Recycling EV waste: A materials challenge and industrial opportunity?

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ReLiB – Short loop recycling

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Critical elements required for batteries

Production of key elements required for battery manufacture (such as Lithium and Cobalt) are limited to a small number of countries.

With the electric car revolution and move towards renewable technology, competition for such resources will inevitably increase.

Co is expensive and 60% of production is currently concentrated in the DRC (mining linked with human rights abuses).[1]

In addition, if current demand continues to rise, models predict that **known reserves of Co and Li will be considerably depleted by 2050.**[2]

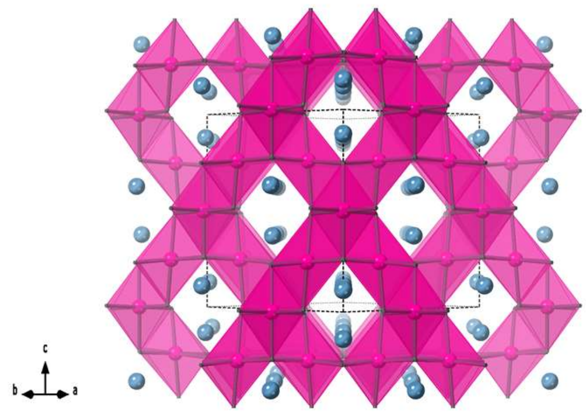
Given the rise in electric vehicles:

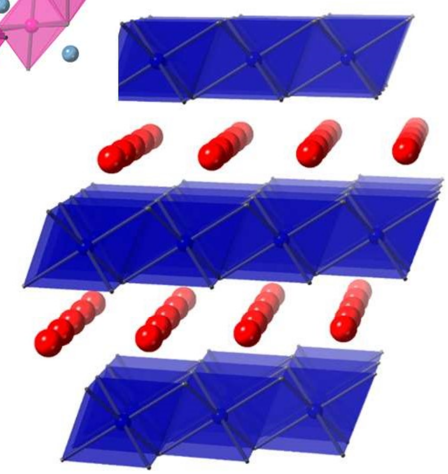
- Vital need to develop strategies to recycle of spent batteries
- Opportunity for UK to take a lead in this area → new UK industries

1. https://wapo.st/2mjkbfk?tid=ss_mail&utm_term=.8870e57e6160 (accessed 30/10/18)
2. A. Pehlken, S. Albach, T. Vogt, Is there a resource constraint related to lithium ion batteries in cars?, Int. J. Life Cycle Assess. 22 (2017) 40–53.

Cathodes (present, near-future and future)

- Current on-road EVs: cathodes contain a mixture of Li-Mn-O(spinel) and NMC (layered oxide).
- Most car manufacturers focusing on utilising NMC/NCA ($LiNi_xMn/Al_yCo_zO_2$), for future EVs (similar properties of $LiCoO_2$ but at a much reduced cost).
- Ultimate aim is to move towards compositions containing less Co such as NMC622 or 811.





Challenges of short loop recycling

Delivering a cost effective process

- Current on road EVs Mn rich cathodes:- low resale value
- Greater impetus to recycle low value cathodes by recovering the materials and converting to high value next generation cathodes (e.g. Spinel→ NMC)
- Subsequent repurposing treatments must be economically justifiable (avoiding high temperature heat treatments etc.)

Gap in characterization of spent batteries

- Vital need for studies of EV batteries at various SOH (state of health): Identifying any changes that have occurred/contaminants (eg. F) will aid in developing regeneration methodologies.

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The binder problem

- Flawed assumption that the binder is inert. However, previous research in our group has shown that PVDF is a powerful low temperature fluorination agent (200-400°C).
- Although the temperature required for fluorination is much higher than what batteries would normally be exposed to, batteries cycled over many years and/or mistreated may create conditions for substitution to occur.
- Even if fluorination has not occurred during cycling, fluorine incorporation is likely to be a problem during regeneration
- Fluorine and oxygen are notoriously difficult to distinguish using common characterization methods.


