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Increasing the Efficiency of Lithium Ion Extraction Using the Electrochemical Method

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Abstract

With the electronics industry advancing rapidly, the demand for lithium-ion batteries is rising globally, creating a need for a reliable lithium recycling method. One such method is the electrochemical extraction of lithium from a source solution to a recovery solution using an ion exchange membrane and lithiated and de-lithiated LiMn_2O_4 -coated electrodes. This method passes an electric current through the electrodes which are submerged in 0.1 mol/L LiCl and 0.05 mol/L KCl solutions for two hours, which is then repeated after the electrodes are swapped between the solutions. Using this method, we have investigated the effects of varying levels of de-lithiation, as well as the method's lithium extraction capability and applicability to a real battery solution of $\text{LiNH}_4\text{OHSO}_4\text{Na}$. From our testing, both the KCl and the battery solution were found to extract lithium from the source solution, confirming the effectiveness of this electrochemical method.

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Background

With the electronics industry growing rapidly, the demand for Lithium-ion batteries as a power source is rising globally. Lithium-ion batteries are at the forefront of energy storage due to their high capacity and long lifespan compared to previous battery types (Scrosati, 2011). As a result, lithium-ion batteries have found a home in common devices such as laptop computers and cell phones, and they are currently being developed for further use in the automotive industry, which could prove to reshape personal transportation in the coming future (Scrosati & Garche, 2009). Lithium-ion batteries can be constructed using a number of different cathode materials, with one prominent material being lithium manganese oxide (LiMn_2O_4). LiMn_2O_4 exhibits high thermal stability while being at a relatively low cost and of a high abundance, which makes it a good candidate as a battery cathode material (Lee et al., 2014).

Considering the high demand for Lithium-ion batteries, there is an inherent need for a dependable, effective recycling method. Simply discarding these batteries is not environmentally friendly, since these batteries contain toxic chemicals that can leak into and contaminate the soil (Campion et al., 2004). Even more drastically, if these disposed lithium ion batteries are not completely drained, they can combust or explode if exposed to certain harsh conditions (“Lithium Battery Disposal,” n.d.; Ribière et al., 2012). On another hand, the sourcing of new lithium from natural deposits is not a very viable option either, considering how scarcely it is found in the world. There are no lithium deposits in North America, making Chile the closest natural source of lithium (Meshram, Pandey, & Mankhand, 2013; Zhou et al., 2017). Additionally, the price of lithium obtained from these sources is increasing yearly and is projected to keep increasing for the foreseeable future (Grosjean et al., 2012). Moreover, given the emergence of Lithium-ion batteries as a popular energy source in the past decade and the

approximated use life of these batteries being 10 years, a recycling method is becoming highly necessary (Scrosati & Garche, 2009).

One promising method for the recycling of lithium involves the electrochemical extraction of lithium from liquid solutions. This process involves the use of both a Li-containing Source solution, a recovery solution, a lithium cathode powder coated electrode, and a de-lithiated cathode powder coated electrode. In the interest of clearer lithium exchange results, the recovery solution would preferably not consist of any lithium before testing. In this system, the non-de-lithiated electrode is submerged in the recovery solution and the de-lithiated electrode is submerged in the source solution. An ion exchange membrane separates the two solutions. The electrochemical testing is done by applying a voltage to the system through the electrodes, which makes Li^+ ions transfer from the source solution to the recovery solution for a period of two hours. The electrodes are then swapped with the opposite electrode, and the voltage is applied to the system once again for the same amount of time (Zhou et al., 2017). A schematic of the electrochemical testing setup can be seen in Figure 1. After the testing, the lithium concentration of the source solution should decrease while the recovery solution's lithium concentration should increase, indicating that the voltage applied to the system successfully transferred lithium to the intended solution.

