C/min), reaction temperature (500°–800°C) and reaction time (0.5–12 h). Three critical indexes, surface area, porosity, and yield will evaluate the reduction reaction. Experimental design methods from Subtask 5.1 and 5.2 will be used.

Subtask 4.4. Synthesis and Characterization of the pSi/G Anodes. **Route B** will be developed with the focus on: particle size of pSi source, feeding ratio of HA to Si, and reaction temperature and time. Feeding ratio is crucial: too much HA may block the pores while too little will lead to insufficient accommodation to the volume change of Si. The surfactant-assisted dispersion method will be applied to overcome a potential challenge of homogenizing the precursor mixture of porous silicon and HA. The impact of surfactant concentration, dispersion method, and drying approach on the homogeneity of the precursor mixture will be investigated. Characterization methods used in Subtask 4.1, 4.2 and 4.3 will be used. Conversion of HA to graphene will be confirmed by a Raman spectrometer; carbon content will be tested by a carbon analyzer.

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Subtask 4.5. Electrochemical Performance Testing of the Anodes. The electrochemical performance of the two advanced anodes PCS and pSi/G, including 1st-cycle reversible capacity, 1st-cycle efficiency, and cycling life, will be compared with two commercial anodes, MCMB and S450-2A. The electrochemical performance testing will be conducted on a Neware CT-3008 battery testing system available at Clean Republic. CR2032 coin-type cells will be prepared with equipment currently available. Current densities and specific capacities will be calculated based on the mass of the target compound of the electrode. Cycle life will be evaluated using a three-electrode system (ECC-REF, EL-Cell GmbH, Germany) for cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS).

Task 5.0 – Combustion and Carbonization Properties of Low-ash Lignite (Humins): Major objectives are to determine potential uses for the low ash humin materials. Primary uses include direct combustion and carbonization to produce char and activated carbon.

Subtask 5.1. Combustion -- Stability and Fireside Ash Behavior: Ignition Temperature: Thermo-gravimetric/differential thermal analysis (TGA/DTA) will be performed on selected low-ash Humins to identify ignition temperature using a procedure adapted from Sato and others.[1] The sample is heated at 20°C per minute to 900°C in dry air. TGA provides the mass loss as a function of temperature and DTA provides an indication of heat flow into and out of the sample. The ignition temperature occurs at the onset of the major exothermic transition. Self-Heating: Self-heating can occur as a result of heat generated by the adsorption of atmospheric moisture and can be a concern. Self-heating increases the stockpile temperature to that at which oxidation reactions could take over. To address this concern, simultaneous differential scanning calorimetric/thermal gravimetric analyses (DSC/TGA) will be performed using procedures by Clemens and Matheson.[2] Three responses will be measured for each sample. Fireside Performance: Mineral and ash behavior will be predicted using computer-controlled scanning electron microscope (CCSEM) analysis to determine size, composition, and abundance of mineral grains. The

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[1] Sato, et al., (2007).
[2] Clemens, et al, (1996).

CCSEM is fully automated, allowing up to three thousand individual particles to be characterized in a single sample. MTI will use CCSEM data, along other benchmarking data, to calculate advanced performance indices. MTI has an extensive database on fuel properties and performance on over 10,000 samples. Indices relating to cyclone slag flow, wall slagging, convective pass fouling, abrasion and erosion wear, strength development of deposits, ash resistivity, and cohesivity of ash will be calculated.

Subtask 5.2. Carbonization Testing to Produce Char and AC: Selected samples of parent lignite and the low-ash humin will be carbonized or pyrolyzed in a 2-inch fluidized-bed reactor to produce char and activated carbon. The chars will be produced at temperatures of 400 to 500 °C in an inert atmosphere. Activated carbon will be produced from the chars at temperatures between 775 to 900 °C in nitrogen and steam atmosphere. The chars and the activated carbon materials will be analyzed to determine surface area using the iodine number method. The char will be analyzed to determine if it has properties for metallurgical applications that include moisture (<12%), ash content (<15%), sulfur content (<1.5%), volatile matter (<2%), mechanical strength (6.9-30 MPa), reactivity index (2.6 – 28% (ASTM test method D-5341)). Microscopic morphological analysis of the char will be performed to determine the swelling and agglomeration behavior of the char. Parametric testing will be performed to determine optimum conditions for both char and AC production.

Task 6.0 – Techno-Economic Analysis of Process: Barr Engineering will be contracted to perform a TEA on the proposed process. The TEA will include a market analysis and commercialization pathway.

3.4 Anticipated Results

The anticipated results of the proposed project are summarized as follows:

- Successful development and demonstration of an integrated overall process including all 3 major processing/product streams (REE, battery anode materials, char/carbons), including processing > 50 kg of coal feedstock for the process.

- Preliminary optimization for total process improvements and economics to reduce ash yield,

maximize battery anode performance, and develop high-value char products.

- Determination of optimal char product use(s) from the as-measured properties following optimal pre-processing conditions for maximal product value.

- Completion of a technical and economic feasibility study that will include detailed process flow diagrams, equipment specifications, mass and energy balances, and economic metric for the technology.

- Assurance of suitable intermediate product (REE and CM concentrate, battery materials) quality with downstream requirements through testing and analysis as needed.

- This project will develop a high performance and economically viable process for generation of a number of high-value carbon and mineral products from the vast lignite reserves in North Dakota. Successful demonstration will develop new avenues for increased lignite use in non-power generation sectors, permitting suitable product diversification.

3.5 Facilities and Resources

UND has exceptional existing analytical, lab-, and bench-scale equipment including SEM-EDS, XRF, XRD, and electrochemical characterization instruments. UND and MTI laboratories are equipped to perform chemical extraction and processing steps and have an atmosphere-controlled glove box for battery construction and controlled furnaces for carbonization steps. Their 10-20 kg/hr bench-scale system constructed for a related REE extraction project will be usable for the REE and HA extractions to be performed in the project. MTI has developed multiple proprietary algorithms for prediction of various performance indicators based on the abundance and association of coal impurities. These algorithms build on MTI's large fuel CCSEM database, technical expertise, and findings from field-testing at mines and plants.

3.6 Environmental and Economic Impacts during Project

The environmental impacts resulting from performance of the proposed work are negligible. Waste streams produced as a part of testing will be disposed of via the existing waste disposal mechanisms available at UND, and any hazardous waste (if produced) will be handled according to UND regulations. Economic impacts include employment opportunities for UND research staff, students and support staff. This project will train the next generation of engineers/scientists that will benefit the North Dakota labor force.

3.7 Technical and Economic Impacts of Proposed Technology

Our technology uses domestic U.S. coals to coproduce multiple high-value solid products. We believe this project will advance the technology of low-rank coal utilization that maximizes the coal value and significantly reduce environmental impacts. The primary outcomes are the multiple high-value solid products and the associated production processes. The proposed project will have the following impacts: 1) it will significantly reduce the overall production cost of high-value products, and maximize the potential low-rank coal value, while reducing environmental impacts by limiting waste streams; 2) it will greatly advance all the three sub-processes that this proposed process is built on towards commercialization: REE extraction from North Dakota lignite coals, Valley City State University steam plant upgrade coupling activated carbon production with steam production, and NDIC/Clean Republic sponsored projects to develop advanced electrode materials for LIBs.

3.8 Project Need

With increasing environmental regulations on fossil-fuel based power and decreasing coal production nationwide, new opportunities for marketable use of lignite coal are required to maintain the existing mining/lignite use infrastructure in the State. The recovery of REEs from ND lignite and related materials has potential to be a significant new industry for the state that will both maintain existing jobs and create new jobs and revenue for the State. Additionally, development of low-cost and high-performance anode materials for Li-ion batteries will significantly improve current battery technology, enabling deployment at

larger scales and in more varied applications, while mitigating a number of safety and reliability issues associated under harsh conditions. Further, generation of high-value coal char-related materials from low-rank coals, particularly lignite, offer substantial benefits to potential resource utilization, particularly in high-capacity markets such as metallurgical coke and activated carbon.

4. STANDARDS OF SUCCESS

The standards of success for the outcomes of the proposed work are summarized as follows:

- Successful development and demonstration of an integrated process for economic production of multiple solid products from coal, including high performance and low cost battery anode materials, REE and CM concentrates, and improved fuel and char properties.

- Preliminary optimization of REE extraction and concentration processes to generate a >90 wt% REE and CM concentrate, with resultant coal ash and humic ash below 5% and 1%, respectively.

- Evaluation of two battery anode material development pathways from coal-derived humic acids, with materials tested to ensure high-quality (>25% improvement over current use materials) and low cost (>20% price reduction) battery anode materials.

- Analysis of residual carbon fractions to evaluate optimal use as a char/fuel product, including determination of activated carbon performance using industrially standardized procedures, and potential metallurgical coke performance.

- Development of a commercialization strategy, including a fully functional economic and financial model for evaluation of economic viability and commercial prospects.

5. BACKGROUND INFORMATION

UND has demonstrated the ability from previous projects to cost-effectively generate REE/CM and anode material products from North Dakota coals, including data below, showing promise for an economical and market-disruptive process.

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5.1 Definition of the Research Problem/Opportunity

Coal has long been a reliable baseload energy source for the United State, but environmental and market forces have reduced coal production for power generation. This transition is forcing states like North Dakota, the host to the world's largest lignite deposit, to seek alternative opportunities to sustain its coal-dependent economic development. Fortunately, a series of recent studies performed at UND has demonstrated that the unique properties of low-rank coals provide an opportunity to extract weakly bonded REE, critical elements, and inorganic impurities from the organic component producing a clean lignite product. The clean lignite product is an excellent feedstock for producing a high-quality HA fraction used to produce carbon-based anode materials for LIBs. The residual material can be used as a high value fuel or for the production char or activated carbon.

5.1.1 Background—REEs Recovery from Lignite

Due to the reliance on imports of CM needed for national energy security and defense, an executive order[3] was signed pursuant to generating a domestic supply of these critical minerals. [4]The REE typically include the lanthanide series (La-Lu), along with scandium and yttrium. Applications for these critical minerals include advanced battery and electronics technologies, catalysts for numerous industries, alloying materials for advanced materials, high power permanent magnets, and others.[5] Further, these REE have little-to-no effective replacements for their unique chemical properties, with market growth expected to significantly rise.[6] Additionally, the current supply of the most critical of these REE is nearly fully produced from a single resource (ion-adsorbed clays) and is projected to only last another 10 to 20 years.[7] Very few concentrated REE deposits exist, requiring large concentrating factors from elevated feedstocks. Through related DOE projects, coal and coal byproducts were found to be a promising domestic source of REE. In

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[3] Exec. Order No. 13817 (2017)
[4] Department of Interior (2018).
[5] NETL (2019).
[6] Department of Interior (2019).
[7] Yang, et al., (2013).

lignite, the REE are associated with the organic matrix of the coal in the form of coordination complexes which can be economically extracted using mild acids.[8]

5.1.2 Background—Carbon Materials Dominates the Commercial LIB Anodes

Graphite is the state-of-the-art anode for LIBs, but its poor low temperature performance, rate capability, and limited theoretical capacity (372 mAh/g) has motivated the development of next-generation anodes. In spite of their high specific capacity, however, these new materials bring many other technical problems including huge volume expansion, low ionic/electronic conductivities, and/or side reactions with electrolyte, leading to unsatisfactory battery performance such as low 1st-cycle efficiency, large irreversible capacity C_{irre}, poor cycle life, poor rate capability and low energy density (**Table 1**).[9] The most effective solutions to those problems include designing effective morphologies (*e.g.,* porous and spheritic

Table 1. Commercial Anode Materials for LIBs.

