Warren J. Baker Endowment
for Excellence in Project-Based Learning
Robert D. Koob Endowment *for Student Success*

PROPOSAL NARRATIVE

I. Project Title: Synthesis and Characterization of Novel Electrode Materials for Lithium-Sulfur Batteries

II. Abstract

Lithium-sulfur (Li-S) batteries are becoming increasingly attractive as one of the most promising advanced secondary batteries with overwhelming advantages of high theoretical capacity (1675 mAh/g) and high energy density (2600 VS. 420 W h/kg of traditional Li-ion batteries). Sulfur is one of the most abundant elements on earth and is an underutilized byproduct from the oil and gas industries. Additionally, in comparison to Li-ion batteries, Li-S batteries have improved safety and lower cost. They are also more environmentally friendly. However, the predominant challenge with lithium-sulfur batteries is capacity drop and low cycle life during usage of the sulfur-based electrode. This project aims to solve this problem by careful design of carbon based nanomaterials to physically and/or chemically confine the sulfur component. Developing the sulfur/carbon nanocomposite will be conducted using well-studied synthesis processes. Characterization of the electrodes will be conducted through analytical techniques *via* scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), x-ray diffraction (XRD), scanning tunneling microscopy (STM), and atomic force microscopy (AFM). Electrochemical evaluation of assembled split cell will consist of cyclability and rate capability testing using the electrochemical testing station. The expected outcome is to achieve high performance of Li-S batteries with long cycle life and maintaining high specific capacity, a goal for emerging advanced energy storage technology, portable electronics, and grid-scale energy station.

III. Introduction

Global interest in battery technology has increased greatly in recent decades because of its impact in portable electronics, grid-scale energy storage, and electric vehicles. However, integrated energy storage is a limiting component in current and emerging technologies that requires intense development if systems are to reach optimum overall efficiency [1]. Current Lithium-ion batteries are nearing the theoretical limit for specific energy density and capacity; however, the demand for higher performing batteries is increasing. Lithium-sulfur batteries will play a vital role in forthcoming technology due to several advantages [2]. Sulfur is one of the most abundant elements on Earth and is an underutilized byproduct from oil and gas industries [3]. Essential to battery performance is energy density; lithium-sulfur batteries with a theoretical energy density of 2600 W-h kg^{-1} offer an overwhelming advantage to the conventional lithium-ion batteries (387 W-h kg^{-1} for $LiCoO_2$/C) [4-8]. Additionally, it has the highest theoretical capacities (1675 mA-h g^{-1}) compared to transition metal oxide cathodes [9]. Its relatively low operating voltage (2.15 V vs Li/Li^{+}) offers improved safety. Moreover, the cost of sulfur ($160 USD per ton, 2012) is much lower than that of transition metals [10]. Sulfur is also more environmentally friendly [11]. The overall chemical reaction is: $S_8 + 16Li \leftrightarrow 8Li_2S$ [12, 13]. However, the main barrier preventing widespread use of lithium-sulfur batteries is its low cyclability due to degradation during operation: upon charging and discharging, sulfur decomposes to polysulfides, which dissolves in the liquid electrolyte. Over time, the overall efficiency will decrease. One milestone of recent Li-S batteries work is from Dr. Nazar's group, who reported improvements in cycling performance in lithium-sulfur batteries in 2009 [14]. Since then, increasing efforts in research have been

focused on the chemical and physical confinement of sulfur to further enhance its electrochemical performance. Carbon-based materials have shown promising performance and research work remains in the structural design of the sulfur electrode to achieve the most efficient confinement of polysulfides. One such promising carbon-based material is carbon nanotubes (CNTs) [15]. It is reported that nanochannel of CNTs have unique electronic tuning properties, suitable for confinement material of sulfur [16, 17].

Battery research and development is inherently multidisciplinary, primarily drawing upon Materials Engineering, Chemistry, Physics, and Electrical Engineering. Materials engineering primarily focuses on the structure, properties, and performance of the major material components of a battery: cathode, anode, and electrolyte. The sciences uncover the thermodynamics and kinetics of the electrochemical reaction involved in battery science. Electrical engineering focuses on the systems integration and optimization by assembling battery packs from battery cells. Through collaboration with Dr. Erik Sapper and Dr. Gregory Scott from the Chemistry Department and Dr. William Ahlgren from the Electrical Engineering Department, this project combines expertise from multiple professors at Cal Poly. Students from across colleges will be involved in various stages of the project. For instance, chemistry students will support the synthesis and characterization of the sulfur nanocomposites through Dr. Scott and Dr. Sapper's AFM/STM Lab. Electrical engineering students will assist with the battery cell assembly and electrochemical testing. Moreover, there will be collaboration with researchers outside Cal Poly. For instance, Dr. Hao has received collaborative invitation from the Department of Materials Science at Florida International University for battery testing. Dr. Sapper has contacts in Boeing with interest in consulting on this project.