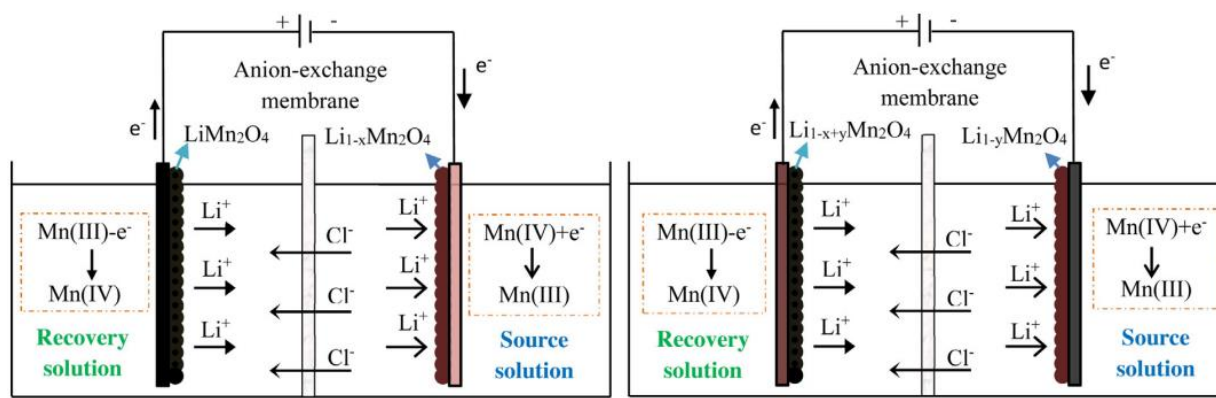


Figure 1: Schematic of an Electrochemical Method Test Setup (Zhou et al., 2017)

Methodology

Cathode Preparation

The cathodes used for the experiments were prepared by casting a lithium slurry on to a piece of carbon paper. Originally this slurry was a mixture of 0.35g of LiMn_2O_4 (for the positive electrode) or de-lithiated LiMn_2O_4 (for the negative electrode), 0.1g of PTFE to bind the lithium to the carbon paper, and 0.05g of conductive carbon black. An amount of ethanol was then added to make the mixture a slurry that could be cast on to the carbon paper. A casting tool, shown in Figure 4, was used to create an even spread of the slurry on the carbon paper that was laid on a sheet of glass to create a smooth surface to cast on. Once both sides of the electrodes were cast upon, they were dried in an oven. After the electrodes were dry, a silver conductive epoxy was used to attach pieces of nickel foil to the electrodes. This was to allow equipment to be attached and pass a voltage through the experiment. The PTFE was then switched out for a different binder, PVDF, due to its better bonding abilities. 0.1g of PVDF was used to substitute the PTFE to keep a 7-2-1 weight ratio for the slurry components. It was later decided to change this ratio to 8-1-1, where 0.40g of LiMn_2O_4 /de-lithiated LiMn_2O_4 , 0.05g (1mL) of PVDF binder, and 0.05g of conductive carbon black were used to create the electrode slurry. 1mL of 1-Methyl-2-pyrrolidinone were added to make the mixture more of a slurry to ease the casting process.



