CENTS: A Research Network for the Sustainable Transport Community

Circular Economy Network+ in Transportation Systems

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CENTS Feasibility Funding Case Study Template

Title of Feasibility Study:

1. Project Team

Names, expertise and photos of project team

Lauren Cooper MChem. 2 years' experience working at WMG on projects related to energy innovation, with 1 year in the battery recycling team working on new methods for recycling lithium ion batteries.

Anwar Sattar, PhD, Meng. Leads battery recycling activities at WMG. Has been working on lithium ion battery recycling since 2016

John Low, PhD, BEng. Focuses on the application of electrochemical engineering for lithium and lithium-ion batteries, taking materials science into technology manufacturing and recycling.

Logo(s) of industry partner(s)

2. Executive summary (max 200 words)

This research has demonstrated that membranes for ion transport have the potential to recover lithium-ion from waste recycling solutions to prepare purified LiOH, which can be fed back into lithium-ion battery production. Proprietary Nafion membranes have been successfully prepared using different methods to improve lithium-ion conductivity, suitable for both solid polymer electrolytes and lithium-ion recovery. H-cell and flow cell were designed and set up for testing of membranes. During testing of the lithium-ion recovery process, ion-transport and pH changes occurred in the absence of an applied cell voltage which suggests the important consideration of ion-selectivity. Commercial Fumatech membranes were also tested in this discovery study, which shows early stage promises for future research into the recovery of purified LiOH battery precursor material.

The problem (max 200 words)

Purify lithium using electrochemical means. Electrochemical methods previously have been used to transport lithium from one electrode to the other but creating a membrane that is selectively permeable to a specific ion, in this case lithium is a big challenge. This work will look into the development of a membrane that is selective to lithium ions, this will allow for the purification of aqueous lithium solutions in a cost effective and efficient manner.

The approach (max 100 words)

A literature survey was carried out to summarise the different practical lithiation methods proposed in literature. The lithiation methods achieving the greatest conductivity values in literature were carried out on fresh Nafion film.
These films were then characterised and tested alongside two commercial ion exchange membranes in an electrochemical H-Cell, to assess whether lithium ions can be transferred from a 1M LiOH solution into water. pH measurements of the water solution over time established that ion movement had occurred. Finally the best performing membrane was tested in a larger scale flow cell set up.

Novelty (max 100 words)

This project is the first demonstration of the ion transport of lithium ions through an ion selective membrane for the purpose of purification.

Results (max 500 words)

Sample Preparation

Sample	Processing Method
Nafion	N/A
Li-Nafion 1	Stirred in 1M LiOH for 12 h at 80 °C Thoroughly rinsed in distilled water.
Li-Nafion 2	Same as previous one, followed by immersion into PC for 1h.
Li-Nafion 3	Dipped in 1M LiOH aqueous solution and kept for 12 h at 80 °C. 12h in 80°C deionozed H_2O.
Li-Nafion 4	48h in 2M LiCl solution under constant stirring. Repeated rinsing with deionized water.
Li-Nafion 5	Boiled in deionized water for 4 hours. Then 10wt% of H_2O_2 for 3h at 80°C. Rinsed in boiling deionized water. Soaked in 2M LiOH under stern stirring at 80 °C for 2 h.
Fumasep FKL-PK-75 Cation Exchange Membrane	N/A
Fumasep FKS-30 Cation Exchange Membrane	N/A

Structural Analysis

X-Ray Diffraction (XRD) analysis of all samples show very similar results. There are no additional peaks observed showing the inclusion of Lithium within the structure, however this is comparable to results found in literature and confirms that the Nafion structure remained intact throughout chemical processing.