Materials type	Anode materials	C_{theo} mAh/g	Ø 1st %	Lithiation V	Pressing density	Cycle life	Rate Capability	Cost	Common Issues	Market share %
Insertion	Natural graphite	340-370	90~93	0.2	1.6~1.85	>1000	Fair	*	Poor low temperature performance	25
	Artificial graphite	310-370	90~96	0.2	1.5~1.8	>1500	Good	**	Poor low temperature performance	65
	MCMB	280-340	90~94	0.2	1.5~1.7	>1000	Very good	***	Low specific capacity	3
	Hard carbons	250-600	80-85	0.52	1.3~1.5	>1000	Very good	***	High C_{irre}, immature technology	1
	Soft carbons	250-1000	80-85	0.52	1.3~1.5	>1000	Very good	***		1
	CNTs	1116	30~60	>0.5	--	<100	Poor	*****	High C_{irre}, high voltage hysteresis, poor cycle life	N/A*
	Graphene	1157	30-60	>0.5	--	<50	Poor	*****		N/A*
	LiTi$_4$O$_5$	175	98~99	1.55	1.8~2.3	>30000	Excellent	***	Very low energy density	4
Alloying	Si/SiO	1600~4200	~60	0.3~0.5	0.9~1.6	<300	Poor	****	Huge volume expansion	<1
	Sn/SnO$_2$	790~993	~60	0.4~0.69	--	<200	Poor	****	Huge volume expansion	N/A*

C_{theo} = theoretical capacity, C_{irre} = irreversible capacity, **Ø 1st =** first cycle efficiency, MCMB = Mesophase Carbon Micro Beads, and CNTs = carbon nanotubes. "N/A*" means not commercialized as a pure anode.

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[8] Laudal, et al., (2018).
[9] Zhang, et al., (2017).

structure), [10] creating hierarchical micro-/nano-structures [11] and fabricating conductive carbon composite/coating.[12] Apparently, carbon-based materials are not only dominating the current anode market but also continue to play critical roles in the next-generation anodes. From the perspective of lowing the cost of LIBs, as an abundant and cheap carbon source, coal or coal byproducts have been extensively used as the feedstock for anode materials such as artificial graphite, turbostratic carbons, and nanostructured carbon materials.[13] Our recent research also found that coal-derived humic acid is an excellent graphene precursor for high-performance electrode materials for LIBs.[14]

5.1.3 Background— Char and AC Production from Lignite

Traditionally, carbonization or pyrolysis has been used to produce gases, tars, and char. Current technology can produce a wide range of products from lignite that include high value carbon materials such as activated carbons. Lignite coal because of the abundance of oxygen functional groups and associated alkali and alkaline earth elements have limited their ability to produce pitch for fibers and chars for metallurgical applications (steel making). [15] Lignite coals are considered a non-caking coal and do exhibit any swelling behavior and are not considered a good feedstock for the production of char for metallurgical applications. However, past work [16] found that the char produced during pyrolysis in a nitrogen atmosphere from a demineralizing ND lignite exhibited extensive swelling and agglomeration behavior. The untreated ND lignite did not show any swelling or agglomeration. This work indicates the potential to change the properties of the char by removing the organically associated inorganic elements.

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[10] Entwistle, et al., (2018), Shen, et al., (2016), Xin, et al., (2012).
[11] Luo, et al., (2015), Zamfir, et al., (2013), Zhang, et al., (2004).
[12] Kucinskis, et al., (2013), Luo, et al., (2015).
[13] Guo, et al., (2017), Hoang, et al., (2018), Wang, et al., (2014), Xing, et al., (2018), Zheng, et al., (1996).
[14] Baker, et al., (2018).
[15] Laudal, et al., (2017).
[16] Kleesattel, et al., (1987), McCollor, et al., (May 1988).

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5.2 Proposed Technology

Figure 1 illustrates the basic interconnectivity of the key processes producing the multiple value-added solid products: REE, high value fuel, char or activated carbon, fulvic acid, and advanced anode materials. Briefly, the synergic process includes: 1) the extraction and recovery of REE and other critical elements by acid leaching lignite, leaving a low-ash lignite for production of high-purity HA, and char or metallurgical coke; 2) removing HA from the low-ash lignite by base extraction leaves a low-ash, low volatiles and high fixed carbon product (humins) as an enhanced feedstock for char that can be used for metallurgical or activated carbon; 3) the low-ash HA is an excellent building block to produce porous carbon

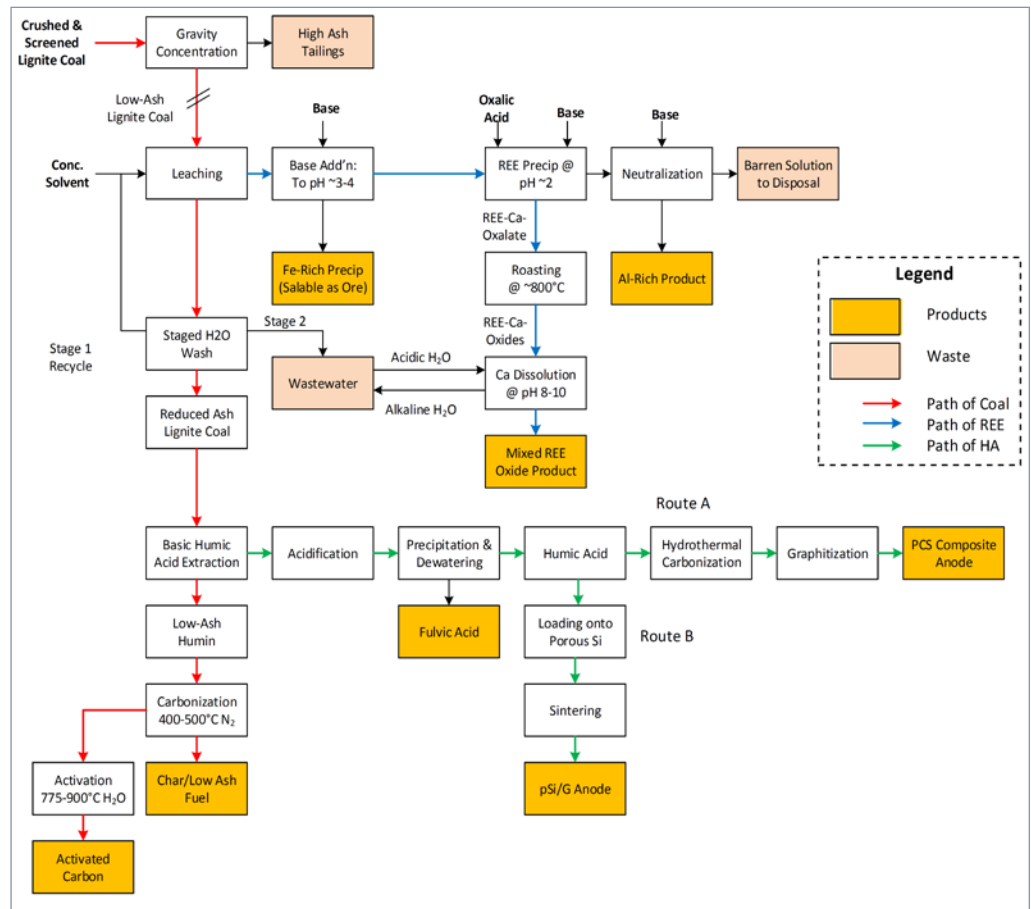**Figure 1**. The proposed technology for producing multiple high-value solid products from lignite. PCS = Porous Carbon Spheres, and Si/G = silicon/graphene.

spheres (PCS) via hydrothermal carbonization technology; 4) HA is also a precursor for graphene production to potentially produce high-performance silicon/graphene anodes; and 5) fulvic acid is recovered as a byproduct by acidifying the basic humus solution to precipitate the HA.

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Recovery of REEs from Lignite: The first step produces a reduced ash lignite for subsequent production of high-value carbon products while recovering REE and critical material bearing ore as a side-product. In particular, certain deleterious elements such as Fe must be removed for optimal performance of anode materials while removal of alkali and alkaline earths increase the value of the residual char. Technical evaluation criteria include: i) production of a carbon product of <5% ash; ii) extracted humic components with an ash content of <1%; iii) Removal of >90% of deleterious elements to subsequent processes, and iv) Generation of high grade (>90%) REE and critical material streams for improved overall plant economics. Using the currently optimized process for REE recovery, physical concentration and ash removal is first conducted utilizing proven coal cleaning techniques, reducing ash constituents in high REE-bearing lignite by as much as 60% (from 40% ash to 15%), while concentrating REE due to their significant organic association as demonstrated in **Figure 2**. Acidic leaching using a dilute mineral acid follows with large portions of the residual metallic ash (non-silicates) being mobilized into a pregnant leach solution (PLS). A large portion of the metals liberated in this process are present in organic forms (the humic and fulvic acid fractions) indicated through rapid kinetics and measured thermal behaviors, with proven metal
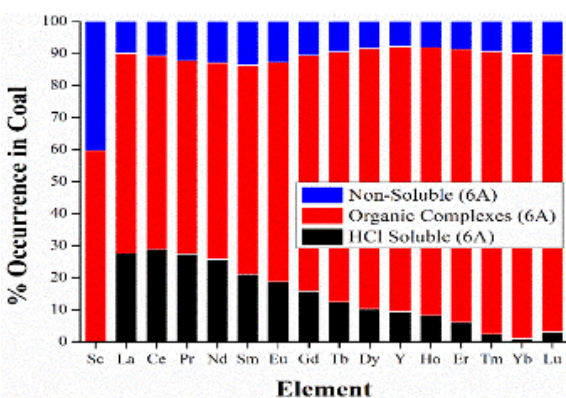
Figure 2. Modes of occurrence of REE in lignite

chelation properties produce a low organic ash composition after extraction.[17] This organic ash has been measured as low as 0.47% for lignites tested, with total coal ash reduced below 10% under relevant leaching conditions for REE recovery. The PLS is neutralized to pH 3-4 to precipitate impurities followed by selective precipitation of the REE using oxalic acid.