Figure 2: Mixture of LiMn_2O_4 and Conductive Carbon Black

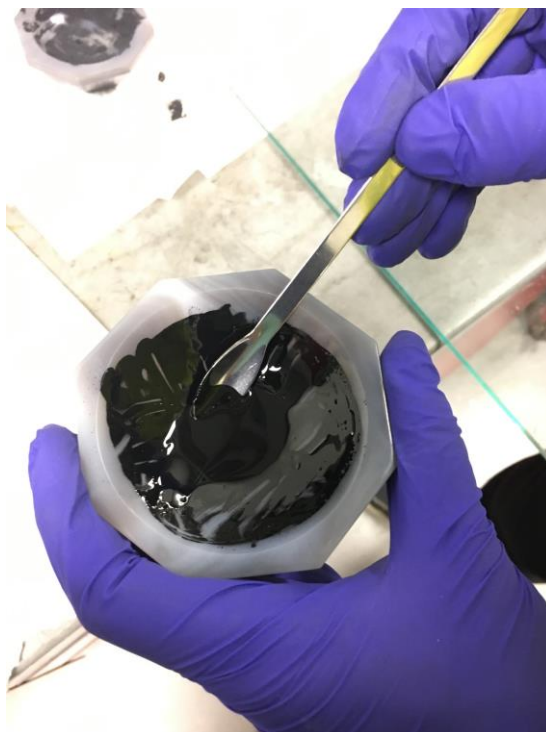


Figure 3: Mixture of LiMn_2O_4 , Conductive Carbon Black, and PVDF

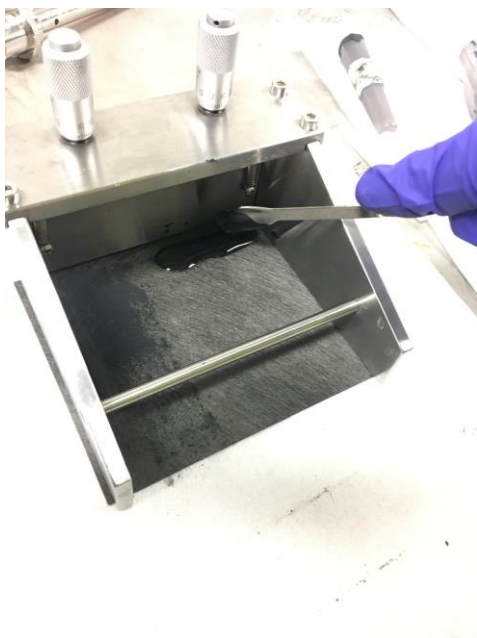


Figure 4: Tool Used to Cast the Slurry on to the Carbon Paper



Figure 5: Finished Electrodes with Nickel Foil Attached to the Top

Solution Preparation

Liquid solutions of 0.05mol/L KCl, 0.1 mol/L LiCl, and a battery solution were necessary to perform the electrochemical testing. To prepare the KCl solution, 1.8637g of KCl was

dissolved in 500mL of deionized water. The LiCl solution had 2.1195g of lithium chloride dissolved into 500mL of deionized water.

The battery solution was provided by Bin Chen and was prepared by the earlier steps of a different lithium-ion battery recycling process. This process is started by shredding the spent battery first, which results in a powder. After sieving, the remaining powder mainly consists of cathode powder, graphite, and some other metals, like Fe, Cu, and Al. This powder mixture is then leached with acid to remove the graphite and other undissolved substances. As a result, the solution changes to a transition metal sulfate. Ammonia water and sodium hydroxide is then added to deposit the Ni, Mn, and Co. The solution that is left is the battery solution used in the electrochemical testing, and contains Li, NH₄, OH, SO₄, and Na.

Experiment Setup and Testing Variables

Electrochemical testing was carried out using 250mL of the 0.1 mol/L LiCl liquid solutions as a source solution and 250mL of either the 0.05 mol/L KCl liquid solution or the battery solution as the recovery solution. Experiments were carried out with the positive LiMn₂O₄ cathode submerged in the recovery solution and with the negative Li_{1-x}Mn₂O₄ cathode submerged in the source solution, as seen in Figure 6. A voltage of 1V would be applied to the system for two hours using a BioLogic VMP3 potentiostat/galvanostat, with which a graph of the current over time would be recorded. Afterward, the cathodes were washed and dried to avoid mixing the contents of the separate solutions. The cathodes were then submerged in the opposite solutions, and 1V of electricity was applied to the system for another two hours. During both stages of the testing, a graph of the changing current over time was recorded digitally. This graph

was used both to verify the state of the reactions taking place and to calculate the capacity of the electrodes in mAh/g.