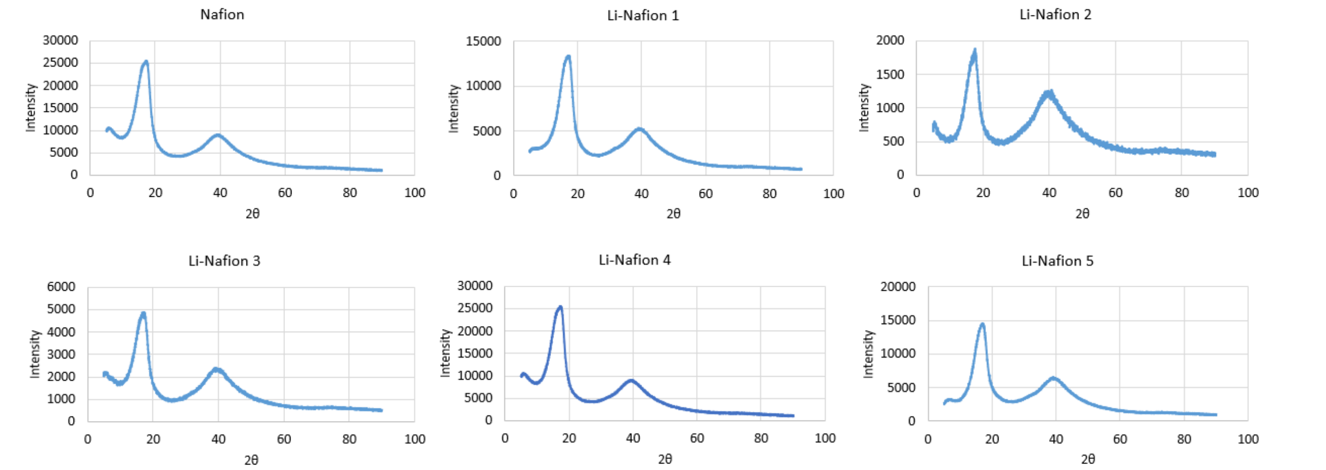

Figure 1 XRD Patterns

Mass Spectrum analysis of the surface of the Lithiated samples via SEM was able to confirm the inclusion of Lithium into the Nafion structure, with a high intensity Lithium peak observed in the spectrum as well as images of the surface. The precise membrane structure could not be established as the intensity of the beam resulted in a breakdown of the polymer structure.

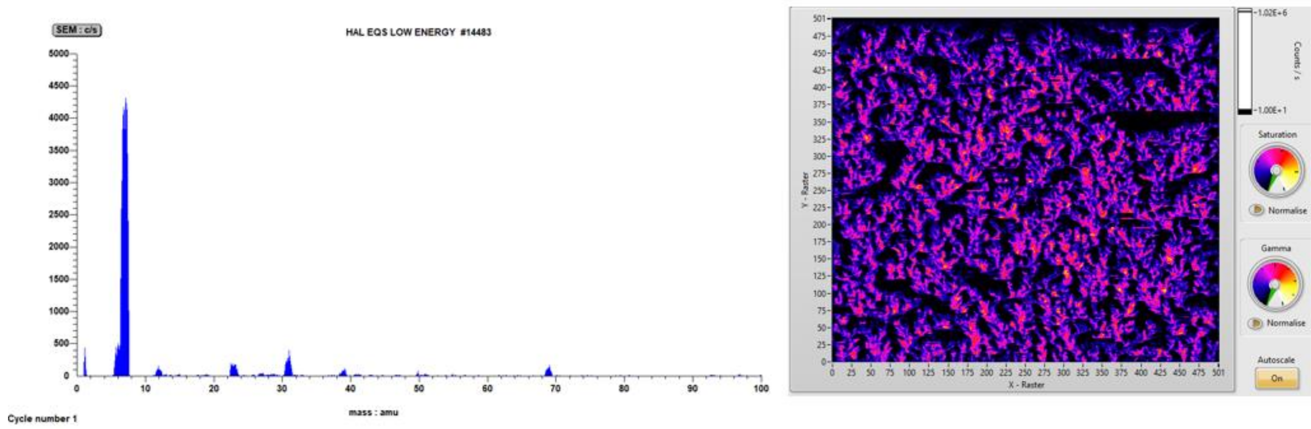

Figure 2 SEM Mass Spectrum.