ELSEVIER

Journal of Fluorine Chemistry 117 (2002) 43–45

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Poly(vinylidene fluoride) as a reagent for the synthesis of K_2NiF_4-related inorganic oxide fluorides

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Abstract

In this paper we report an improved route to the synthesis of K_2NiF_4-related inorganic oxide fluorides, such as $Sr_2TiO_3F_2$ and $Ca_2CuO_2F_2$ using low-temperature fluorination of precursor oxides with poly(vinylidene fluoride). Use of this fluorinating agent results in high quality samples, without SrF_2 or CaF_2 or other impurities, which are commonly seen for alternative fluorination routes.
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Keywords: Poly(vinylidene fluoride); K_2NiF_4; Inorganic oxide fluoride; Low-temperature fluorination

RSC Advances

PAPER

Direct regeneration of cathode materials from spent lithium iron phosphate batteries using a solid phase sintering method

Journal of Power Sources

Short communication

Recovery and heat treatment of the $Li(Ni_{1/3}Co_{1/3}Mn_{1/3})O_2$ cathode scrap material for lithium ion battery

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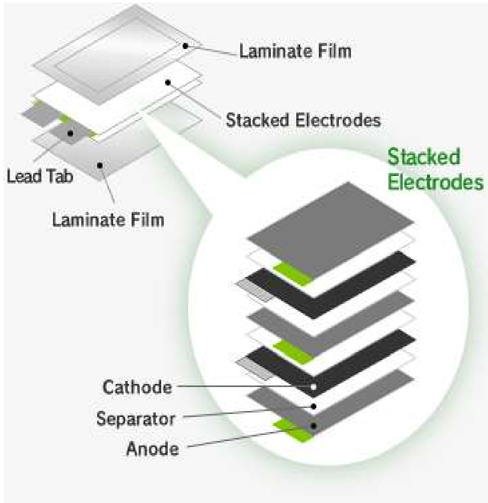
Tackling the characterization gap

Diamond proposal

Investigations of failure mechanisms in real Li ion battery pouch cells and its implications on recycling

- In depth study looking at batteries in various SOH (Pristine, QA failed and end of life). To date, most studies tend to focus on catastrophic failure whereas this study will aim to identify potential issues with batteries entering the recycling chain. This should aid in improving the efficiency of any subsequent regeneration experiments. (March 2019)



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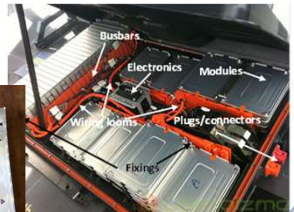
Current short loop methods