The REE-barren solution is neutralized with a precipitate containing a large amount of Al and Mg produced. Further REE purification is conducted from the oxalate precipitate through calcination to an oxide, and selective dissolution of Ca. A >80% REE concentrated ore has been produced.

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[17] Spark, et al., (1997).

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This process is currently optimized for economic and optimal recovery of the REE and CM, not for coal and organic ash reductions. Adjustments to this process, including but not limited to higher acid concentration leaching, multi-stage leaching, and use of additives and metal complexants to bind inorganic components of the coal will be investigated. Further optimization of the REE and critical mineral recovery process, such as additional selective precipitations or solvent extraction may be required to economically concentrate and recover these minerals.

Development Advanced Anode Materials from HA: The low ash, high HA content in the treated lignite provides an opportunity to develop low-cost and high-performance carbon-based composite anodes for LIBs. The ultimate outcome is market-ready high-rank anodes with competitive performance-price performance. Therefore, two commercial anodes MCMB (pure carbon) and S450-2A (blended Si-C and graphite),[18] which are the high-end commercial anodes in each category are used as a reference to set the benchmark battery performance: 1) 1st-cycle reversible capacity 450 mAh/g for pure carbon anodes (MCMB 280-340 mAh/g), and 800 mAh/g for carbon composite anodes (S450-2A 450 mAh/g), 2) 1st-cycle Coulombic efficiency >80%, 3) Cycling life with 80% of capacity retention >1000 for pure carbon anodes (MCMB >1000), and >300 for composite anode (vs S450-2A: 200 cycles); 4)Pressing density >1.4 g/cm^3 for pure carbon anodes (MCMB 1.3-1.5 g/cm^3) and >1.7 g/cm^3 for composite anodes (S450-2A: 1.65-1.75 g/cm^3); and 5) Cost lowered by 20%: pure carbon anodes $15/kg (MCMB ~$18/kg) and composite anodes $20/kg (S450-2A: ~$25).

Two routes are proposed to prepare advanced carbon composite anodes, leveraging the unique properties of lignite-derived HA (**Figure 1**). In **Route A,** lignite-derived HA is directly converted to porous carbon spheres (PCS) by the hydrothermal carbonization (HTC) process at ~200°C and self-generated pressure. Soft-templates (*i.e.*, surfactants) or metal precursors (*e.g.,* Sn^{2+} and Si) can be incorporated into the HTC process to tune the morphology of spheres or form carbon/metal composites, respectively. Low-temperature graphitization (<1800°C) of the PCS or its composites will produce the final anodes. The

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[18] BTR Ltd., (2019).

feasibility of a chemical-activation step during the graphitization will be also investigated. In **Route B**, the commercially available porous silicon (pSi) particles are homogenized in an ammonia solution of HA with the assistance of surfactants to obtain HA-coated pSi particles. Sintering the dried HA-coated pSi particles at 900 °C under inert atmosphere will convert the HA to graphene matrix and yield the final pSi/G composite anodes. Alternatively, the pSi can be prepared via magnesiothermic reduction (MR) of commercially available silica particles: $SiO_2 + 2Mg \rightarrow Si + 2MgO$, followed by the removal of MgO with HCl solution.[19] The key components of our approach are:

High-purity HA: High purity of HA is crucial as ash and metal impurities, which inevitably exist in the conventional HA extracted directly from coal, will deteriorate the battery performance of LIBs. Our experience in making electrode materials for LIBs established a targeted ash content of <0.5%. We have developed a proprietary procedure for extracting nearly ash-free HA from leonardite in a relevant project, aiming to synthesize graphene-modified cathode materials. Results show 1) HA extraction yield of 50%-60% with ash content only 0.12%, and 2) specific capacity increased by 15% using graphene produced from HA. This HA extraction procedure has been tested at pilot-scale to producing one kilogram of HA, taking advantage of the established pilot-scale tank reactor used in the REE project. The yield of HA is similar to the small scale, with an ash-content of around 0.47%.

Hydrothermal Carbonization: HTC is the most promising technology for fabrication of CS due to its simplicity, low-temperature (~200C°) operation, tunable size, morphology and porosity, easy to form composites, and post-functionalizable surface chemistry, especially being applicable to a broad of low-cost carbon feedstock. [20] Humic substances, the major organic component of coal, can be divided into three fractions: HA (soluble at high pH), fulvic acid (soluble at all pH) and humin (insoluble at all pH). HA typically contains a variety of components linked together, including quinone, phenol, catechol, and carbohydrate moieties. [21] Lignite or naturally oxidized lignite (leonardite) is the most abundant and

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[19] Entwistle, et al., (2018).
[20] Berge, et al., (2011), Titirici, et al., (2012).
[21] Stevenson, (1994).

commercialized source of HA. The chemical structure of HA in lignite closely resembles lignocellulose in many ways: hydrophobicity, rich aromatic subunits, abundant phenolic hydroxyl groups, and solubility. Therefore, we hypothesize HTC of HA will resemble that of lignocellulose. The carbohydrate fraction of HA will participate the classic HTC process, whereas the other components will either co-react (*e.g.*, amino acids), be hydrolyzed into small organic side-products, or remain unaffected at large (*e.g.*, aromatic units and inorganics). The overall outcome is a micro-sized spherical carbon material featured with reduced oxygen content and functional groups, increased hydrophobicity, and highly cross-linked aromatic core. Our preliminary results (Section 2.3.1) support our hypothesis.

Micrometer-sized pSi: In **Route B,** we select micro-sized pSi instead of Si nanoparticles as pSi has a much higher critical particle size (~1.5 µm) for fracture during the charging/discharging of LIBs than nanoporous particles (~150 nm).[22] Our approach takes advantage of this feature avoiding problems caused by nanomaterials while retaining advantages in cycling life and rate capability. The microparticles also have a higher pressing density, and thus higher volumetric energy density, and more importantly, remarkably lower production cost than nanoparticles.

Synthesis of pSi: In this project we will start with commercially available pSi. Long-term we prefer to synthesize pSi to control the microstructure and morphology by design and more importantly, to further reduce the cost. Current synthesis usually involves significant amounts of toxic chemicals (e.g., HF acid), expensive precursors, and very sophisticated reaction control. The magnesiothermic reduction has emerged as an effective way to convert silica to silicon with varying degrees of structural and morphological control. [23]The features of low cost, a single-step reaction, and mild conditions make this approach highly scalable. The main challenge is to precisely control the extent of reduction to fully reduce silica and avoid over-reduction to form Mg_2Si [24] and will be addressed.

Low-temperature Graphitization: For **Route A**, the main purpose of using low-temperature graphitization

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[22] Shen, et al., (2016).
[23] Bao, et al., (2007), Entwistle, et al., (2018).
[24] Bao, et al., (2007).

is to partially retain the turbostratic carbon microstructures to increase the specific capacity of the anode. The secondary purpose is to facilitate the chemical activation (e.g. KOH and LiOH) to introduce more pores to the CS by HTC, well-known to have limited porosity and surface area. We will also investigate the feasibility of using a weak base, LiOH, as the activation agent. The residual lithium can function as a "pre-lithiation" agent, improving the 1st-cycle columbic efficiency. For **Route B**, this graphitization process converts the thin layer of HA coated on the surface of pSi particles to graphene. This conversation has been proven at relatively low temperature in literature as well as our preliminary work. [25]

Combustion and Carbonization Properties of Low-ash Lignite (Humin): The residual carbon fraction will be a low-ash, high fixed carbon, high heating value material providing multiple market opportunities including use as a premium combustion fuel as a powder or a briquette, to produce a high-value char or activated carbon.[26] The fireside performance of the low ash lignite is expected to be significantly improved and will be examined through detailed analysis of the impurities in the fuel and an assessment of the potential to form wall slag, convective pass deposits, and fine particulate.[27] A decrease in reactivity is expected impacting its ignition temperature, and spontaneous heating may become an issue. These will be addressed in laboratory testing. The use of the material to produce a char or activated carbon will also be examined following industry proven methods currently used by activated carbon manufacturers such as Calgon and Norit and the use of the char for briquettes (Royal Oak). In addition, pyrolyzed char will analyzed to determine if it has properties for metallurgical applications.

5.3 Preliminary Laboratory Scale Data

REEs Recovery from Lignite. The proposed process for REE has been tested extensively under optimizing parameters for REE recovery and concentration, and has been successful in producing a refiner-feed concentrates of >80% REE on a cation basis, of which easily separable and non-toxic materials comprised the residuals. Further, ash reductions from 40 wt% to <10 wt % have been achieved through the process

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[25] Xing, et al., (2017).
[26] Schobert, (1995).
[27] Benson, et al., (2015).

under various conditions, along with organic ash components of <0.5%. Acid impregnation into the coal matrix was not observed during all acid leaching tests, and is not anticipated to be a challenge.

Advanced Anode Materials from Lignite-Derived HA

a) *via Route A:* our preliminary results indicate: 1) HTC can convert HA to nearly mono-dispersed micro-sized carbon particles (**Figure 3b**), 2) the surfactant can assist the formation of perfect spheres (**3c**), 3) the morphology of the
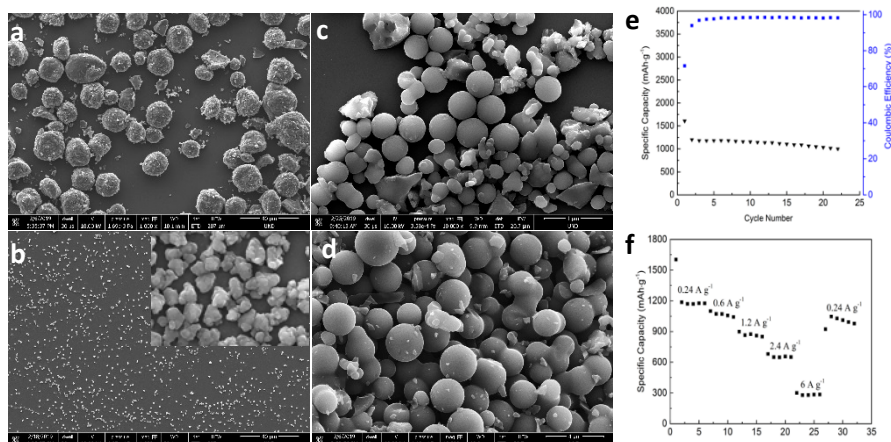

Figure 3. SEM images of MCMB (a), PCS (b), PCS with surfactant before (c) and after (d) sintering at 700°C, specific capacity (e) and rate capability (f).

spheres maintains during the thermal process (**3d**), 4) the 1st-cycle capacity over 1000 mAh/g (**3e**) is much higher than MCMB, and 5) the 1st-cycle efficiency 50% need be improved by increasing the sintering temperature.

