RECYCLING AND UPCYCLING SPENT LITHIUM-ION BATTERIES

The Growing Lithium ion Battery Market Creates Parallel Disposal Problem

Advances in the commercial development of lithium ion batteries have spawned significant growth of new products in a wide range of industries. The demand of lithium-ion batteries is being fueled by growing popularity of hybrid and electric vehicles, grid and renewable energy storage, as well as battery backup applications in healthcare, military and telecommunications. According to a Frost & Sullivan analysis, the global lithium-ion battery market in 2013 was $18 billion -- and is estimated to quadruple by 2020[1].

The growing lithium-ion battery market creates a parallel disposal problem. Unless new technologies are developed to deal with spent lithium-ion batteries, environmental and economic issues will undoubtedly proliferate. There is currently no known commercial technology for the large scale recycling cathode materials of multiple chemistries. The hazardous waste classification of spent Li-ion batteries encumbers their transportation, treatment, and disposal costs[2]. The disposal cost of this hazardous waste can far exceed the value of the raw materials used in the original lithium ion batteries.

Applying American Manganese's Patented Hydrometallurgical Process to Create Better Batteries

American Manganese has developed an innovative processing technology to treat metal oxide materials for a wide range of markets. The low-cost, environmentally friendly hydrometallurgical process was initially developed to recover manganese from lower grade resources. The technology was developed

[1] http://ww2.frost.com/news/press-releases/innovation-across-key-industries-quadruple-revenues-lithium-ion-batteries/

[2] Gaines, L. (2014) The future of automotive lithium-ion battery recycling: Charting a sustainable course. *Sustainable Materials and Technologies* 1-2: 2-7.

over a five year period, following extensive bench and pilot scale testing that was partially funded by the Canadian Government through the National Research Council, Industrial Research Assistance Program (NRC-IRAP).

The research undertaken by American Manganese successfully lead to patents issued in the United States of America (US Patent No. 8460631), Republic of South Africa (Patent No. 2013/01364) and China (Patent No. 201180050306.7). The technology is patent pending in several other jurisdictions[3].

Details of the technology and applications are described in the patents and peer reviewed publication in the February, 2012 edition of the Minerals and Metallurgical Processing Journal. A paper was also presented in the Battery Power 2012 conference in Denver, Colorado.

The American Manganese Recovery Process: Overview

The process uses a unique combination of commercial equipment to selectively leach the metals of interest and precipitate them as high purity intermediate products (such as manganese carbonate), which can then be reformulated to multiple end products (such as manganese metal, electrolytic manganese dioxide, chemical manganese dioxide or lithium manganese oxide). The overall process is requires significantly less energy than conventional processing by implementing an ambient temperature reduction leach in place of high temperature reduction roasting. In addition, a low energy water recovery method was developed to enable water reuse in a closed loop fashion. These unique combinations of unit operations form the core of the American Manganese patent.

American Manganese has successfully completed a pilot plant study to process resource material and extract high purity compounds. These compounds were subsequently processed to make cathode materials and functional rechargeable lithium-ion button cells. A photograph of the pilot plant is provided in Figure 1. Figure 2 shows the high purity manganese carbonate that was generated from the pilot study. Figure 3 shows the intermediate products created to manufacture button cells.

[3] Issued patents: US Patent No. 8460631 and Republic of South Africa Patent No. 2013/01364. Patent currently pending in other jurisdictions.

Figure 1. Hydrometallurgical Pilot Plant for Recovery of Manganese from Artillery Peak, Az Resources

Figure 2. High Purity Manganese Carbonate Produced from Hydrometallurgical Pilot Trial