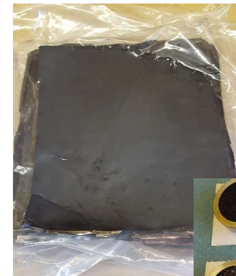
Disassembly

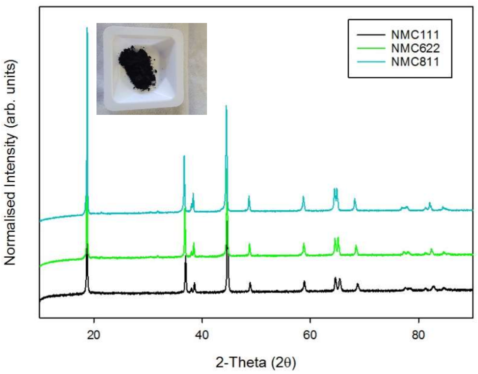
Shredding/Sieving

Leaching/precipitation

Heat treatment

Reassembly





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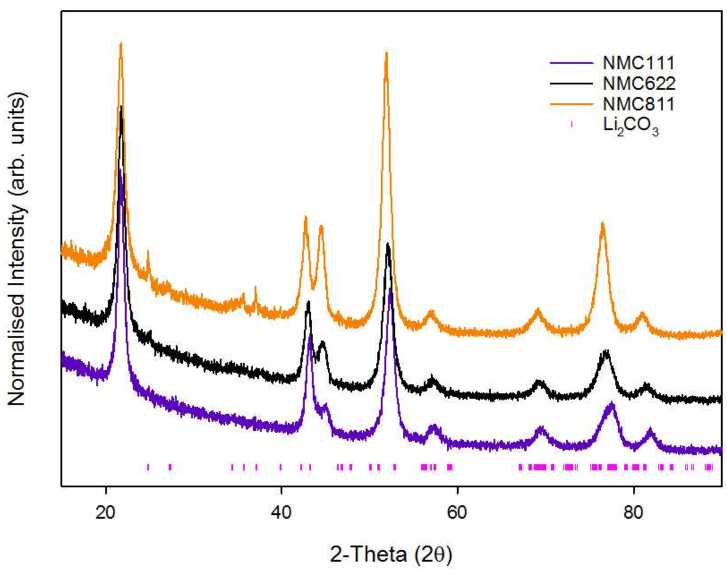
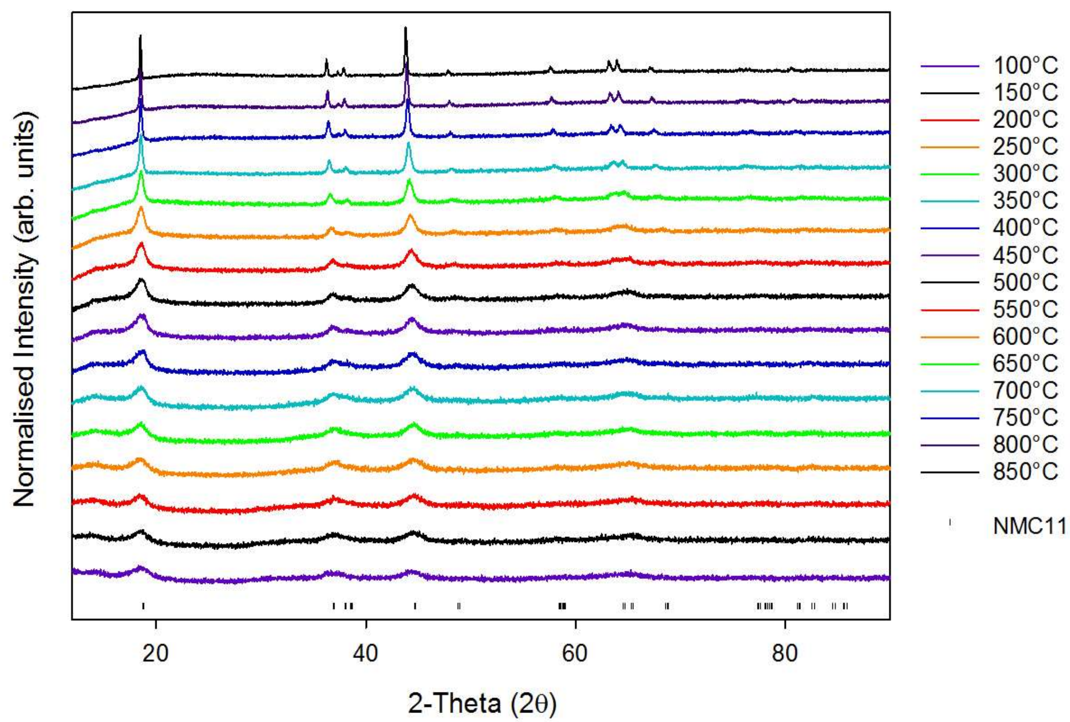
Developing low T regeneration routes

In order to investigate potential regeneration routes, regeneration studies performed using solutions that would be typical for a standard leaching procedure → newly developed ReLiB project methodologies

Able to synthesize NMC at an intermediate (550°C) and low (100°C) temperatures

Joint ISIS/diamond proposal submitted to study changes in structure of NMC (synthesised at 100°C) with increasing temperature.