b) via Route B: The pSi/G anode is synthesized via a procedure similar to **Route B,** validating the proof of concept with the only difference being the use of SiO instead of porous Si. The XRD pattern in **Figure 4a)** confirmed the co-occurrence of the disproportion reaction ($2SiO \rightarrow Si + SiO_2$) and the conversion of HA to graphene. The SEM image **4b)** clearly shows Si
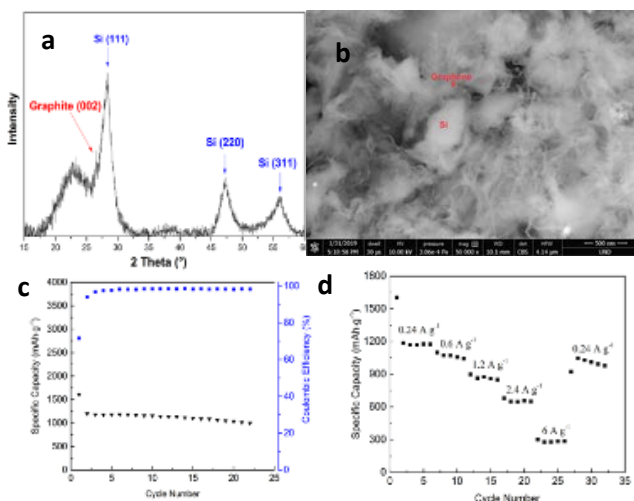
Figure 4. XRD profile (a), SEM image (b), specific capacity (c), and rate capability (d) of the pSi/G anodes.

embedded in a network of wrinkled graphene sheets. The electrochemical performance is satisfactory for an unoptimized procedure: 1st-cycle reversible specific capacity of 1600 mAh/g, 1st-cycle efficiency of 73%, capacity not fading significantly for 22 cycles, and good rate capability. Since the proposed **Route B** is even simpler (no disproportion reaction and no need for removal of SiO_2 by hydronic acid), we strongly

believe it is feasible.

Literature Review

Porous Carbon Sphere is a Highly Desired Anode. PCS has many desirable properties such as high specific capacity and good rate capability due to their structure characteristics, including: 1) short Li diffusion paths; 2) tunable porosity and high surface area to facilitate rapid charge transfer and minimize polarization effects; 3) tunable microstructure, crystallinity, morphology, and composition for high specific capacities.[28] Compared with the spheritic MCMB, the commercial anode with the best rate capability, PCS can have higher capacity due to the porous structures. Studies have shown PCS prepared by HTC can easily incorporate other high capacity anodes (*e.g.,* metal oxides and alloys) to prepare CS-based composites improve performance. [29] However, most carbon sphere anodes prepared by HTC are using simple carbohydrates (*e.g.,* glucose) as the carbon precursor. CS anodes by HTC of natural resources (*e.g.,* real biomass, coal) and their extractives (*e.g.,* HA) are more challenging but much more practical towards commercialization of this technology.

Current Progress on Si/G Anode Materials. Si/G, taking advantages of the high capacity of silicon and superior conductivity of graphene is among the most promising next-generation anode.[30] A recent review summarized the current progress of Si/G anodes as following:[31] 1) most of reports are using Si nanoparticles instead of pSi, 2) graphene is exclusively prepared via the Hummer's method from graphite,[32] 3) no single example shows satisfactory battery performance in all aspects, and 4) the high cost of Si nanoparticles and graphene is cost prohibitive for commercialization. Recent research shows that HA can be made into graphene-like materials with quality comparable to reduced graphite oxide,[33] though graphene prepared by this approach cannot be directly applied in electrode materials due to the ash content and metallic impurities. Our synthesis technology, combing in-situ synthesis graphene from high purity HA with the micro-sized

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[28] Roberts, et al., (2014).
[29] Demir-Cakan, et al., (2008), Roberts, et al., (2014), Yang, et al., (2013).
[30] Lee, et al., (2010), Xin, et al., (2012).
[31] Cen, et al., (2018).
[32] Hummers and Offeman, (1958).
[33] Beall, et al., (2014), Powell and Beall, (2015), Xing, et al., (2017).

pSi is a facile and low-cost way to synthesize Si/G anodes as supported by our preliminary results.

5.4 Anticipated Technical and Economic Benefits over Current State-of-the-Art

- Co-production of multiple high-value solids from ND lignite through process integration will significantly reduce the overall production cost and maximize the low-rank coal value

- The upgraded process that integrates production of high-quality carbonaceous feedstock into the REE recovery dramatically advances the commercialization potential of REE recovery from coals.

- Combination of in-situ synthesis of graphene from HA and micro-sized pSi has remarkable technical and economic advantage over the current technology of making the Si/G anodes.

- Exploring the fundamental understanding of HTC will broaden its applications besides LIBs, such as catalysis, water purification, energy storage, CO_2 sequestering, bioimaging and drug delivery.

- The capability of supplying low-cost HA as a graphene precursor will accelerate the commercialization of our existing project aimed at production of HA-derived graphene modified cathode materials for LIBs.

- The char production using the higher quality feedstock at a lower cost will enhance the quality of the char products while also significantly increasing the profit of the new technology.

5.5 Comparative Technical, Economic, and Market Discussion

A previous TEA (REE Phase I) indicated that our REE technology is economically feasible, but stand-alone technology (coal by-products not used) would not become profitable until a scale of 50 ton/hr. On the contrary, the REE recovery with AC and steam production configuration would become profitable (the net revenue per dry ton lignite is $620), even at a small scale of 2.5 ton/hr. Therefore, monetization of multiple products is critical. Our technology that coproduces multiple products should be more profitable. The main revenue gain will come from the high-value anode materials with projected selling price ($21,000/ton). **Table 2** estimated the base case economic metrics

Table 2. The Total Value of the Multiple Solid Products Produced by One Ton of Lignite Coal.

Solid Products	Quantity (kg)[a]	Market Price ($/kg)[b]	Value ($)
REE/CM	0.96 (90 wt% oxide basis)	262	251
Char	224	1.4	313
Anodes (PCS)	127	21	2667
Fulvic Acid	35	3	105
Total			3336

[a] Calculated by the estimated overall yield.

[b] Estimated according to the selling price of a similar product.

of the proposed technology. More details are required in the TEA, but we expect the net value will be

substantially higher than the $620/ton. In addition, reported profit margins for anode materials are about

30%, based on that the net value from the anode development alone will be $800 per ton of feedstock.

This proposed technology is based on several relevant projects which have made substantial progress

towards the commercialization. The REE extraction project is close to the end of the Phase II goal of pilot-

scale demonstration. Valley City State University (VCSU) steam plant upgrade coupling AC production

with steam production is issuing revenue bonds for the construction of the plant. Clean Republic's plan to

open a new factory to develop advanced cathode materials for LIBs is in process. The global market

potential of the solid products is listed in Table 3. The market volume size and market value by 2020 are based on the predicted trend by recent market reports.[34, 35, 36, 37]

Table 3. The Market potential of the Multiple Solid Products by 2020

Solid Products	Potential Market	Market Volume (Kilo-ton)	Annual Growth (%)	Market Value ($Billion)	Coal Utilized (Million ton)
REE/Critical Elements	Electronics, catalysis, alloys, magnets	130	3.75[1]	33	133
Char	Metallurgical fuel, barbecue, industrial, filtration,	10,000	2	$5.8	100
Activated Carbon	Water purification, Mercury capture	2,100	14.7	$3.0	21
Anodes	LIBs	200	15	4	1.6
Fulvic Acid	Bio-fertilizer, health supplements, cosmetic	760	4.45	0.23	22

6. QUALIFICATIONS

UND Key Personnel: Dr. Michael Mann, PI, Chester Fritz Distinguished Professor of Chemical

Engineering, and Executive Director of IES has been working in the energy field since 1981. He has over

215 publications and over 35 years of continuous funding covering a wide range of conventional and

renewable technologies. He was a key member of the UND team led to the commercial demonstration

[34] Department of Interior, (2019).
[35] P&S Intelligence, (2019).
[36] Market Research Store, (2017).
[37] Albemarle Corp., (2016).

of the combined AC/steam production as VCSU and ist the PI on UND's REE project. Dr. Xiaodong Hou, Research Assistant Professor, is a material chemist with over 15 years' experience in synthesis and characterization of advanced functional materials. He has over 30 peer-reviewed publications and five patents. He is leading multiple projects directly related to the electrode materials for LIBs. Mr. Nolan Theaker, Research Engineer, is co-PI and the technical lead on UND's Phase II REE extraction and concentration project and has expertise in multi-phase reaction and material systems, hydrometallurgical processing and separations, and electrochemical reaction development.

MTI Key Personnel: Dr. Steven Benson, president of MTI, co-PI, has 40+ years of experience in the lignite industry and has worked extensively with coal beneficiation, combustion, gasification and air pollution control technologies. He is a world class expert on the inorganic/organic geochemistry of lignite coals, has recently led the project on AC production coupling with steam production at VCSU, and involved in multiple efforts related to REE recovery including partnership with UND on two DOE-funded efforts,

Clean Republic Key Personnel: Dr. Yong Hou, co-founder and VP for Research has 20+ years of experience in the lithium battery industry and has developed 14+ cells and battery pack products that are successfully marketed. He has been concentrating on the development of electrode material since 2014 and Clean Republic is in the process of commercializing a technology spin off from the collaboration with UND IES. He is leading several other projects related to energy storage application of LIBs.

7. VALUE TO NORTH DAKOTA

North Dakota produces over 30 million tons of lignite annually. The state's economy is heavily invested in the production and use of lignite. Successful completion of the proposed project will open new high value commercial opportunities for lignite use. New industries will be realized if successful commercialization of the technology is achieved, providing new opportunities for high-paying jobs and new tax revenues for the State. Although the proposed project is focused on extraction and concentration of REEs from lignite, additional processing steps will be required to arrive at final REE products. Having

a lowest cost source of REE concentrate in the Nation will encourage location of these processors/refiners in North Dakota, further increasing employment and revenue opportunities. Additionally, development of battery manufacturing in the state utilizing North Dakota based feedstocks and processes allow for additional potential of new industrial growth. Finally, production of exportable carbon products from lignite coal enables increases in mining, processing, and shipping industries for delivery to consumers.

8. MANAGEMENT

The team assembled to perform the proposed work includes UND Institute for Energy Studies (IES), Clean Republic LLC, Microbeam Technologies Inc, and the North Dakota Lignite Research council. The team brings together the expertise required to develop a synergistic process that can co-production of multiple high-value solid products from North Dakota lignite coals.

Dr. Michael Mann will be the principal investigator/program manager. He will be responsible for managing resources and schedule, and coordinate meetings with all the project participants/sponsors. He will also be responsible for coordination of all projects within the UND IES and will work with the project team to ensure that all personnel, equipment and other resources are available to efficiently conduct the project.