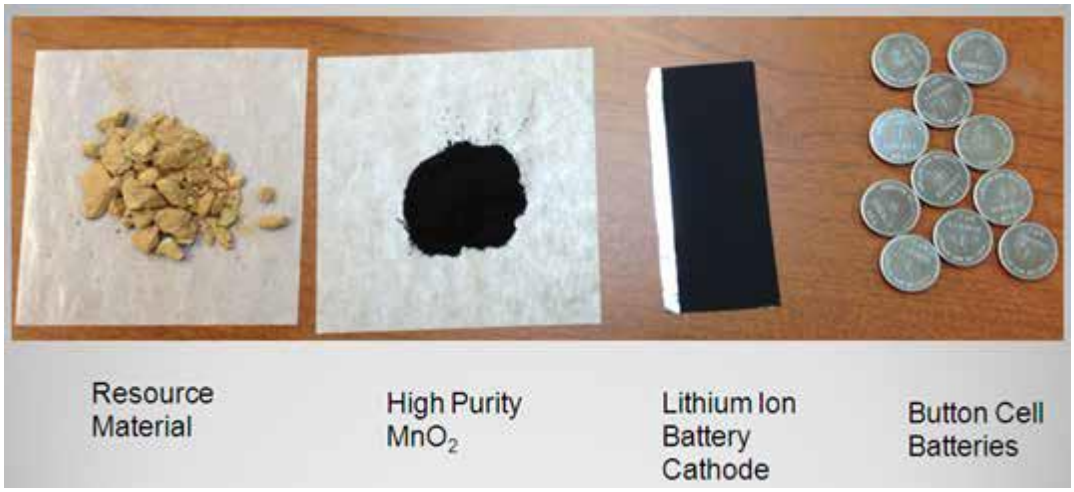

Figure 3. Resource to Battery Research

Figures 4 to 15 shows the step-by-step processes undertaken to produce button cells from cathode material produced from resource material sourced from Artillery Peak, Arizona.

			
Figure 4: Lithium manganese dioxide produced by double controlled heat treatment of chemical manganese dioxide From American Manganese Artillery Peak material mixed with lithium carbonate.	Figure 5: Weighing Artillery Peak LMO.	Figure 6: Adding isopropyl alcohol to suspend the cathode material.	Figure 7: Mixing cathode “ink”.

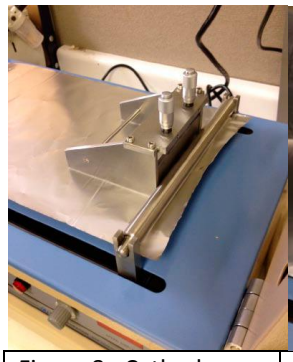	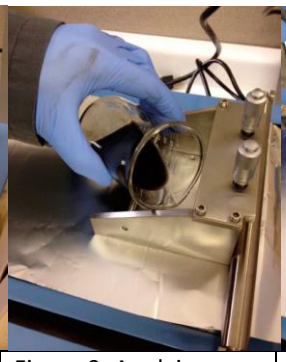	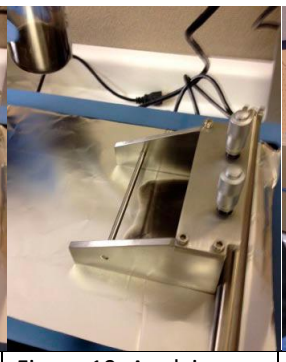	
Figure 8: Cathode coating device.	Figure 9: Applying cathode “ink” to aluminum foil.	Figure 10: Applying even thickness of material.	Figure 11: Baking cathode material.

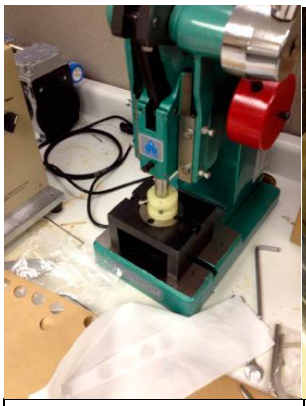

Figure 12: Punching cathode disks.

Figure 13: Assembling button cell in controlled humidity chamber.

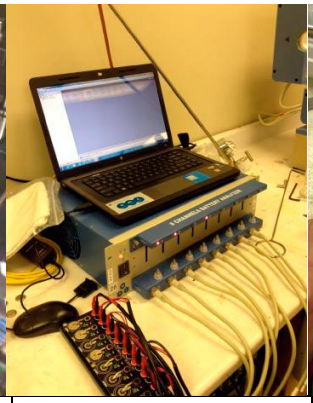

Figure 14: Charging button cells made from Artillery Peak material.