<u>H-Cell Tests</u>

An electrochemical H-Cell was created (Figure 3) and used to test the membranes for Lithium-ion transport using pH measurements. One compartment of the cell contained deionised water whilst the other contained 1M LiOH. 2cm x 10cm Platinum plated Titanium mesh was used as the electrodes to apply 5V of potential from a power supply.

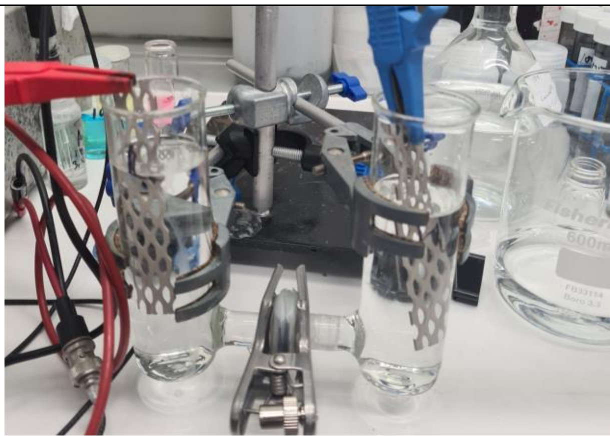

Figure 3 H-Cell set up

H-Cell experiments using both Nafion and Lithiated Nafion samples showed significant pH changes without the application of potential, with ion transport instead being directed by mass transport. A greater current was measured when applied to the Lithiated membrane suggesting that the lithiation process had improved ion conductivity through the membrane. It was not clear from the results whether the application of potential increased the rate of transfer or not. Figure 5 shows the results for FKL-PK-75 obtained from FuMATech which shows much greater promise for selective Lithium recovery as pH changes were not observed until the potential was applied. FKS-30 was unsuccessful as no current was able to pass through the membrane likely due to its much greater membrane thickness resulting in too much resistance.

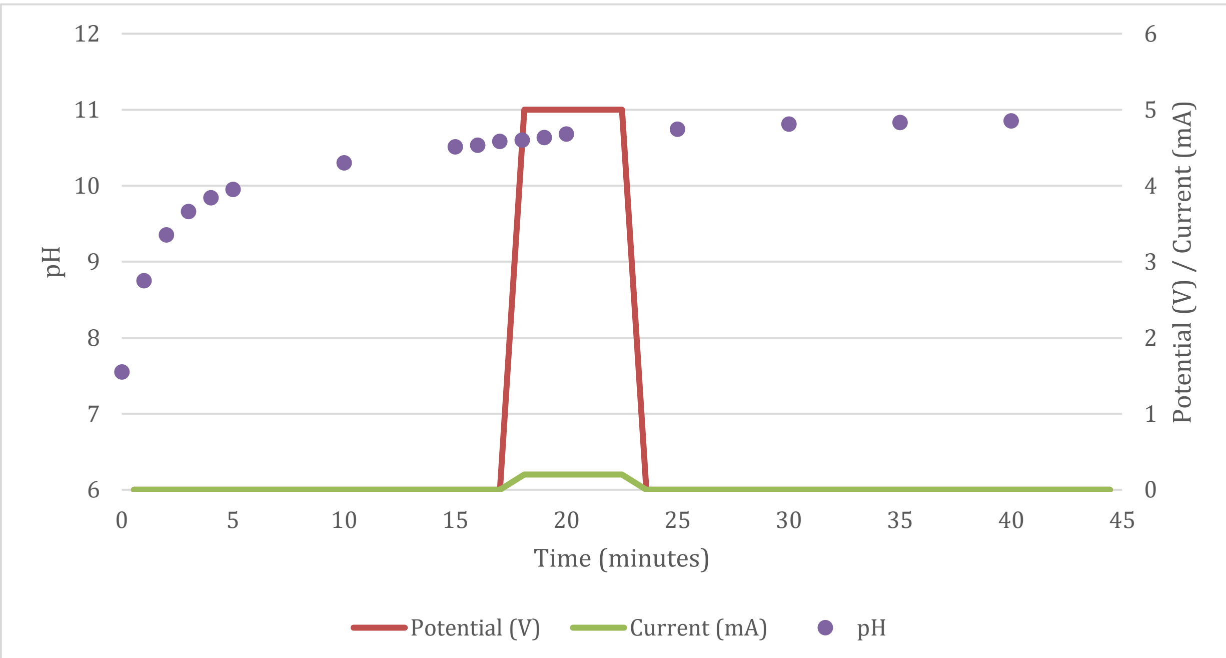

Figure 4 Change in pH over time after 1M LiOH was added to second compartment of H-cell with Lithiated Nafion membrane.