This is the first student project on battery cell research and development at Cal Poly. This presents the unique opportunity to lay the foundation for future student involvement in battery technology at Cal Poly. Because this project draws upon collaboration among multiple disciplines both within and outside the Cal Poly community, it can attract attention from academic institutions, industry, funding agencies, current students, and future students. Many universities worldwide conduct research for energy storage. This is especially true in California with large industry players in portable electronics, electric vehicles, and renewable energy. As batteries are becoming increasingly important, the demand for engineers with experience in batteries is also increasing. As a result, a new field of battery engineering is now emerging. For example, San Jose State University now offers an Engineering Master's program in the Specialization in Battery Technologies. These indications reveal the rapid growth of battery engineering and its role in worldwide technologies in the future.

IV. Objective(s)

The goals of this research project are to investigate the confinement effect of the sulfur/carbon electrode and further enhance the electrochemical performance of sulfur electrode for Li-S batteries. In doing so, it will develop students' hands on research abilities and further Cal Poly's presence in battery research on the national and international scale to attract attention from researchers in related fields. To achieve this goal, the following sections are consisted in this project: (1) Experimental Setup: the awarded funding will help on purchasing necessary chemicals, equipment, and experimental supplies. (2) Experimental work includes: materials synthesis, characterization and electrochemical evaluation. All the three parts will have scientific data obtained and further analysis to draw conclusions (3) Dissemination: we are aiming to publish this work as one journal paper and present at professional research conferences. The funding will also be used as the support for conference registration and travelling.

V. Methodology

Materials Synthesis

- Pristine sulfur will be synthesized by mixing $Na_2S_2O_3$ and HCl in aqueous solution, followed by filtering and washing. The sulfur powder will be dried overnight in an air-oven before use. Cut CNT will be obtained by Raw CNTs opened and cut into segments of 0.2−1 μm in length by refluxing in HNO_3.

- The Sulfur/CNT nanocomposite will be synthesized by chemical reaction deposition as described above by adding CNT in the solution during reaction and followed by heat treatment in the tube furnace with Argon gas flow.

Characterization

- Morphology of prepared electrodes will be analyzed by SEM and AFM. The elemental composition and mapping results of the samples will be determined by XRD and EDS.
- The amount of sulfur in the composites will be determined by TGA. Surface characterization of the electrodes will be investigated by FTIR and STM.

Electrochemical Evaluation

- The electrochemical properties will be tested in a half cell using lithium metal as a counter electrode and evaluated by cyclic-voltammetry (CV), galvanostatic charge/discharge and electrochemical impedance spectroscopy (EIS). Specific capacity, rate capability and cyclability will be assessed using these experiments.

VI. Timeline

Table I: Tentative Timeline of Tasks/Milestones

Objectives	Subtasks	Nov.	Dec.	Jan.	Feb.	Mar.	Apr.	May.	Jun.
Experimental Setup	Materials/Equipment Acquisition	x	x						
	SOP/Safety Report	x	x						
Experimentation	Sulfur Synthesis		x	x	x	x			
	CNT Treatment		x	x	x	x			
	Sulfur Loading		x	x	x	x			
	Battery Assembly		x	x	x	x			
Characterization	SEM/EDS/TGA/STM/ XRD/FTIR/AFM			x	x	x	x		
Electrochemical Testing	Cycle Test			x	x	x	x		
Dissemination	Poster/Publication					x	x	x	x

VII. Final Products and Dissemination

The final product will be replicates of lithium-sulfur battery cells. The final dissemination will consist of intellectual property, a journal publication, posters, and presentations. The formal journal publication will be submitted to Journal of the Electrochemical Society. Furthermore, the publication and poster will be presented at the 2018 Electrochemical Society Conference, specifically the Li-ion Batteries and Beyond division (May 13 -17). A poster and PowerPoint presentation will also be presented at the Cal Poly all-college Project Expo to faculty, students, and industry professionals.

VIII. Budget Justification

The total proposed budget is $4955. The estimated travel expense for airfare and ground transportation for the ECS Conference for students is $1350. The total operating expenses of $3605 is divided as experimental supplies, chemicals, printing, shipping, and registration. The major costs are split cell apparatus for battery assembly ($596), the lithium foil ($687) and the electrolyte ($227) and other chemicals. Main vendors will be Sigma-Aldrich and MTI Corporation. The remaining costs will cover student registration at the ECS Conference ($545), poster printing for the ECS Conference and the Cal Poly Project Expo ($100), and shipping costs for equipment, supplies, and materials ($300).

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CAL POLY

PROPOSAL BUDGET

Student Applicant(s): Glenn Lee Luc Tousignant	
Faculty Advisor: Dr. Yong Hao	
Project Title: Synthesis and Characterization of Novel Electrode Material for Lithium-Sulfur Batteries	**Requested Endowment Funding**
Travel *subtotal*	**$1350**
Travel: In-state	**$**
Travel: Out-of-state	**$1350**
Travel: International	**$**
Operating Expenses *subtotal*	**$3605**
Non-computer Supplies & Materials	**$2660**
Computer Supplies & Materials	**$**
Software/Software Licenses	**$**
Printing/Duplication	**$100**
Postage/Shipping	**$300**
Registration	**$545**
Membership Dues & Subscriptions	**$**
Multimedia Services	**$**
Advertising	**$**
Journal Publication Costs	**$**
Contractual Services *subtotal*	**$0**
Contracted Services	**$**
Equipment Rental/Lease Agreements	**$**
Service/Maintenance Agreements	**$**
TOTAL	**$4955**

Project Bibliography

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