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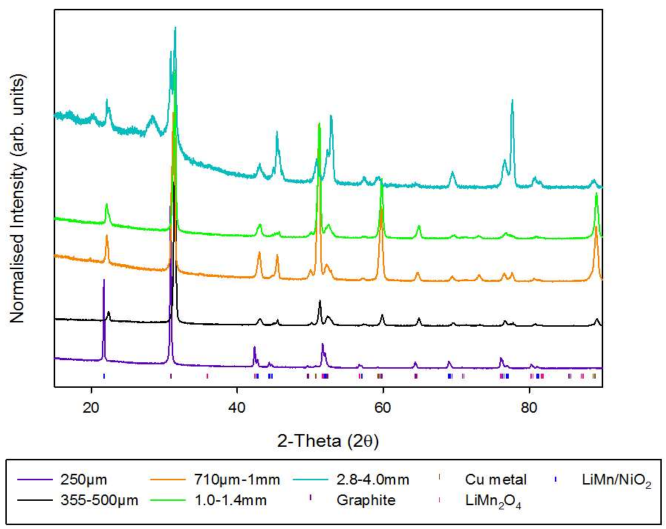
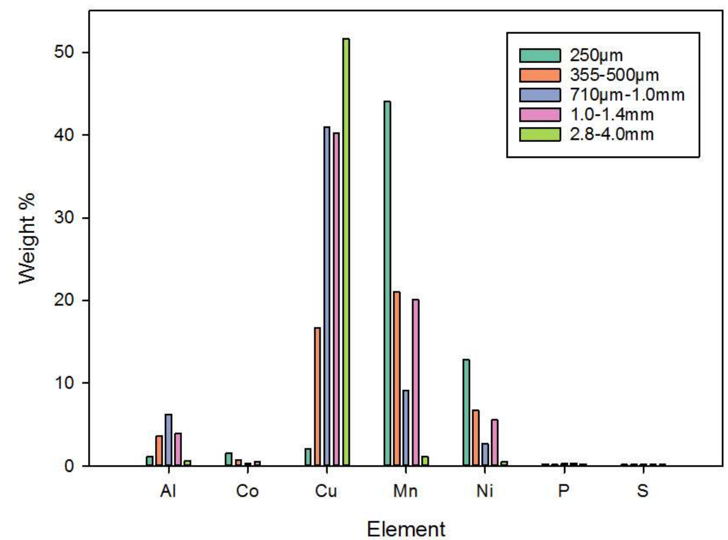
Characterisation of shredded/manually separated battery components

- Cryogenically shredded – the battery is dipped in liquid nitrogen before being shredded. The recovered material is then fed through a number of sieves to aid separation.
- Manually dismantled – current collector sheets coated in electrode

Verdict? Poor separation. Only the smallest fraction can be considered for regeneration.





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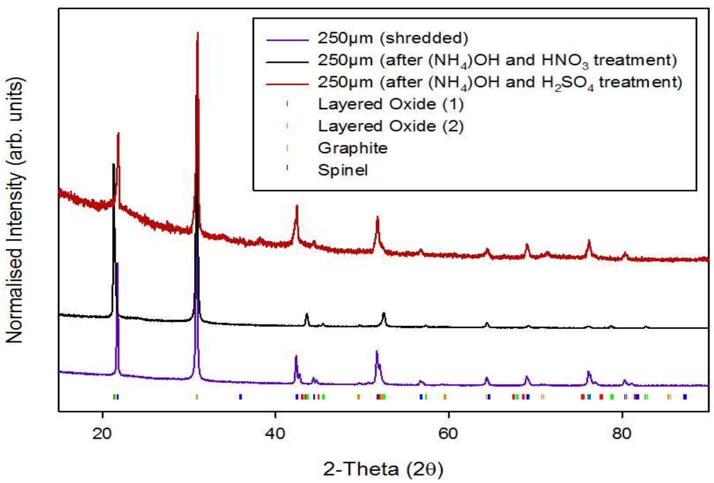
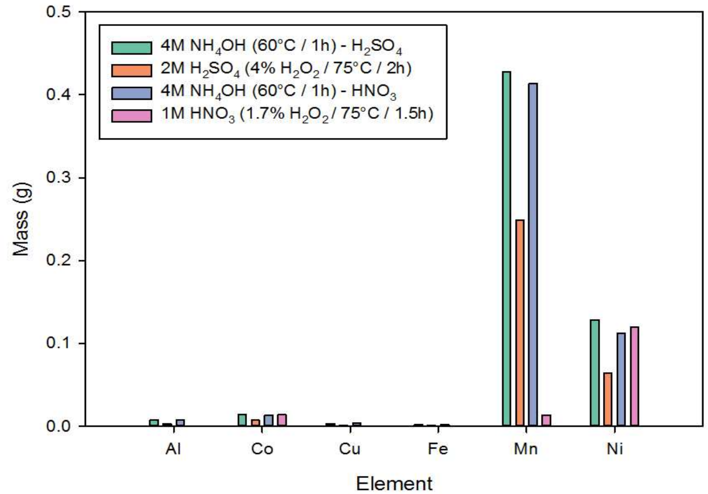
Materials processing

