



The Chemical Company

Final Report

Project # DE-EE0000563

Process for Low Cost Domestic Production of LIB Cathode Materials

Anthony Thurston

BASF Corporation

23800 Mercantile Road

Beachwood, OH 44122

anthony.thurston@basf.com

Tel: 216-360-5043

NETL Project Manager: John Tabacchi

Department of Energy Final Project Report

DOE Award # DE-EE0000563

Award Recipient: BASF Corporation

Project Title: Process for Low Cost Domestic Production of LIB Cathode Materials

Principle Investigator

Anthony Thurston
BASF Corporation
23800 Mercantile Road
Beachwood, OH 44122
anthony.thurston@basf.com
Tel: 216-360-5043

Project Team Members

Stephen Sheargold, PhD
BASF Corporation
23800 Mercantile Road
Beachwood, OH 44122
stephen.sheargold@basf.com
Tel: 216-360-5056

Zhong Shi, PhD
BASF Corporation
23800 Mercantile Road
Beachwood, OH 44122
zhong.shi@basf.com
Tel: 216-360-5110

Subcontractors

Farasis Energy, Hayward CA
SKC Powertech, Inc, Mt. Olive NJ

Executive Summary

The objective of the research was to determine the best low cost method for the large scale production of the Nickel-Cobalt-Manganese (NCM) layered cathode materials. The research and development focused on scaling up the licensed technology from Argonne National Laboratory in BASF's battery material pilot plant in Beachwood Ohio. Since BASF did not have experience with the large scale production of the NCM cathode materials there was a significant amount of development that was needed to support BASF's already existing research program. During the three year period BASF was able to develop and validate production processes for the NCM 111, 523 and 424 materials as well as begin development of the High Energy NCM. BASF also used this time period to provide free cathode material samples to numerous manufacturers, OEM's and research companies in order to validate the materials. The success of the project can be demonstrated by the construction of the production plant in Elyria Ohio and the successful operation of that facility. The benefit of the project to the public will begin to be apparent as soon as material from the production plant is being used in electric vehicles.

Project Objectives and Goals

The goal of the project was to scale up the production of cathode materials that were developed in the laboratory and to ultimately validate the process by producing cathode material in the production plant at a few

tons level. BASF would sub contract the building and testing of 18650 cells with Farasis Energy and commercial partners for the production and validation of a Li ion battery pack for use in an EV application.

Initial Statement of Work

Task 1.0: Project Management and Planning

Includes all work elements required to revise and maintain the Project Management Plan and to manage and report on activities in accordance with the plan.

Task 2.0: Synthesis of Advanced NCM in a semi-batch laboratory scale process

Synthesis of Advanced NCM in the lab to evaluate production parameters prior to transition to the Pilot Plant, a detailed study on the individual process steps where the amount of material produced is 1 to 5 kilograms.

Subtask 2.1: Production of Advanced NCM in the Pilot Plant level

The transfer of the lab process to the Pilot Plant to validate and fine tune the processing conditions as needed to meet the specifications set during the lab scale process.

Subtask 2.2: Production Trials for Advanced NCM at the Production Plant level

The transfer of the Pilot process to the Production Plant to validate and fine tune the processing conditions as needed to meet the specifications set during the Pilot scale process.

Task 3.0: Synthesis of High Energy NCM (Layered-Layered materials) and the High Voltage Spinel materials in a laboratory scale semi-batch scale process. Synthesis of High Energy NCM (Layered-Layered materials) and the High Voltage Spinel materials in the lab to evaluate production parameters prior to transition to the Pilot Plant, a de-tailed study on the individual process steps where the amount of material produced is 1 to 5 kilograms.

Subtask 3.1 Production of High Energy NCM (Layered-Layered materials) and the High Voltage Spinel materials in the Pilot Plant. The transfer of the lab process to the Pilot Plant to validate and fine tune the processing conditions as needed to meet the specifications set during the lab scale process.

Task 4.0: Complete electrochemical testing of all materials

Make and Test Prototype 18650 Cells. BASF will refine cell level and battery pack level PHEV specifications and reinforce cathode material specifications (capacity, cycle life, cost, power, calendar life, etc.). Cathode materials produced throughout the project will be incorporated into 18650 cylindrical power/energy PHEV cells by Farasis and tested by BASF, Farasis, and our commercial automotive partners.

Subtask 4.1: Prototype LIB Pack Production

Make and Test Prototype Battery Pack – BASF is in discussions with several auto OEMs and during this project, BASF will refine cell level and battery pack level PHEV specifications and reinforce cathode material specifications (capacity, cycle life, cost, power, calendar life, etc.).

Actual Accomplishments

BASF was able to complete all the above tasks. The prototype packs have been assembled by SKC Power-tech using BASF's NCM 111 and NCM 424 and have been tested and approved by SKC for use in an EV application. Farasis Energy has completed testing of BASF's NCM 523 and has approved the material for large scale production. The Production plant has successfully produced both NCM 111 and NCM 523 at multi ton levels and has met all internal specifications with the production material. The development of the high energy NCM and the high volt-

age Spinel have been completed to the Pilot Plant level and samples have been provided to partners for evaluation.

Project Activities

The original hypothesis was that the scale-up and development of the NCM materials would be accomplished using existing rotary tube furnaces and that contamination issues would be minimized by the selection of raw materials and process parameters. It was quickly discovered that the rotary tube furnace could not be utilized without going to a ceramic tube due to contamination issues and furthermore the residence time was not sufficient to fully react the lithium with the NCM precursor. At this point BASF made a decision to invest in a new and dedicated battery materials pilot plant and as a result the development was slowed down due to the installation of the pilot plant that was completed in August of 2010. During the construction phase BASF was able to focus on the lab phase of the research and development and was able to utilize equivalent equipment for large scale testing and initial sample development. After the completion of the pilot plant BASF was able to focus on the scale-up and development of the NCM materials. The other deviation from the original plan was the development of the LFP cathode. Since BASF was not comfortable with its patent situation and licensing was taking longer than expected, it was decided to postpone the LFP development and expand on the NCM materials. Although this did not present a problem for the overall project it did result in a change from the proposal. Not included in the proposal was the development of the High Energy NCM and the High Voltage spinel, due to the significant interest in these two materials BASF decided to advance the development process and run some early scale up experiments to provide free samples to cell producers for evaluation and feedback. It is well known that there are risks associated with advancing from the research phase to the development phase too quickly but due to the market demand and time required to commercialize a new material it was viewed as an acceptable risk. There is still much to do both at the research level and at the development stage for the High Energy NCM and High Voltage Spinel materials however BASF has been able to make both significant improvements in the product quality as well as increasing the potential production capacity. The overall impact of changing from the less expensive rotary tube furnace to a more sophisticated tunnel type kiln did require significant process changes that had to be addressed in the research but the result was a higher quality product that is meeting the end user requirements.

Final Products

At present the only materials that have been fully developed and are in commercial production are the NCM 111, 523 and 424 cathodes, extensions to alternate formulations such as 325, 622, 811 are possible but still require some additional development and validation time. The production plant in Elyria will be able to produce any and all of the NCM based materials.

Patents/Applications

USSN 13/168516 filed on 6/24/2011 "Process for Synthesis of a Layered Oxide Cathode Composition"

Acknowledgment: "This material is based upon work supported by the Department of Energy [National Energy Technology Lab under Award Number(s) DE-EE0000563."

Disclaimer: "This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof."