FINAL REPORT

Final reports will be published on the Cal Poly Digital Commons website (http://digitalcommons.calpoly.edu).

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

II. Project Completion Date:
Synthesis and characterization of the sulfur/CNT nanocomposite was completed in May 2018. Battery assembly and electrochemical testing was completed in January 2019.

III. Student(s), Department(s), and Major(s)
(1) Glenn Lee, Materials Engineering
(2) Nicholas Greco, Materials Engineering
(3) Gabriela Remulla, Materials Engineering

IV. Faculty Advisor and Department
Dr. Yong Hao, Materials Engineering

V. Cooperating Industry, Agency, Non-Profit, or University Organization(s)
California Polytechnic State University, San Luis Obispo
University of California, Los Angeles

VI. Executive Summary
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 Wh/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. 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 focused on embedding sulfur nanoparticles into a carbon-based nanomaterial, carbon nanotubes. The physical and/or chemical confinement of sulfur was hypothesized to reduce the chemical degradation of the sulfur cathode under prolonged charging and discharging cycles. The composite synthesis of the sulfur/carbon nanocomposite was conducted by using chemical oxidation of the carbon nanotubes and heat treatment in an argon-filled environment (Figure 1). Characterization of the electrodes was conducted through analytical techniques via scanning electron microscopy (SEM) (Figure 2), thermogravimetric analysis (TGA), and X-ray diffraction (XRD) (Figure 3). Collaboration with the University of California, Los Angeles allowed for the assembly of
coin cells batteries and the electrochemical evaluation of the batteries. The cathode consists of an 80:10:10 ratio of the sulfur/CNT composite, carbon black, and PVDF binder, respectively. Battery coin cell of type 2032 were assembled in an argon-filled glovebox with a lithium foil anode and a 1 M LiPF$_6$ in EC:DMC electrolyte. Galvanostatic charge and discharge tests (Figure 4) were carried out on a Landt Battery Testing Systems CT 2001A.

![Composite Synthesis Schematic](image)

**Fig. 1** Composite Synthesis Schematic.

![SEM images](image)

**Fig. 2** SEM images captured on SEM FEI Quanta 200 of (A) Raw MWCNT (B) cMWCNT (C) MSC (D) cMSC (E) HT MSC (F) HT cMSC.

![XRD scans](image)

**Fig. 3** XRD scans on XRD Siemens D5 000 of cMWCNT and MWCNT (left); and cMSC and MSC (middle). TGA results of MSC, cMSC, HT MSC, HT cMSC composites measured from 25 °C to 400 °C at constant ramp rate of 10 °C/min. Heat treated composites are in bold (right).
The cycling performance of the MSC and cMSC composite in the Li-S battery system is lower than typical experimental measurements. This is likely attributed to the high instability of the sulfur nanocomposite in the 1 M LiPF$_6$ in 1:1 EC:DMC electrolyte. This suggests the use of a more suitable electrolyte system for future cycling performance of sulfur-based cathodes in lithium-sulfur battery systems. Despite the poor cycling performance, the cMSC cathode exhibited signs of improved capacity performance when compared to that of the MSC cathode.

VII. Major Accomplishments

(1) Synthesis of a sulfur infused carbon nanotube composite (MSC and cMSC) with approximately 68 weight percent sulfur. X-ray diffraction confirmed the presence of sulfur and carbon nanotubes in the synthesized composite. Scanning electron microscopy confirmed the particle size of the synthesized composite. Thermogravimetric analysis confirmed the composition of sulfur and carbon nanotubes in the composite.

(2) Synthesis and characterization results were presented in May at the poster session of the 2018 Electrochemistry Society Conference. A technical presentation was also given at the Materials Engineering Technical Conference to industry professionals, students, and faculty. Lastly, a poster was also presented at the Cal Poly Project Expo.

(3) Galvanostatic cycling showed higher current density in cMSC than in MSC. This correlates with the hypothesis that sulfur nanoparticles may infiltrate the inner diameter of the carbon nanotubes and improve the electrochemical performance of lithium-sulfur batteries.

VIII. Expenditure of Funds

The funds for this project were used for the purchase of chemicals, lab supplies, testing equipment, and registration and travel for presenting a student poster at the 233rd Electrochemical Society Meeting. The necessary chemicals for this project include the precursors for synthesizing the composite, the anode, solvents, electrolytes, and other additives necessary in all commercially constructed electrochemical batteries. The lab equipment purchased were essential for the chemical processing of precursors.
IX. Impact on Student Learning

This project embodies the Cal Poly “learn-by-doing” spirit by enabling undergraduate students to gain first-hand experience from the ground level in conducting current research in alternative lithium-ion energy storage. Specifically, this work analyzes the electrochemistry of lithium-sulfur batteries, which is among the promising candidates for energy storage research. Students learned how to design and conduct experimental research based on state-of-the art literature research. The first part of this project involved the synthesis of the sulfur/CNT composite. After purchasing chemicals, lab supplies, and equipment, students set up lab space to conduct experiments. Throughout the project, students gained proficiency using widely practiced materials characterization techniques, such as thermogravimetric analysis, X-ray diffraction, and scanning electron microscopy. These techniques were used to confirm successful synthesis of the sulfur/CNT composite. Students also utilized engineering tools, such as Python and Excel, to analyze and plot data acquired from various experimental tests. The second part of this project took place at UCLA, where students gained the unique opportunity to work in a lab designed specifically for electrochemistry. Students assembled their coin cell batteries in an argon-filled glove box and performed electrochemical measurements of their batteries. These processes are fundamental techniques used in experimental research in electrochemical energy storage. In addition to the technical aspects of conducting research, students presented their work at various professional conferences to students, professors, and industry professionals. Continuing undergraduate students expanded this project to a new direction during Summer session of 2018 by investigating the electrochemical performance of sulfur nanocomposites with a CNT/PEDOT dual confinement as cathode for Li-S batteries. The Baker and Koob endowment enabled students to design and carry out their own experimental research based on state-of-the-art literature research from the guidance of a faculty supervisor that served students well in their future career path.