The project has been organized by task, with task leads/team members responsible for completion of each task. UND IES will be responsible for overall project management and, with technical assistance from MTI, will direct the laboratory-scale efforts in Tasks 2 and 4. Clean Republic LLC will direct the subtaks5.5 effort involving battery performance testing and evaluation of the proposed technology. Figure 5 shows the
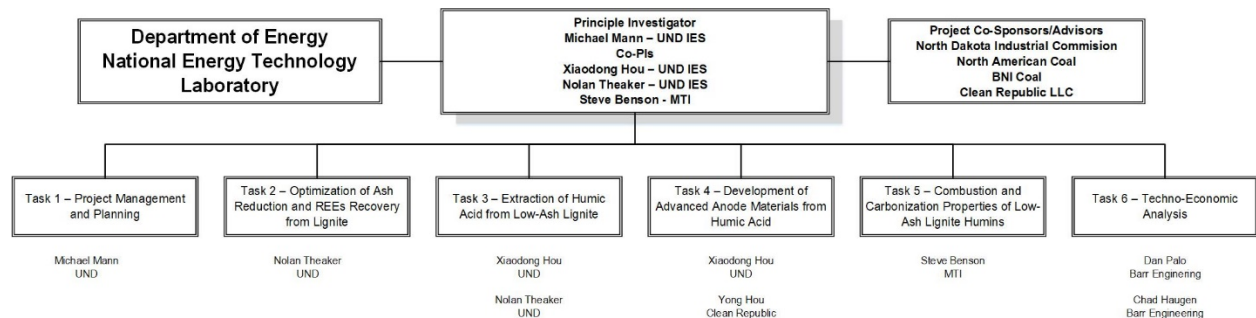

Figure 5. Project organization

management structure for the project that is designed to manage the project by task with key individuals identified to lead the specific areas. The Tasks will be performed by teams of the individuals listed in Figure 5. Cost management will be coordinated by the Administrative Resource Manager who will be responsible for tracking all costs for each of the project tasks.

Project meetings and conference calls will be held, at least, on a weekly basis to conduct project activities, review project timelines, upcoming milestones/deliverables, costs and challenges associated with the completion of the project tasks. Microsoft Project management tools will be utilized. Project review meetings with sponsors will also be held on a quarterly basis to ensure communication and discussion of accomplishments, plans and management of project risks.

Intellectual property management and discussions have been initiated. During the course of the project, any new findings will be promptly documented and patent applications to protect the intellectual property filed as necessary. Discussions with potential commercial sponsors have been initiated regarding further development and scale-up of the technology and will be continued on a semi-annual basis as the project progresses.

9. TIMETABLE

The proposed project timeline is 36 months, with an estimated start date of October 1st, 2019. The project Gantt chart is displayed in Figure BB. Major milestones and planned completion dates are provided in Table 4.

CO-PRODUCTION OF MULTIPLE HIGH-VALUE SOLID PRODUCTS FROM LIGNITE COALS
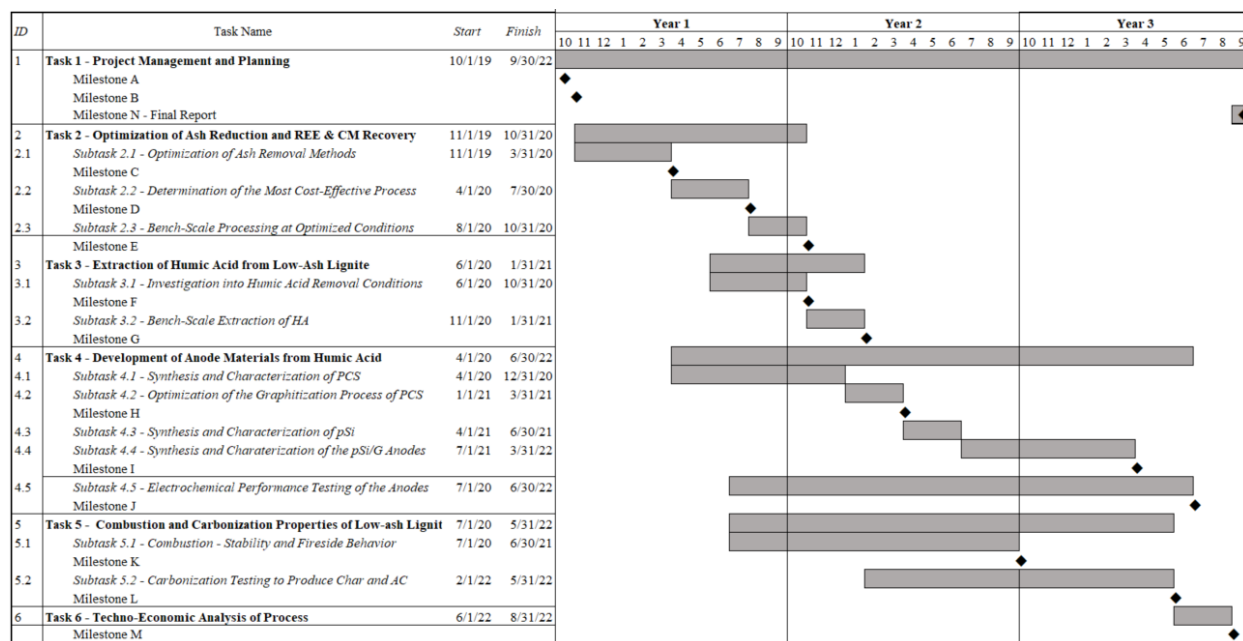
Figure 6. Project Gantt chart.

Table 4. Project Timing and Milestones

Budget Period	ID	Task Number	Description	Planned Completion Date	Actual Completion Date	Verification Method
1	A	1	Update project management plan	10/16/19		PMP file
1	B	1	Kick-off meeting	10/31/19		Presentation file
1	C	2.1	Complete the optimization of ash reduction method	3/31/20		Quarterly Report
1	D	2.2	Determine the REE & CM recovery process	7/30/20		Quarterly Report
1	E	2.3	Complete the bench-scale testing on full ash & REE process	10/31/20		Task Report
1	F	3.1	Determine the optimum method for HA extraction	10/31/20		Quarterly Report
2	G	3.2	Complete bench-scale extraction of HA	1/31/21		Task Report
2	H	4.1 &4.2	Determine the synthetic process for PCS anodes	3/31/21		Quarterly Report
3	I	4.3 & 4.4	Determine the synthetic process for pSi/G anodes	3/31/22		Quarterly Report
3	J	4.5	Complete the battery performance evaluation	6/31/22		Task Report

2	K	5.1	Complete the combustion behavior study	9/31/21		Quarterly Report
3	L	5.2	Determine the optimum conditions for both char and AC production	5/31/22		Task Report
3	M	6	Techno-economic analysis complete	9/01/22		Task Report
3	N	1	Final Technical Report	11/30/22		Final Report

10. BUDGET

The following table gives the summary of the total project budget and the requested funding for each of the cost share partners and the Department of Energy. To simplify the subcontracting and equipment purchases, those costs have been assigned solely to the Department of Energy and only personnel/benefits have been assigned to the cash cost share partners.

Budget Category	Total Project	DOE	NDIC	NAC	BNI	Clean Republic
Personnel	$356,771	$190,049	$82,218	$27,828	$27,828	$28,848
Fringe Benefits	$58,289	$31,050	$13,432.81	$4,546	$4,546	$4,713
Travel	$10,778	$6,042	$4,736	$0	$0	$0
Equipment	$80,000	$80,000	$0	$0	$0	$0
Supplies	$25,050	$14,043	$11,007	$0	$0	$0
Subcontracts	$284,876	$284,876	$0	$0	$0	$0
Other Direct Costs	$38,500	$21,584	$1,916	$0	$0	$15,000
Indirect Cost	$210,361	$121,980	$44,191	$12,626	$12,626	$18,939
Total Cost	$1,064,625	$749,625	$157,500	$45,000	$45,000	$67,500
Percent of Total		70.4%	14.8%	4.2%	4.2%	6.3%

Personnel

Salary estimates are based on the scope of work, and the labor rate used for specific personnel is based on their current salary rate. Generic labor categories have also been established with average labor rates. The table below gives the personnel cost breakdown. Any reference to hours worked on this grant is for budgeting purposes only. The University tracks employee's time based on effort percentage and will not track or report employees time worked on this project in hours. Final numbers may not agree due to rounding.

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Personell	Hours	Cost
Michael Mann, Project Manager	235	$119,443
Xiaodong Hou, Co-PI	1,165	$132,977
Nolan Theaker, Co-PI	655	$73,159
Engineer	1,560	$170,463
Graduate Student	7,060	$24,996
Undergraduate Student	2,840	$143,461
Resource Manager	300	$90,151

Fringe Benefits

Fringe benefits are estimated fro proposal purposed 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 based upon the current rates for each labor category.

Travel

A breakdown of travel is presented in the table below and includes travel required by DOE to review meetings and sampling field trips. Costs have been estimated based on available airfare and lodging rates, conference fees, standard per diems and other UND travel policy. Estimates are broken down as follows:

Purpose of Travel	Depart From	Destination	No. of Days	No. of Travelers	Lodging per Traveler	Flight per Traveler	Vehicle per Traveler	Per Diem Per Traveler	Cost per Trip
DOE Proejct Review Meeting	Grand Forks	Pittsburgh	2	1	$250	$800	$50	$122	$1,222
Technical Meeting	Grand Forks	Unknown	3	1	$325	$800	$50	$183	$1,358
Coal Sampling Trip	Grand Forks	Center, ND	1	2	$0	$0	$100	$61	$322
DOE Proejct Review Meeting	Grand Forks	Pittsburgh	2	1	$250	$800	$50	$122	$1,222
Technical Meeting	Grand Forks	Unknown	3	1	$325	$800	$50	$183	$1,358
Technical Meeting	Grand Forks	Unknown	3	1	$325	$800	$50	$183	$1,358
DOE Proejct Review Meeting	Grand Forks	Pittsburgh	2	1	$250	$800	$50	$122	$1,222
Technical Meeting	Grand Forks	Unknown	3	1	$325	$800	$50	$183	$1,358
Technical Meeting	Grand Forks	Unknown	3	1	$325	$800	$50	$183	$1,358

Equipment

A high temperature oven is required to make the anode materials as proposed. A budgetary quote for an oven meeting the requirements of the project is included.

Equipment Item	Qty	Unit Cost	Total Cost	Basis of Cost	Justification of Need
High Temperature Oven	1	$80,000	$80,000	Cataolog Price	Required for preparation of anode materials
Total Equipment Cost			$80,000		

Supplies

A listing of supplies requested for this work is presented below. The cost is estimated based upon previous work on our rare earth project and other related battery development projects.