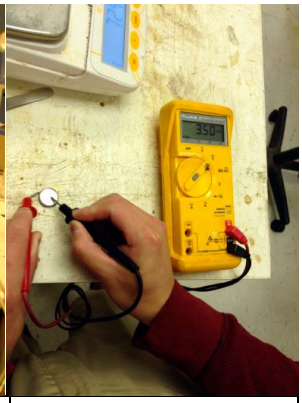

Figure 15: Measuring cell voltage, 3.5 Volts

American Manganese and strategic partner Kemetco Research Inc. ("Kemetco") developed this method starting from low grade Mn raw ore. The breakthrough of this proprietary process employs hydrometallurgical techniques in lieu of typical comminution or costly roasting, ultimately generating superior cathode material.

Advantages Over Current Methods: Increased Purity, Decreased Safety Risk

Crushing and grinding operations in conventional mining processes are known to introduce metallic impurities. Avoiding these metal shard residues left behind from wear of steel grinding equipment is a critical factor in overall lithium-ion battery safety: even immeasurable metallic impurities in the parts-per-billion can potentiate thermal runaway, causing over-heating, or even fire and explosion. The tolerances for these impurities are so low that there are no current analytical methods to measure. As such, all lithium-ion battery manufacturers test every battery manufactured for a few weeks to reduce (but not eliminate) the occurrence of thermal run-away of production batteries prior to sale.

American Manganese's processing scheme can also be tailored to improve the chemical purity of the overall cathode compound. This can be accomplished with the integration of proven hydrometallurgical techniques such as selective leaching, selective precipitation, ion exchange, solvent extraction or

selective crystallization. Creating a higher purity product will prevent build-up of impurities at the solid-electrolyte interface (SEI) layer which is known to cause battery fade and decreased performance after repeated charge and discharge cycles.

Opportunity to Recycle and Upcycle Spent Lithium-Ion Batteries

American Manganese aims to capitalize on the patented technology and proprietary know-how to recycle cathode materials used in high tech lithium ion batteries. The cathode is the most expensive component of the battery's cost (comprising 20 – 30% of the cell material cost). The cathode is the component that stores and discharges energy within the battery; it consists of lithium imbedded in a base metal oxide matrix. Eventually, even rechargeable Li-ion batteries will exhaust their useful life.

With lithium ion battery recycling in its infancy, American Manganese aims to be an industry leader in the recycling of cathode materials used in lithium ion batteries. The spent cathode represents an ideal resource material to be processed with American Manganese's proprietary hydrometallurgical process.

The flexibility of the American Manganese process also creates a unique opportunity to upcycle spent cathode material. Unlike recycling, upcycling shifts resource material back up the supply chain into a usable form, without degradation to latent value. Upcycling can be repeated in perpetuity, reducing waste and need for virgin stock. This would create an opportunity where spent batteries could be returned to the original manufacturer and reprocessed to new product. A significant value proposition could be achieved by eliminating the hazardous waste classification of existing spent batteries. The shipment of materials classified as hazardous waste is highly regulated. With rules differing in different jurisdictions, dealing with the regulatory requirements to transport spent batteries can be extremely restrictive, time consuming and expensive. Upon development of a viable upcycling industry, it is possible to have spent batteries reclassified as a useful raw material instead of a hazardous waste. If successful, shipment of spent batteries could follow the same established international regulations as new batteries.

The value propositions for advancing American Manganese's technology for the treatment of spent cathode materials are:

1. CREATING A HIGHER VALUE PRODUCT

- The potential to process spent cathode materials to produce higher purity products that improve lithium-ion battery performance and safety.
- Ability to tailor treatment process to precipitate cathode materials with tailored structures and chemistries for improved battery performance

2. CREATING AN UPCYCLING INDUSTRY FOR SPENT BATTERIES

- Potential to recycle all lithium and base metal matrix compounds
- Applicable to multiple lithium ion battery chemistries including, LiCoO2, LiNixMnyCozO2 and LiMn2O4 and other chemistries.