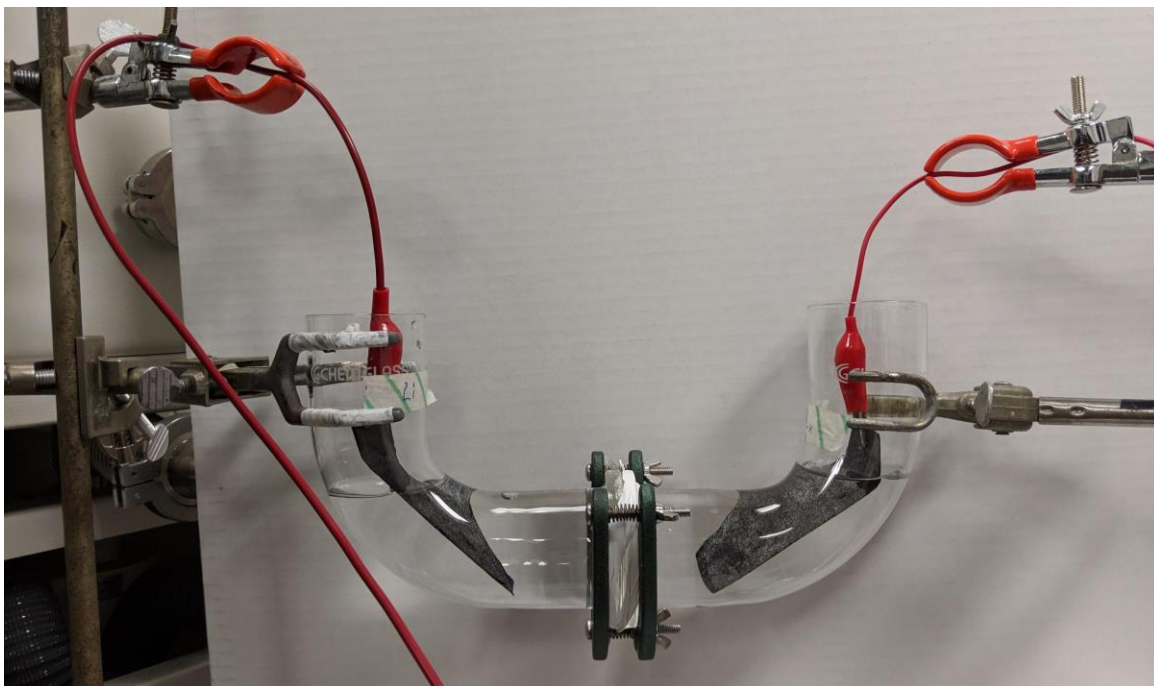


Figure 6: Electrochemical Test Setup Used for Experiments

De-Lithiation and XRD Testing

The de-lithiation process was carried out by first creating a mixture of 400mL DI water, 19.4g of sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$), and 4g of LiMn_2O_4 . After the complete dissolving of the sodium persulfate and LiMn_2O_4 in the DI water with a magnetic stirrer, the mixture was placed in an 80°C oven for either 3 hours or 6 hours. By heating the mixture for a longer period of time, more lithium comes out from the LiMn_2O_4 structure. After the heating, the mixture was then put through a pressurized filter, leaving only the resulting $\text{Li}_{1-x}\text{Mn}_2\text{O}_4$ powder. To verify the integrity of the structure, both the 3 hour and 6 hour de-lithiated powder were examined using an XRD. Both powders were found to be structurally intact relative to the original LiMn_2O_4 powder, meaning that they would be viable for electrochemical testing.



Figure 7: Compression Filter Setup for De-lithiation Process



Figure 8: De-lithiated $\text{Li}_{1-x}\text{Mn}_2\text{O}_4$ Powder (Left) and LiMn_2O_4 Powder (Right)