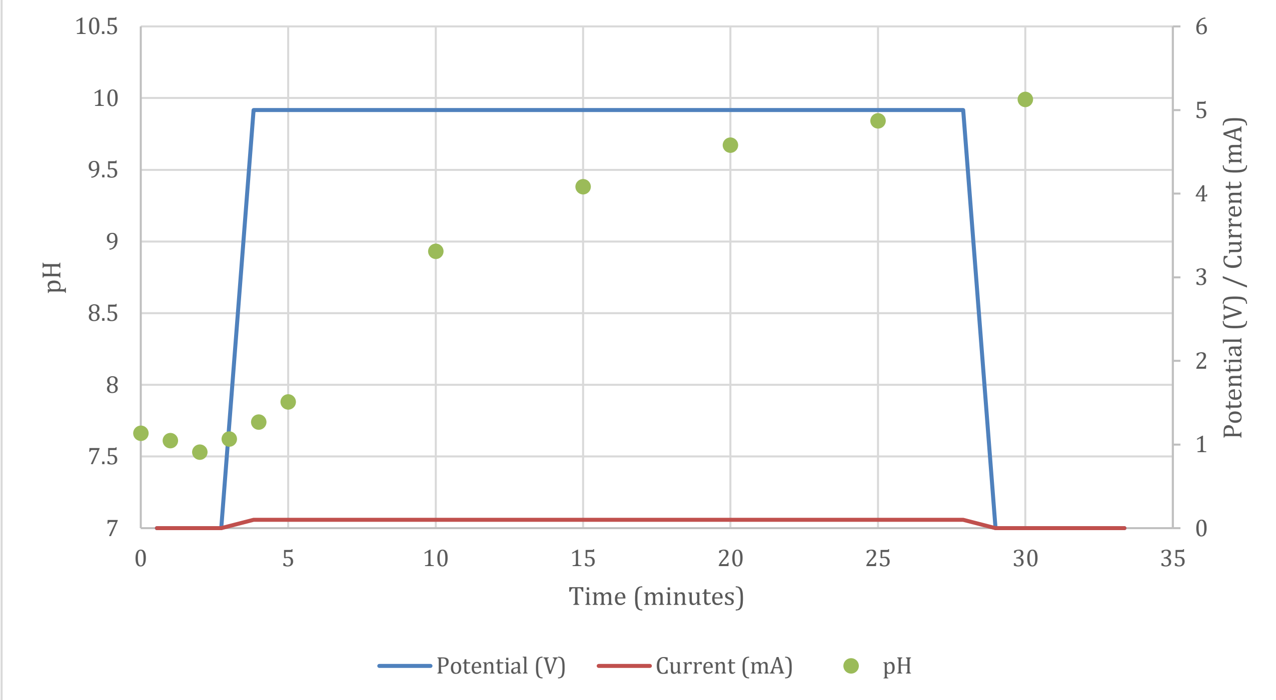

Figure 5 Change in pH over time after 1M LiOH was added to second compartment of H-cell with FKL-PK-75.

Flow cell

FKL-PK-75 was then trialled in a larger flow cell set up (Figure 7). The same Pt/Ti mesh electrodes as previously described were used, with a flow rate of 100ml/min for both the water and 1M LiOH solutions. This set up showed comparable results to the H-Cell showing that the membrane can be used with larger volumes of LiOH. After 90 minutes the pH of the solution was continuing to increase however the rate of ion transfer is still low, with only a 0.002M solution of LiOH (calculated from the pH) produced after 90 minutes. Further work to increase the rate of ion transfer would be required for this to be applied to a commercial recycling process.