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General Category of Supply	Cost	Basis of Cost	Justification of Need
Repair/replacement pH probes	$750	Catalog price	Anticipating needed to replace one pH probe
Battery Assembly	$7,000	Vendor price	Componets for cell assembly
Chemical Reagents	$800	Catalog price	Based upon quantities used in current REE project
Chemical Reagents	$7,500	Catalog price	Acids, Mg powder, silica and other reagents
Lab Consumables	$1,500	Vendor price	Gloves, goggles, weighing paper/boats etc
Samplte containers	$1,000	Vendor price	cups, vials etc for sample storage and shipment
Samplte containers	$500	Vendor price	cups, vials etc for sample storage and shipment
Stock gas bottles	$6,000	Vendor price	Intert gas for sintering
Total	**$25,050**		

Fees – Equipment Use and Laboratory Services / Other

A series of laboratory and analytical tests are required to complete the project. The following table gives a breakdown of these costs, with the basis of costs being established equipment use rates at UND, as well as advertised rates various laboratory service providers.

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General Description	Cost	Basis of Cost	Justification
ICP-MS Analysis	$7,500	Experience from current REE work	Determine quantities of REE in various fractions / optimization of process
Sample Prep	$1,000	Experience from current REE work	Preparing sample for the ICP-MS analysis
Raman Sectroscopy	$1,000	Estimated hourly rate	Confirmation of Graphene and Si
X-ray Diffractometer	$1,000	Estimate of total hours & current hourly rate	Si and Graphite phase identification and purity measurement
Scanning Electron Microscope	$2,000	Estimate of total hours & current hourly rate	Visual observation of precursors and final product for particle size, porous structure morphology
Transmission Electron Microscope	$1,000	Estimate of total hours & current hourly rate	Visual observation of precursors and final product for particle size, porous structure morphology
TOC carbon analyzer	$1,000	Estimate of total sample number & current per sample rate	Carbon content analysis of carbon spheres and final anode products
BET surface area analyzer	$1,000	Estimate of total sample number & current per sample rate	Surface area, and pore size analysis of porous silicon, carbon spheres and final anodes
X-ray photoelectron spectroscopy	$1,000	Estimate of total sample number & current per sample rate	Analyze the oxidation state of silicon
Particle size analyzer	$1,000	Estimate of total sample number & current per sample rate	Particle size analysis of porous precursors and final product
Electrochemical workstation	$1,000	Estimate of total sample number & current per sample rate	Test the electrochemical properties of anode materials
Raman Sectroscopy	$1,000	Estimated hourly rate	Confirmation of Graphene and Si
X-ray Diffractometer	$1,000	Estimate of total hours & current hourly rate	Si and Graphite phase identification and purity measurement
Scanning Electron Microscope	$2,000	Estimate of total hours & current hourly rate	Visual observation of precursors and final product for particle size, porous structure morphology
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Electrochemical workstation	$1,000	Estimate of total sample number & current per sample rate	Test the electrochemical properties of anode materials
Total	$38,500		

Subcontracts

Two subcontracts are included in this budget. The role of each subcontractor is described in the narrative of the proposal. The subcontract amounts are listed here and detailed budgets from each is available upon request.

Sub-Recipient	Role	Cost
Microbeam Technologies Inc	Bid from Microbeam Techologies / Perfom char analysis	$149,916
Barr Engineering	Bid from Barr Engineering / perform TEA	$134,960

Indirect Costs

The indirect cost rate included in this proposal is the federally approved rate for UND of 39%. The indirect cost method is the Modified Total Direct Cost method, defined as the total direct cost of the project minus equipment in excess of $5000, the first $25,000 of each subcontract in excess of this value, tuition remission, and in-kind cost share contributions.

11. MATCHING FUNDS

A breakdown of the funding sources for the project is provided in Table 5.

Table 5. Matching funds and funding breakdown by support source

Support Source	Cash	In-Kind	TOTAL	% of Project
DOE	$749,625	$0	$749,625	70.4
NDIC	$157,500	$0	$157,500	14.8
NACC	$45,000	$0	$45,000	4.2
BNI	$45,000	$0	$45,000	4.2
Clean Republic	$0	$67,500	$67,500	6.3
TOTAL			**$1,064,625**	**100**

12. TAX LIABILITY

No outstanding tax liabilities to the state of North Dakota

13. CONFIDENTIAL INFORMATION

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15. APPENDICES

15.1 Budget Summary and Budget Justification

The following table gives the summary of the total project budget and the requested funding for each of the cost share partners and the Department of Energy. To simplify the subcontracting and equipment purchases, those costs have been assigned solely to the Department of Energy. In-kind contributions from project sponsors are listed in the "Other Direct Costs" line and are described in more detail in the section detailing that budget category.

Budget Category	Total Project	DOE	NDIC	NAC	BNI	Clean Republic
Personnel	$356,771	$190,049	$82,218	$27,828	$27,828	$28,848
Fringe Benefits	$58,289	$31,050	$13,432.81	$4,546	$4,546	$4,713
Travel	$10,778	$6,042	$4,736	$0	$0	$0
Equipment	$80,000	$80,000	$0	$0	$0	$0
Supplies	$25,050	$14,043	$11,007	$0	$0	$0
Subcontracts	$284,876	$284,876	$0	$0	$0	$0
Other Direct Costs	$38,500	$21,584	$1,916	$0	$0	$15,000
Indirect Cost	$210,361	$121,980	$44,191	$12,626	$12,626	$18,939
Total Cost	$1,064,625	$749,625	$157,500	$45,000	$45,000	$67,500
Percent of Total		70.4%	14.8%	4.2%	4.2%	6.3%

Personnel

Salary estimates are based on the scope of work, and the labor rate used for specific personnel is based on their current salary rate. Generic labor categories have also been established with average labor rates. The table below gives the personnel cost breakdown. Any reference to hours worked on this grant is for budgeting purposes only. The University tracks employee's time based on effort percentage and will not track or report employees time worked on this project in hours. Final numbers may not agree due to rounding.

Personnel	Hours	Cost
Michael Mann, Project Manager	235	$21,978
Xiaodong Hou, Co-PI	1,165	$45,387
Nolan Theaker, Co-PI	655	$22,606
Engineer	1,560	$52,675
Graduate Student	7,060	$171,590
Undergraduate Student	2,840	$40,191
Resource Manager	300	$6,080

CO-PRODUCTION OF MULTIPLE HIGH-VALUE SOLID PRODUCTS FROM LIGNITE COALS

Fringe Benefits

Fringe benefits are estimated for proposal purposes only. Upon award implementation, only the true cost of each individual's fringe benefit plan will be charged to the project. Fringe benefits are estimated based upon the current rates for each labor category.

Travel

A breakdown of travel is presented in the table below and includes travel required by DOE to review meetings and technical conferences. Costs have been estimated based on available airfare and lodging rates, conference fees, standard per diems and other UND travel policy. The sampling trips will include travel to mines or power plants or other locations of interest in the state. Estimates are broken down as follows:

Purpose of Travel	Depart From	Destination	No. of Days	No. of Travelers	Lodging per Traveler	Flight per Traveler	Vehicle per Traveler	Per Diem Per Traveler	Cost per Trip
DOE Proejct Review Meeting	Grand Forks	Pittsburgh	2	1	$250	$800	$50	$122	$1,222
Technical Meeting	Grand Forks	Unknown	3	1	$325	$800	$50	$183	$1,358
Coal Sampling Trip	Grand Forks	Center, ND	1	2	$0	$0	$100	$61	$322
DOE Proejct Review Meeting	Grand Forks	Pittsburgh	2	1	$250	$800	$50	$122	$1,222
Technical Meeting	Grand Forks	Unknown	3	1	$325	$800	$50	$183	$1,358
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Equipment

Equipment Item	Qty	Unit Cost	Total Cost	Basis of Cost	Justification of Need
High Temperature Oven	1	$80,000	$80,000	Cataolog Price	Required for preparation of anode materials
Total Equipment Cost			$80,000		

Existing equipment leveraged for this project include the REE bench-scale extraction and concentration equipment, an atmosphere controlled glove box, electrochemical testing equipment, and a number of furnaces and gas analyzers, in addition to material characterization equipment (XRD, SEM/EDS, etc.) The high temperature oven quoted in the equipment is based upon vendor quotes.

Supplies

The bulk of the supplies will be associated with operation of the various laboratory testing tasks. Due to this, the supplies budget is an estimate based upon previous project experience of using this equipment and anticipated flow rates/quantities of chemicals.

CO-PRODUCTION OF MULTIPLE HIGH-VALUE SOLID PRODUCTS FROM LIGNITE COALS

General Category of Supply	Cost	Basis of Cost	Justification of Need
Repair/replacement pH probes	$750	Catalog price	Anticipating needed to replace one pH probe
Battery Assembly	$7,000	Vendor price	Componets for cell assembly
Chemical Reagents	$800	Catalog price	Based upon quantities used in current REE project
Chemical Reagents	$7,500	Catalog price	Acids, Mg powder, silica and other reagents
Lab Consumables	$1,500	Vendor price	Gloves, goggles, weighing paper/boats etc
Samplte containers	$1,000	Vendor price	cups, vials etc for sample storage and shipment
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Stock gas bottles	$6,000	Vendor price	Intert gas for sintering
Total	**$25,050**		

Fees – Equipment Use and Laboratory Services / Other

This budget line includes several different categories of fees. The project scope of work includes characterization of selected feedstocks. A series of laboratory and analytical tests are required to complete the project. The following table gives a breakdown of these costs, with the basis of costs being established equipment use rates at UND, as well as advertised rates various laboratory service providers.

General Description	Cost	Basis of Cost	Justification
ICP-MS Analysis	$7,500	Experience from current REE work	Determine quantities of REE in various fractions / optimization of process
Sample Prep	$1,000	Experience from current REE work	Preparing sample for the ICP-MS analysis
Raman Sectroscopy	$1,000	Estimated hourly rate	Confirmation of Graphene and Si
X-ray Diffractometer	$1,000	Estimate of total hours & current hourly rate	Si and Graphite phase identification and purity measurement
Scanning Electron Microscope	$2,000	Estimate of total hours & current hourly rate	Visual observation of precursors and final product for particle size, porous structure morphology
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15.2 Letters of Support and Cost Share Contributions

WADE BOESHANS
President and General Manager

AN ALLETE COMPANY

March 11, 2019

Dr. Michael Mann
UND Institute for Energy Studies
2844 Campus Road, Stop 8153
Grand Forks, ND 58202

Re: Support of the proposal entitled "Co-Production of Multiple High Value-Added Solids from Lignite Coals" submitted in response to DE-FOA-0001992 "Maximizing the Coal Value Chain"

Dear Dr. Mann:

BNI Energy, Inc. is pleased to support the proposal from the University of North Dakota Institute for Energy Studies to develop a synergistic process that co-produces multiple high value-added solids from North Dakota Lignite Coals. We have been following the work you have been doing in the area of rare earth element extraction and are encouraged by your results. The new work you are proposing has the potential to significantly increase our value proposition. We realize that although the potential volume of lignite that would be processed using your proposed technology is less than our utility market, the value is many times higher. As such, this project is strategic to the future of the Lignite Industry and a good fit with the mission of our company.