- Identified possible purification routes from the literature.
- First step – treat shredded fraction with 4M $(NH_4)OH$ (60°C/1h) to remove Cu and Al impurities. [1]
- Second step – Select leaching acid to leach key metals from shredded material.
  - 2M H_2SO_4 / 4% (v/v) H_2O_2 / 2h / 60°C [1]
  - 1M HNO_3 / 1.7% (v/v) H_2O_2 / 1h / 75°C / S:L = 50 [2]

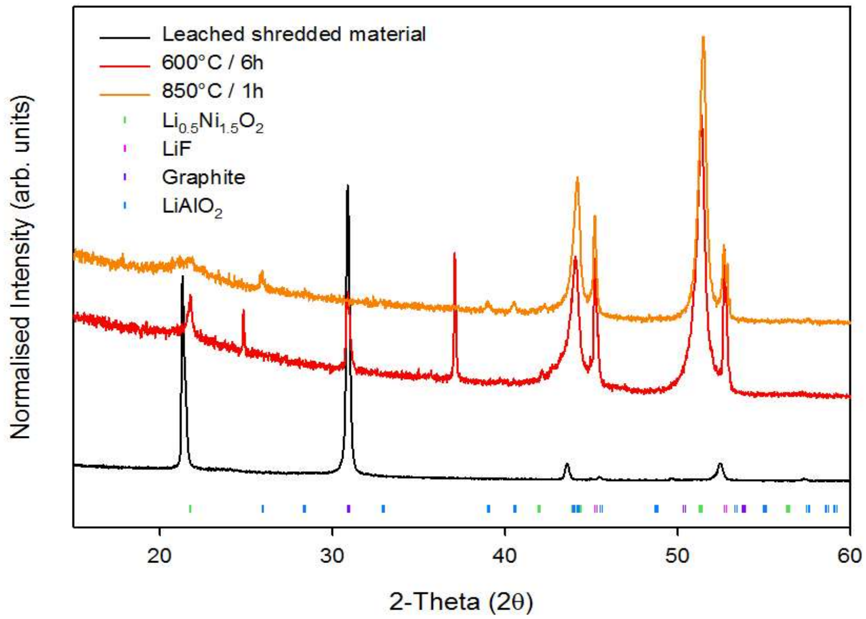




Shows the importance of knowing which phases are present in shredded material before attempting purification – Nitric acid appears to remove the spinel phase while sulfuric removes the layered phase.

1. A.A. Nayl, R.A. Elkhashab, S.M. Badawy, M.A. El-Khateeb, Acid leaching of mixed spent Li-ion batteries, A. J. Chem. 10 (2017) S3632–S3639.
2. C.K. Lee, K.I. Rhee, Preparation of $LiCoO_2$ from spent lithium-ion batteries, J. Power Sources. 109 (2002) 17–21.

Regeneration challenges



- After leaching, attempted to burn off the graphite and polymer.
- Added a lithium source to regenerate the layered phase at the same time.
- Instead of regeneration, LiF formation occurs.

Summary of current progress

- Accomplishments
  - Developed low temperature routes to regenerate NMC from typical solutions produced from leaching experiments
  - Initial purification studies of shredded material
  - Diamond Beamtime awarded to characterise the condition of materials within EV batteries.
  - Battery from Nissan leaf (Mileage of ~40,000) removed and sent to Eco-bat for shredding.

- Current/Future work
  - Conducting the same leaching/heat treatments on the manually dismantled coated electrodes
  - Evaluating methodologies used to detach electrode materials from current collectors – does the route used have any impacts on short loop recycling?
  - Outreach activities being developed in conjunction with chembam (https://chembam.com).

Thank you for listening

Any questions?