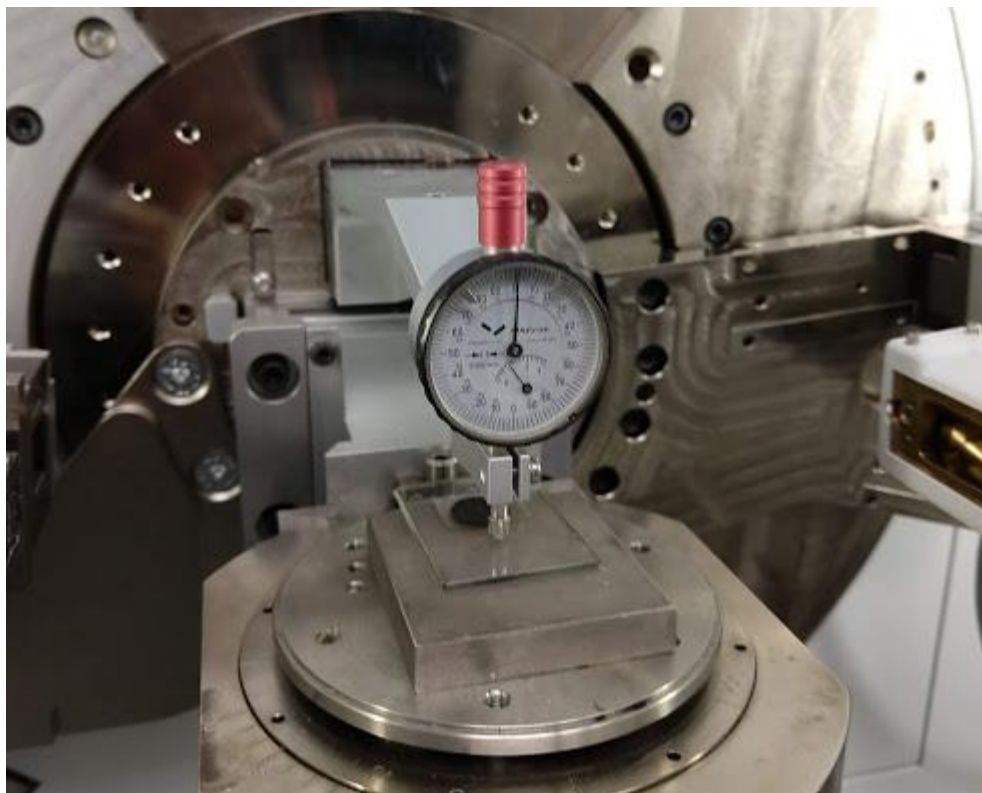


Figure 9: XRD Machine

Coin Cell Making

Coin cells were created similarly to how the electrodes were prepared. There were coin cells created using the original LiMn_2O_4 powder, the 3-hour de-lithiated LiMn_2O_4 powder, and the 6-hour de-lithiated LiMn_2O_4 powder to test the capacity of each. A lithium slurry was cast on the rough side of aluminum foil strips as shown below in Figure 10. Once an even coating was applied using the casting tool, the foil was dried in the oven. Once dry, a hole punch was used to create ten 14mm diameter cathodes coated in the slurry of each powder. Each cathode was weighed individually then dried in a vacuum oven. Once dry, the cathodes were moved into the vacuum glove-box so that they do not react with the moisture in the air. The cathodes were

placed into the shell of a coin cell and pressed shut. The capacity of these coin cells was found using the capacity testing machine shown in Figure 13.