As the owner and operator of the Center Mine near Center, ND and a potential benefactor of the result from your work, BNI Coal is pleased to provide a total of $45,000 ($15,000 per year for three years) in cost-share in support for your project, subject to project award by the U.S. Department of Energy.

If you have any questions or require additional information, please contact us.

Sincerely,

Wade Boeshans

LaVern K. Lund
Vice President – Business Development

Telephone: 972-448-5400
Email: vern.lund@nacoal.com

March 18, 2019

Dr. Michael D. Mann
Distinguished Professor, Chemical Engineering
Executive Director, Institute for Energy Studies
University of North Dakota
Collaborative Energy Center, Room 246
2844 Campus Road, Stop 8153
Grand Forks, ND 58202-8153

Re: Value-Added Solids from Lignite Coals
     DE-FOA-0001992

Dear Mike:

North American Coal Corporation (NACoal) is interested in supporting UND's application to DOE for developing value added solids from lignite coals. We believe your project could help open new markets for the lignite industry and provide our economy with useful, low cost carbon products.

NACoal is the largest lignite producer in the United States and one of the top 10 coal producers in the United States. We mine and market coal for use in power generation, SNG production, activated carbon production, as well as, providing selected value-added mining services for other natural resources companies. Our corporate headquarters are in Plano, Texas, near Dallas, and we operate surface coal mines in North Dakota, Mississippi, Texas, New Mexico, and Louisiana.

NACoal is pleased to provide a total cost-share of up to $45,000, over the 3-year term of the project, subject to project award by US Department of Energy and final review.

Best of luck on your application.

Very truly yours,

THE NORTH AMERICAN COAL CORPORATION

LaVern K. Lund
Vice President – Business Development

March 16, 2019

Dr. Michael Mann
UND Institute for Energy Studies
2844 Campus Road, Stop 8153
Grand Forks, ND 58202

Re: Support of the proposal entitled "Co-Production of Multiple High Value-Added Products from Lignite Coals" submitted in response to DE-FOA-0001992 "Maximizing the Coal Value Chain"

Dear Dr. Mann:

This letter expresses our support for your project titled "Co-Production of Multiple High Value-Added Products from Lignite Coals" submitted in response to DE-FOA-0001992 "Maximizing the Coal Value Chain" proposal call. This proposal is in direct alignment with our company's goals to develop materials that will increase the capacity, improve the performance, and reduce the cost of lithium ion batteries. We have been very impressed with the advances you have made in the development of cathode materials, and are incorporating some of the results from your previous work into our battery production. We have great hopes that the work proposed here will result in significant advances in anode production, and something we can use to enhance our product line. As a North Dakota company, we are also excited to see you proposing to use North Dakota lignite, which increases the overall value-chain of one of the primary resources of our state. The fact that your work offers the potential to turn this low-cost feedstock into a valuable end product is exciting to us and we see many benefits as we work towards expanding our battery production line in your state.

We would like to support your project financially by offering to provide support for testing of your batteries by giving you access to and helping test your products at our Battery Test Center. We estimate the value of this support at $22,500 per year for the three years of your project. We will also be available for meetings, consultation, and other exchange as needed to help your project progress.

We are looking forward to working with you on this new venture. As always, don't hesitate to call with any questions.

Sincerely

Michael Shope
CTO - Clean Republic LLC

March 15, 2019

Via email: michael.mann@engr.und.edu

Dr. Michael Mann
University of North Dakota
Institute for Energy Studies
243 Centennial Drive, Stop 8153
Grand Forks, ND 58202-8153

RE: Letter of commitment for University of North Dakota Institute for Energy Studies' (UND-IES) proposal entitled "*Co-production of Multiple High Value-Added Solids from Lignite Coal*," submitted in response to Department of Energy Funding Opportunity Announcement Number: DE-FOA-0001992.
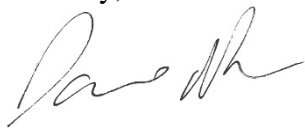
Dear Dr. Mann:

Barr Engineering Co. (Barr) is pleased to provide this letter of commitment to provide research and consulting support for the proposed project, *Co-production of Multiple High Value-Added Solids from Lignite Coal*.

This letter confirms Barr's commitment for the availability of Mr. Chad Haugen (Project Manager) and Dr. Daniel Palo (Project Principal) to support this project, along with additional team members from Barr. Barr's estimated time commitment to this work totals approximately 1050 hours of staff time over the project duration. These hours will be utilized to conduct technoeconomic analysis, market review, and commercialization planning. Additional hours are allotted for project meetings and project management and support for UND-IES.

This letter also confirms that the proposed project is a high priority for Barr, and we will arrange for the necessary resources and personnel to be available to complete our scope of work within the timeframe of the project.

We are excited about continuing our collaboration with UND-IES and look forward to a successful proposal outcome. If there are any questions about our commitment to this project, please do not hesitate to contact us according to the information provided below.

Sincerely,

Dr. Daniel R. Palo, PhD, PE
Vice President, Sr. Process Engineer
dpalo@barr.com
801-333-8421

Chad Haugen
Project Manager, Sr. Process Engineer
chaugen@barr.com
952-842-3618

Dr. Michael Mann   March 16, 2019
University of North Dakota
Institute for Energy Studies
243 Centennial Drive, Stop 8153
Grand Forks, ND 58202-8153

RE: Letter of commitment for University of North Dakota Institute for Energy Studies' (UND-IES) proposal entitled "Co-production of Multiple High Value-Added Solids from Lignite Coal," submitted in response to Department of Energy Funding Opportunity Announcement Number: DE-FOA-0001992.
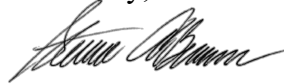
Dear Dr. Mann:

Microbeam Technologies Incorporated is pleased to provide this letter of commitment to provide testing of the coal residue materials for the proposed project, Co-production of Multiple High Value-Added Solids from Lignite Coal.

This letter confirms the availability of Dr. Steve Benson for 380 hours to support this project, along with additional team members Microbeam. Microbeam's estimated time commitment to this work totals approximately 1240 hours of staff time over the project duration. These hours will be utilized to determine the potential uses of the residual coal materials through testing and analysis as described in the scope of work.

This letter also confirms that the proposed project is a high priority for Microbeam, and we will arrange for the necessary resources and personnel to be available to complete our scope of work within the timeframe of the project.

We are excited about continuing our collaboration with UND-IES and look forward to a successful proposal outcome. If there are any questions about our commitment to this project, please do not hesitate to contact us according to the information provided below.

Sincerely,

Steven A. Benson, PhD
President

15.3 Resumes of Key Personnel

MICHAEL D. MANN, PhD

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:

2009-14: College of Engineering (Associate Dean 2013-14; Associate Dean for Research 2009-13

2008:   Interim Dean, UND School of Engineering and Mines:

1999 – Present: UND Department of Chemical Engineering (Professor, 2006-present; Chair 2005-13; Associate Professor, 1999-2006):

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):

PUBLICATIONS (selected from over 150)

- Michael Mann, Daniel Laudal, and Steve Benson, "Maintaining Coal's Prominence in a Carbon Constrained World", Keynote presentation: 2017 International Conference of Coal Science & Technology, Sept 2017.

- Daniel Laudal, Brittany Rew, Steve Benson and Michael Mann, "Technical and Economic Feasibility Analysis of Integrating Activated Carbon with Heating Plant", 2017 International Pittsburgh Coal Conference, Sept 2017

- Mann, M.D.; Knutson, R.Z.; Erjavec, J.; Jacobson, J.P.; "Modeling Reaction Kinetics for a Transport Gasifier", *Fuel* 83 2004 1643-1650.

- Chenguri Qu, Mo Zhang, and Michael Mann, "Effect of Combustion Temperature on the Emission of Trace Elements under O_2/CO_2 Atmosphere during Coal Combustion", IOP Conference Series Earth and Environmental Science, 2018.

- U.S. Patent Number 6,053,954, Methods to Enhance the Properties of Hydrothermally Treated Fuels, 2000

- 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"*

- 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.

- 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.

- 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.

- Feng Xiao, Alemayehu Bedane, Julia Zhao, Michael Mann, and Joseph Pignatello, "Thermal Air Oxidation Changes Surface and Adsorptive Properties of Black Carbon (Char/Biochar)", Science of the Total Environment, 2018.

SYNERGISTIC ACTIVITIES

Dr. Mann's principal areas of expertise include multidisciplinary and integrated energy and environmental projects emphasizing a cradle-to-grave approach; development of energy strategies coupling thermodynamics with political, social, and economic factors; selection of optimum utilization processes emphasizing renewable energy and clean coal technologies; and integration of effluent treatment and emission controls.

Major active projects include "Preparation of Graphene-Modified $LiFePO_4$ Cathode for Li-ion Battery", "Investigation of Rare Earth Element Extraction from North Dakota Coal Related Feedstocks", and "Supercritical Treatment Technology for Water Purification" .

Xiaodong Hou Ph.D.

Institute for Energy Studies, College of Engineering & Mines, University of North Dakota

EDUCATION AND TRAINING

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

2009 **Ph.D.   Polymer Chemistry and Physics**, Shanghai Jiao Tong University, Shanghai, China.

2005 **M.S.   Chemical Engineering**, Shaanxi University of Science and Technology, Shaanxi, China.

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

RESEARCH AND PROFESSIONAL EXPERIENCE

2017- **Research Assistant Professor** Institute for Energy Studies (IES), UND. Research Interests: development of advanced energetic materials (*e.g.*, electrode materials for Li-ion batteries).

2014-2016 **Senior Chemist/Lecturer** Advanced Material Characterization laboratory, Institute for Energy Studies, UND. Main research interests and expertise: synthesis and characterization of advanced chemical materials, environmental sampling and analysis, and crystal structural analysis.

2013-2014 **Interim Lab Director** and **Analytical Chemist** Environmental Analytical and Research Laboratory, UND. Principal areas of expertise: apply various spectroscopic, chromatographic and microscopic techniques to determine the chemical components of broad environment samples.

2010-2013 **Postdoctoral Research Associate** Chemistry Department, UND. Research interests: synthesis and characterization of covalently bonded hierarchical nanomaterials.

2005-2009 **Ph.D. Graduate Research Assistant** Shanghai Jiao Tong University, Shanghai, China. Research interests: Synthesis and characterization of advanced inorganic nanoparticles/block copolymer hybrid materials for energy application.

PUBLICATIONS

1. Baker, J.; Xu, S.; Mann, J.; Dockter, A.; Hou, Y.; Mann, M.; **Hou, X**. Preparation of Lithium Ion Battery Cathode Composites Using Leonardite-Derived Humic Acid, 2018 AIChE Annual Meeting, Pisttburg, PA. October 26-29, **2018**. https://www.aiche.org/conferences/aiche-annual-meeting/2018/proceeding.