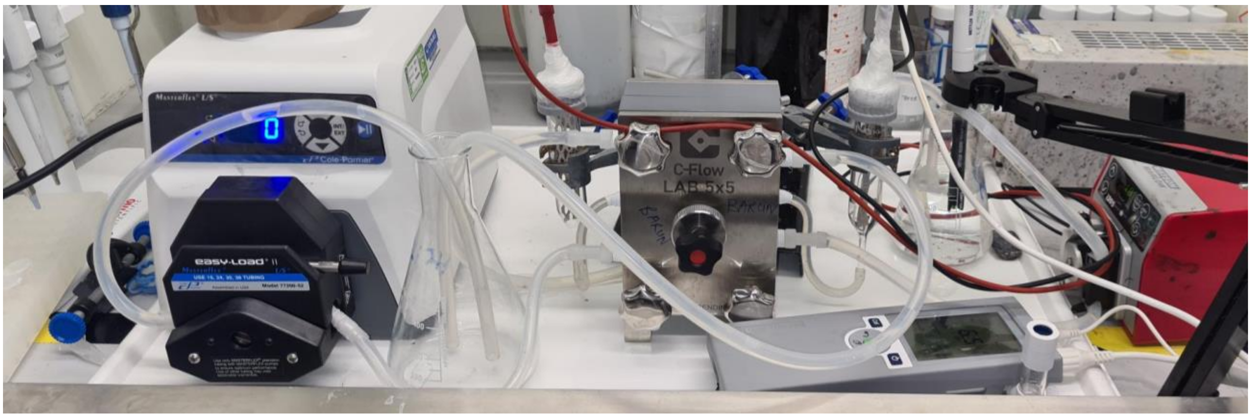

Figure 6 Flow-Cell

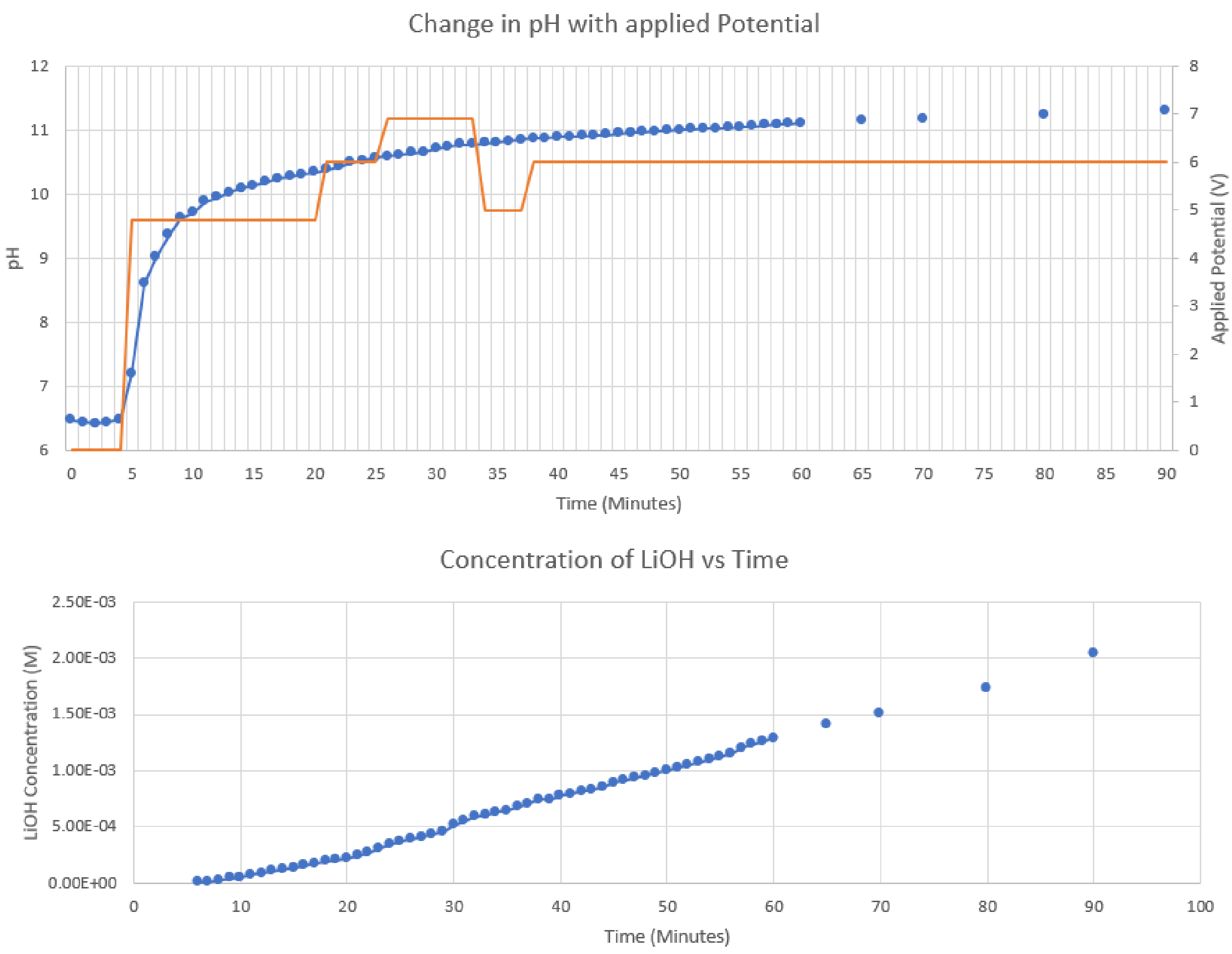

Figure 7 Above: pH change over time with applied Voltage for FKL-PK-75. Below: The concentration of LiOH in the water compartment at a given time, calculated from the measured pH.

Key finding 1 (max 50 words)

Lithiation of Nafion film can increase lithium-ion conductivity of the membrane allowing for greater lithium-ion transport. However, lithium ions could not be selectively transported with the application of a potential at the electrodes, evidence from the H-Cell investigation suggests that the lithium ions are able to pass through the membrane without the application of any potential and so these membranes would not be selective enough for purification.

Key finding 2 (max 50 words)

Commercial cation exchange membranes were able to transfer Lithium ions from a 1M LiOH solution to a water solution with the application of a potential. The membrane could be used in a larger scale flow cell to achieve comparable results, however the rate of lithium-ion transfer remained low and so further work to increase the rate of ion transport is required.

Testimonial from ECR/project team (max 500 words)

How has the project and involvement in CENTS impacted your skills and aspirations?

Lauren Copper, early career researcher in WMG, says: 'Undertaking this project has been very beneficial to me as I have gained a lot more experience in the design and set up of a complex experimental set up. I have been introduced to new methods of analysis for materials such as SEM and EIS and intend to do further learning and training to be able to use these in my future work. Going forward I am excited to continue to work on the design and testing of new battery recycling methods, this project has affirmed that this is a very exciting area of research with lots of areas of investigation to pursue'

Testimonial from Industry partner(s) (max 500 words)

We are continuing to communicate with various project partners, particularly European Metal Recycling (EMR) who are developing a battery recycling process and are very interested in this project. Further funding for the project has been secured from the RECOVAS project which is led by EMR and where Lauren will continue to work on this project and present her future results there. The platform gives us an avenue to scale it up in the future

Impact (max 200 words)

We will publish a literature survey in a relevant journal that will give an overview of the work carried put in this field.

The process we are developing can be used in the primary mining and secondary recycling industry for lithium purification. In both cases, the potential to reduce energy input and the ability to increase throughput is enormous.

Next steps (max 200 words)

Follow on funding has been achieved through APC RECOVAS project, which is seeing Lauren Copper working as a FTE researcher.

Survey of the literature has been completed, which is being prepared for journal submission.

Research challenges and opportunities with lithium-ion recovery process have been identified, which is being scoped for proposal preparation to funding agency (in discussion with partners).

*CENTS team will be in touch towards the completion of the CENTS project (end of 2022) to update the impact section

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