3. ELIMINATING THE HAZARDOUS WASTE CLASSIFICATION OF SPENT LITHIUM – ION BATTERIES

- Creating an industry where spent batteries become a useful raw material rather than a waste.
- Avoid costly and time consuming regulatory requirements to ship spent lithium-ion batteries for disposal.

A process flow diagram for recycling and upcycling spent lithium-ion batteries with the American Manganese process is shown in Figure 12.

AMERICAN MANGANESE CONCEPTUAL LITHIUM ION BATTERY RECYCLING AND UPCYCLING FLOWSHEET

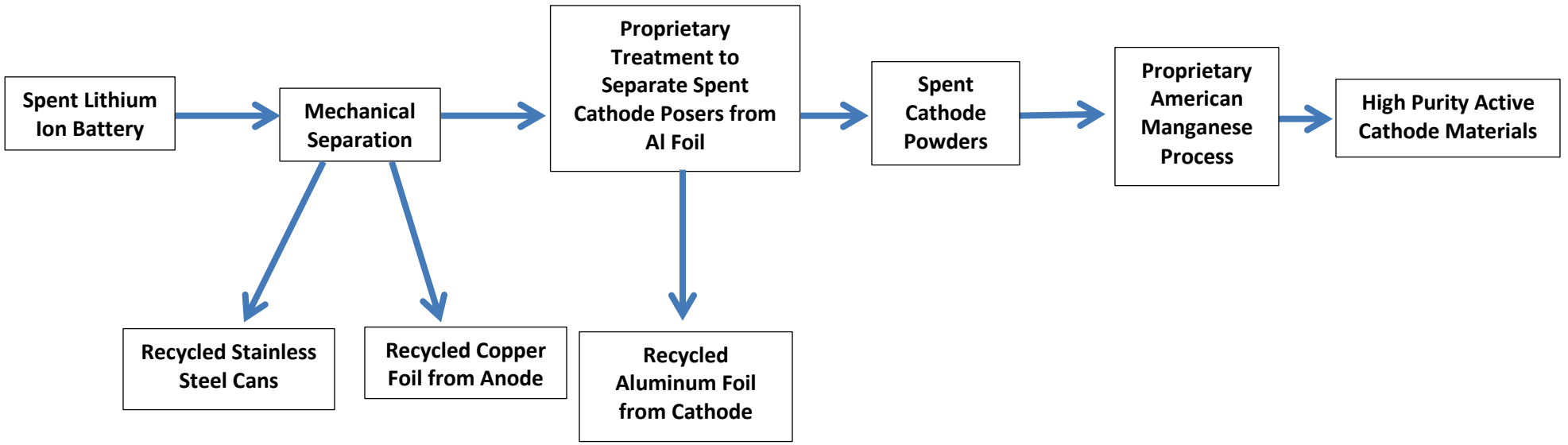

Figure 12. American Manganese Conceptual Lithium-Ion Battery Recycling and Upcycling Flowsheet.

Next Phase of Work

The next phase of work will be a 4 to 6 month study to establish "proof of concept" that the American Manganese process can be adapted to work for the recovery and re-formulation lithium ion battery cathodes. To streamline the overall process, the initial focus will be to process cathode materials of different chemistries (such as lithium manganese, lithium cobalt, lithium manganese nickel cobalt and lithium iron phosphate). The target is to prove that rejuvenated products can be produced and working lithium ion battery buttons cells can be made.

Data gathering and operational observations can be used to refine procedures needed for further advancement. The following tasks can be considered for planning and merit evaluation purposes.

1. **LITHIUM-ION CATHODE POWDER PROCUREMENT:** Approximately 5-10 kg of representative feed for testing is required. It is desirable to test cathode materials of different chemistries such as lithium manganese oxide, lithium cobalt, lithium manganese nickel cobalt, and lithium iron phosphate. In order to streamline the proof of concept phase, the test work will focus on treating fresh cathode powders procured from commercial suppliers.