PATENTS

1. Xiaodong Hou and Yong Hou. A Low Cost and Reproducible Synthetic Procedure for Mass Production of LiFePO$_4$ Cathode Materials for Lithium Ion Batteries. Application in process.

2. Xiaodong Hou. Preparation of Graphene-Modified Lithium Iron Phosphate Cathode Materials for Lithium Ion Batteries. Application in process.

SYNERGISTIC ACTIVITIES

1) As the PI, leading the project "Lignite-Derived Graphene/Si Nanocomposite Anode for Li-ion Battery", Funding sources: UND VPR Post-Doctoral Program, Amount: $140,000, Total Award Period Covered: 7/1/2019-6/30/21.

2) As the PI, leading the project "Preparation of Graphene-Modified LiFePO$_4$ Cathode for Li-ion Battery", Funding sources: NDIC/Renewable Energy Council/Clean Republic LLC, Amount: $486,238, Total Award Period Covered: 2/1/2018-1/31/20.

3) As the PI, completed the project "A Low Cost and Reproducible Synthetic Procedure for Mass Production of LiFePO$_4$ Cathode Materials for Li-ion Batteries", NDIC Venture Grant Phase I/II, Amount: $246,988, Total Award Period Covered: 1/1/2017-12/31/2017.

4) Dr. Hou presented on the conference "Lithium Battery Materials & Chemistry/ Battery Safety 2017" in Arlington, Virginia, 10/30/2017-11/2/2017. Title: "A Low-Cost Synthetic Procedure for High Consistent LiFePO$_4$ Cathode Materials".

STEVEN A. BENSON, PH.D.

President

Microbeam Technologies Incorporated

Grand Forks, ND 58203

Education and Training

Minnesota State University	Chemistry	B.S. 1977
Pennsylvania State University	Fuel Science	Ph.D. 1987

Research and Professional Experience

1991 – Present President, Microbeam Technologies Incorporated. Dr. Benson founded Microbeam Technologies Incorporated (MTI), a spin-off company from the University of North Dakota to conduct service analysis of materials using automated methods aimed at assessing efficiency and reliability problems in renewable and fossil energy conversion systems. Dr. Benson is responsible for technical direction, data interpretation and proposal preparation.

2015 – 2017 Associate Vice President for Research, Energy & Environmental Research Center, University of North Dakota -- Dr. Benson was responsible for developing and managing projects on the clean and efficient use of fossil and renewable fuels.

2008 – 2015 Professor, Chemical Engineer and Chair, Petroleum Engineering Program and Institute for Energy Studies – coordinated energy related education and research activities that involve faculty, research staff, and students.

1986 – 2008 Associate Director for Research/Senior Research Manager, EERC, UND -- Dr. Benson was responsible for the direction and management of programs related to integrated energy and environmental systems development.

1984—1986 Graduate Research Assistant, Fuel Science Program, Pennsylvania State University.

1983 – 1984   Research Supervisor, Distribution of Inorganics and Geochemistry, Coal Science Division, UND Energy Research Center -- He was responsible for management and supervision of research on coal geochemistry.

1977 – 1983   Research Chemist, Energy Resources Development Administration (ERDA) and U.S. Department of Energy Grand Forks Energy Technology Center, Grand Forks, North Dakota.

Selected Publications and Presentations

1. Laudal, D. A., Benson, S.A., Palo, D., and Addleman, R.S., Rare Earth Elements in North Dakota Lignite Coal and Lignite-Related Materials, ASME, J. Energy Resour. Technol 140(6), 062205 (Apr 09, 2018) (9 pages).

2. Laudal, D. A., Benson, S.A., Addleman, R.S., and Palo, D., Leaching behavior of rare earth elements in Fort Union lignite coals of North America, International Journal of Coal Geology, Volume 191, 15 April 2018, Pages 112-124.

3. Benson, S.A., Crocker, C.R., Hanson, S.K., McIntyre, K.A., Just, B.J., Raymond, L.J., Pflughoeft- Hassett, D.F, Srinivasachar, S., Barry, L.T. and Doeling, C.M., "JV Task 115- Activated Carbon Production from North Dakota Lignite – Phase IIA," Final Report, U.S. Department of Energy Cooperative Agreement No. DE-FC26-98FT40321, June 2008

4. Ma, Z.; Iman, F.; Lu, P.; Sears, R.; Vasquez, E.; Yan, L.; Kong, L.; Rokanuzzaman, A.S.; McCollor, D.P.; Benson, S.A. A comprehensive slagging and fouling prediction tool for coal-fired boilers and its validation/application, Fuel Process. Technol, 2007, 88, 1035–1043.

Synergistic Activities

- Lignite Energy Council, Distinguished Service Award, Research & Development, 1997, 2003, 2005, and 2008. College of Earth and Mineral Science Alumni Achievement Award, Pennsylvania State University, 2002; Science and Technology Award, Impacts of Fuel Impurities Conference, 2014.

Nolan L. Theaker

Research Engineer, Institute for Energy Studies

University of North Dakota, Grand Forks, ND 58202

Education and Training

University of Louisville	Chemical Engineering	B.S. 2016
University of Louisville	Chemical Engineering	M.Eng. 2017
University of North Dakota	Chemical Engineering	Pursuing PhD

Research and Professional Experience

2017-Present   Research Engineer, UND Institute for Energy Studies.

Responsibilities include high-level innovative research and development of novel concepts for submission of funding proposals. Developed into a major technical role in a project involving extraction and purification of rare earth elements from coal and coal-associated feedstocks. Coordinated and conducted efforts associated with downstream rare earth element concentration operations that have resulted in the development of final process flow diagrams. Key contributor to multiple proposals involving REE extraction and/or concentration from multiple feedstocks with innovative ideas for significant cost reductions. Currently co-PI on REE Phase II project work, and leading day-to-day research activities on the project.

2016-2017   Research Assistant, University of Louisville Conn Center.

Research involved design and operation of multi-stage electrochemical reactor scheme for efficient production of fuels from CO_2. Developed nano-functionalized electrocatalysts for improvements in activity and selectivity for targeted reactions in two phase reaction systems. Gained invaluable experience in heterogeneous catalyst chemistry and surface reactions, metal-based catalyst characterization, and deactivation pathways under carbon-based reducing conditions.

2014-2015   Coop Engineer, University of Kentucky CAER.

Research involved improvement and operation of a DOE bench-scale CO_2 capture unit in multiple reaction conditions. Evaluation and comparison of catalyst performance in a holistic view for CO_2 capture was conducted, including novel organic and enzymatic catalysts. Conducted characterization of catalyst degradation and deactivation mechanisms and experiments to improve cyclic lifetimes in harsh process conditions.

Publications/Presentations

1. **Theaker, N.**, Strain, J. M., Kumar, B., Brian, J. P., Kumari, S., & Spurgeon, J. M. (2018). Heterogeneously Catalyzed Two-Ctep Cascade Electrochemical Reduction of CO_2 to Ethanol. *Electrochimica Acta, 274*, 1-8. doi:10.1016/j.electacta.

2. Laudal, D., Benson, S., **Theaker, N**. Investigation of rare earth element extraction from North Dakota Coal-Related Feed Stocks. 2018 NETL Rare Earth Elements Review Meeting. April 10, 2018. Pittsburgh, PA.

3. Laudal, D., Benson, S., **Theaker, N**., Rew, B. Examination of an REE-rich lignite by combined density separations and leaching. 2018 Clearwater Clean Energy Conference. June 6, 2018. Clearwater, Fl.

4. Rew, B., **Theaker, N**., Laudal, D., Holden, J. Determination of rare earth element modes of occurrence in lignite coal. 2018 AIChE Conference. November 1, 2018. Pittsburgh, PA

5. Theaker, N., Laudal, D., Benson, S., Rew, B., Lucky, C. Bench-scale testing update – recovery of rare earth elements from lignite coal. 2019 SME Annual Conference. February, 2019. Denver, CO.

Synergistic Activities

Conducting research on REE occurrence and characterization in lignites and extraction from lignite-derived fly ash feedstocks. Electrochemical characterization experience.

Yong Hou, Ph.D.

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

EDUCATION BACKGROUND

2007   **Ph. D.** Systems Engineering, University of Shanghai for Science and Technology, China

1992   **M.S.** Systems Engineering, University of Shanghai for Science and Technology, China

1983   **B.S.** Electronics Engineering, Hunan University of Art and Science, China

RESEARCH AND PROFESSIONAL EXPERIENCE

2008-present   **Co-founder, VP for Research,** Clean Republic SODO LLC, Grand Forks, ND

Direct the development and production of advanced electrode materials for Li-ion battery and Li-ion battery packs

2008~2012   **Adjunct Professor** Department of Technology, University of North Dakota

2007~2008   **VP for Product Development** Neosonic Li-Polymer Energy Co., Zhuhai, China

Direct the development of new Li-Polymer battery using for light electric vehicles, and Direct the Neosonic business department, ERP system and testing Laboratory

1995-2002   **Founder/General Manager** Shanghai Zhongdian International Company, China

Founded and turn the company's PC service business into the No.1 wholesaler of Compaq Computers in China with an annual revenue of $13M and 50+ employees

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

1983-1989   **Electronics Engineer**   Hunan Puyuan Engineering Machinery Company, China

PUBLICATIONS

1. 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, September **2007**.

2. Yong HOU, Fu-yuan XU, Wei CHENG. "A Microeconomic Model of Optimized Investment Project on the Substitution of Renewable Energy", Commercial Research, **2008-05**.

3. 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**.

4. ZHU Hongbo, GAO Yan, HOU Yong, TAO Li; "Real-time pricing strategy for smart grid based on Markov decision processes"; Systems Engineering – Theory and Practice; Dec. **2017**

5. Hongbo Zhu, Yan Gao and Yong Hou, "Real-Time Pricing for Demand Response in Smart Grid Based on Alternating Direction Method of Multipliers", Mathematical Problems in Engineering, vol. 2018, Article ID 8760575, 10 pages, **2018**

PATENTS

Yon Hou. A New Control System and Lithium Ion Battery Pack for Hybrid E-Bike (China Patent: 2017205084553)

SYNERGISTIC ACTIVITIE

1. As the Co-PI, Dr. Yong Hou completed the project "A Low Cost and Reproducible Synthetic Procedure for Mass Production of LiFePO4 Cathode Materials for Li-ion Batteries", Funding sources: NDIC, Amount: $246,988, Total Award Period Covered: 1/1/2017-12/31/2017.

2. As the Co-PI, Dr. Yong Hou is conducting the project "Preparation of Graphene-Modified LiFePO4 Cathode for Li-ion Battery", Funding sources: NDIC, Amount: $490,139, Total Award Period Covered: 2/1/2018-1/31/2020.

3. As the principal investigator, Dr. Yong Hou is leading the project "Advanced Integrated Solar-LFP Battery Powered Water Pump System for Remote Farm fields". NDIC, Amount: $30,000, Total Award Period: 5/01/2018 – 4/30/2019.