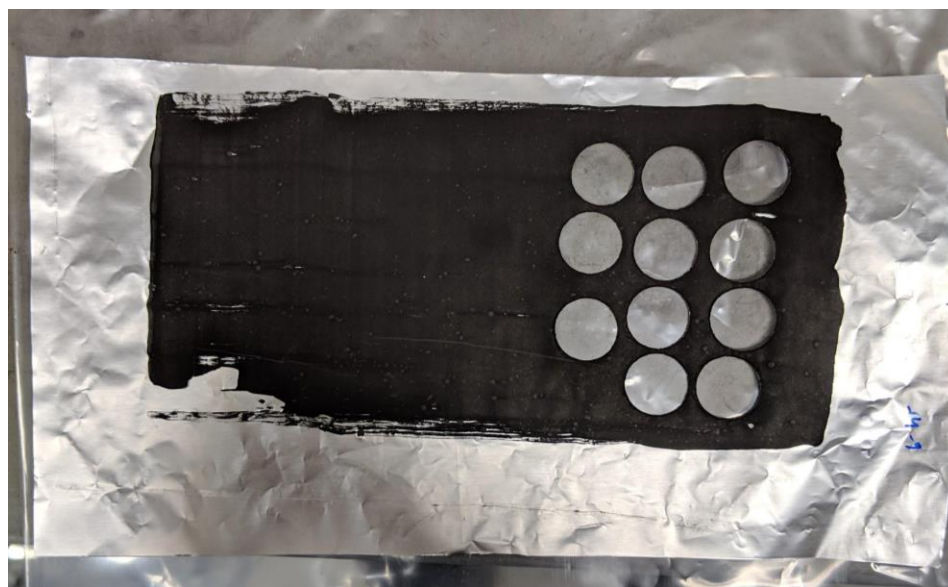


Figure 10: Lithium Slurry Cast on Aluminum Foil After Hole Punching



Figure 11: Vacuum Glove Box Used to Create Coin Cells



Figure 12: Coin Cell Made with LiMn_2O_4 Cathode

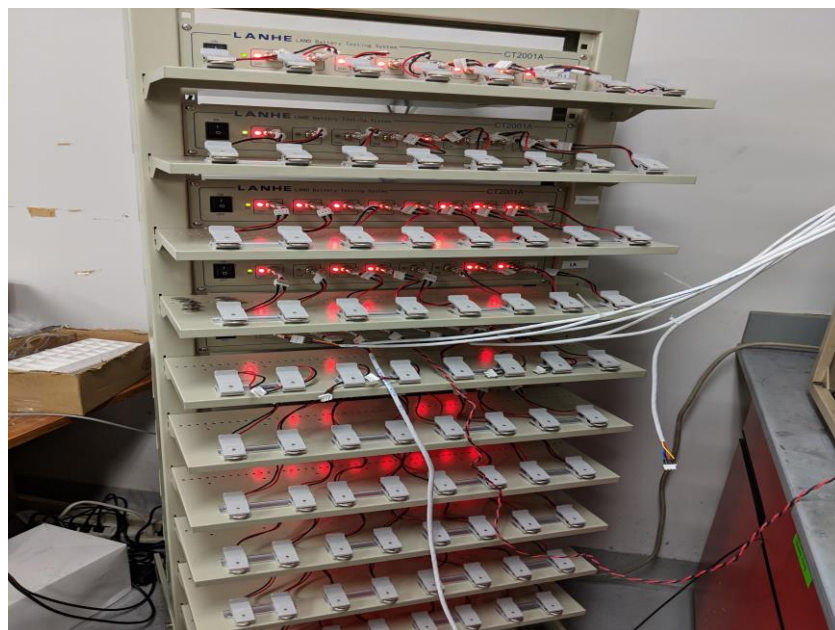


Figure 13: Capacity Testing Machine

ICP Testing

After the second two-hour cycle of the electrochemical experiment concluded, samples were taken of both the source solution (LiCl solution) and the recovery solution (KCl solution or the battery solution) for later ICP analysis. This would be compared with baseline samples taken

from before the beginning of the experiment. For the increased accuracy of the ICP results, samples had to have fit within a 20 - 200 parts per billion (ppb) range. Therefore, to fit within this range, all KCl solution samples were diluted 100x, all LiCl solution samples 10,000x, and all battery solution samples 50,000x. Results from ICP tests were given in terms of ppb concentrations and were analyzed as a difference between the original, untested solutions and the post-test solutions to examine the change in lithium concentration for each solution as a result of the electrochemical testing.