2. **METAL EXTRACTION TESTS (LEACHING):** The American Manganese leach process will be applied and parameters varied to test the recovery of key components with the focus on selectivity and yield.

3. **UPGRADING TESTS:** Test work will be conducted on select solutions to see if a premium product is achievable with purification and post-treatment steps to target improved performance of rejuvenated batteries.

4. **REFORMULATION & CHARACTERIZATION TESTS:** Select reformulated lithium-ion battery cathodes will be characterized for chemical composition and physical structures.

5. **PRODUCTION OF BUTTON CELL BATTERIES:** Ultimately, proof of concept will be established when working lithium-ion batteries are produced from the reprocessed cathode materials. Following establishing proof of concept, a decision point will be reached to determine the next stage of development.

American Manganese Inc. is actively exploring many potential sources funds for this work, including: government research grants available in Canada or the United States; a strategic alliance with an established company in the lithium ion battery, electric vehicle, or materials recycling businesses; a joint venture with a funding group; and sale of a royalty on future use of the American Manganese Patented Process.

# # #

PROPOSAL

1. PROJECT TITLE

Recycling Spent Lithium-Ion Batteries

2. OBJECTIVE

The propose of this project would be to establish "proof of concept" for adopting the American Manganese flow sheet for recycling cathode material from spent lithium batteries. Kemetco has successfully conducted bench and pilot scale tests in the past to develop the flow sheet for producing high grade material from low grade manganese resource for American Manganese Inc. This research will focus on adopting the patented hydrometallurgical flow sheet for processing cathode powder for use in lithium ion batteries and prove that fresh cathode material can be generated.

3. STATEMENT OF WORK (SCOPE OF WORK)

The following tasks will be undertaken to achieve the Objective as defined in Section 2. It must be emphasized that the exact experimental program may change as more information is obtained.

Research Methodology:

TASK 1: CATHODE POWDER PROCUREMENT

Approximately 5-10 kg of representative cathode powders is required for testing. This material can be provided from commercial suppliers. Various analytical testing such as ICP and XRD will be applied to determine the characteristics of the starting materials.

TASK 2: LEACHING STUDY

Leaching optimization will be investigated with a guidance of American Manganese leach process. The leach selectivity and recovery will be carefully studied using vital parameters to achieve the best conditions for recovering of key components.

TASK 3: PURIFICATION AND UPGRADING STUDY

Various hydrometallurgical techniques such as selective precipitation, solvent extraction and ion exchange will be applied to investigate the possibility of upgrading the solution characteristics for the final product. Producing premium products using purification and post-treatment steps will be studied to improve performance of the final products.

TASK 4: FINAL PRODUCT CHARACTERIZATION

Primary products will be characterized considering chemical composition and physical structure. The task will be accomplished by reformulation of new cathode materials using techniques that may result in improved products.

TASK 5: PRODUCTION OF BUTTON CELL BATTERIES

Lithium-ion batteries will be ultimately produced using the recovered cathode materials from the previous tasks. Successful results in this step will lead the project to the next stage of the development.

4. DELIVERABLES

Reporting and deliverables will be as follows:

- A summary of all research findings will be issued at the end of each test with relevant details to the project sponsor;
- A comprehensively written report summarizing all research findings will be issued at the end of the test program.

5. ESTIMATED PROJECT SCHEDULE

The project will begin upon receipt of project financing. It is estimated that the test work will take 6 months to complete. The report will be issued four weeks after completion of bench testing.

Note: Every effort will be made to complete each task on schedule provided that the project is not delayed due to reasons outside the control of Kemetco. If it is anticipated that the project will not be completed within the proposed schedule, the project sponsor will be notified as soon as possible.

Table 1. Estimated Project Schedule

TASK	WEEKS												
	1-2	2-4	4-6	6-8	8-10	10-12	12-14	14-16	16-18	18-20	20-22	22-24	24-26
Cathode Powder Procurement	■												
Leaching Study		■	■	■	■								
Purification and Upgrading Study				■	■	■	■	■	■				
Final Product Characterization									■	■			
Production of Button Cell Batteries											■		
Reporting												■	■