Results

Coin Cell Testing

The coin cells were tested in a charge/discharge apparatus to obtain a capacity value for each of the cathodes used: 3-hour de-lithiated, 6-hour de-lithiated, and original. As seen in Figures 14 and 16, both the original and 6-hour de-lithiated cathode coin cells displayed the expected charge/discharge behavior, while the 3-hour cathode coin cell did not. This was a result of the 3-hour cathode powder used for the coin cell testing not being successful and therefore having a significantly degraded structure. However, the 3-hour de-lithiated powder used in the electrochemical testing was from a different de-lithiation trial which was verified by XRD to be structurally intact. Nonetheless, from the charge/discharge testing, the capacity of the original and 6-hour cathode coin cell was found to be 106 and 83 mAh/g, respectively. These values would then later be compared to capacity values obtained from the current-time graphs obtained from the electrochemical testing.

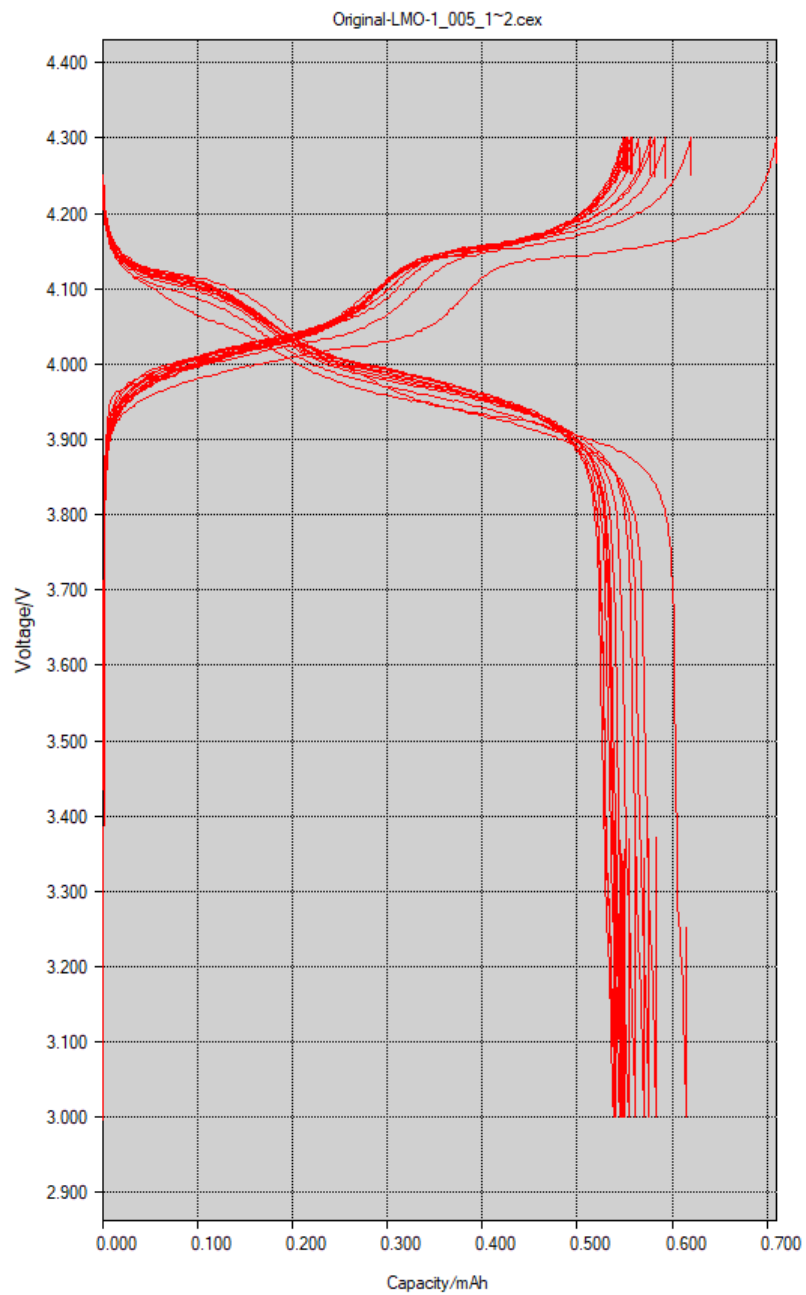


Figure 14: Charge/Discharge graph for LiMn_2O_4 Coin Cell

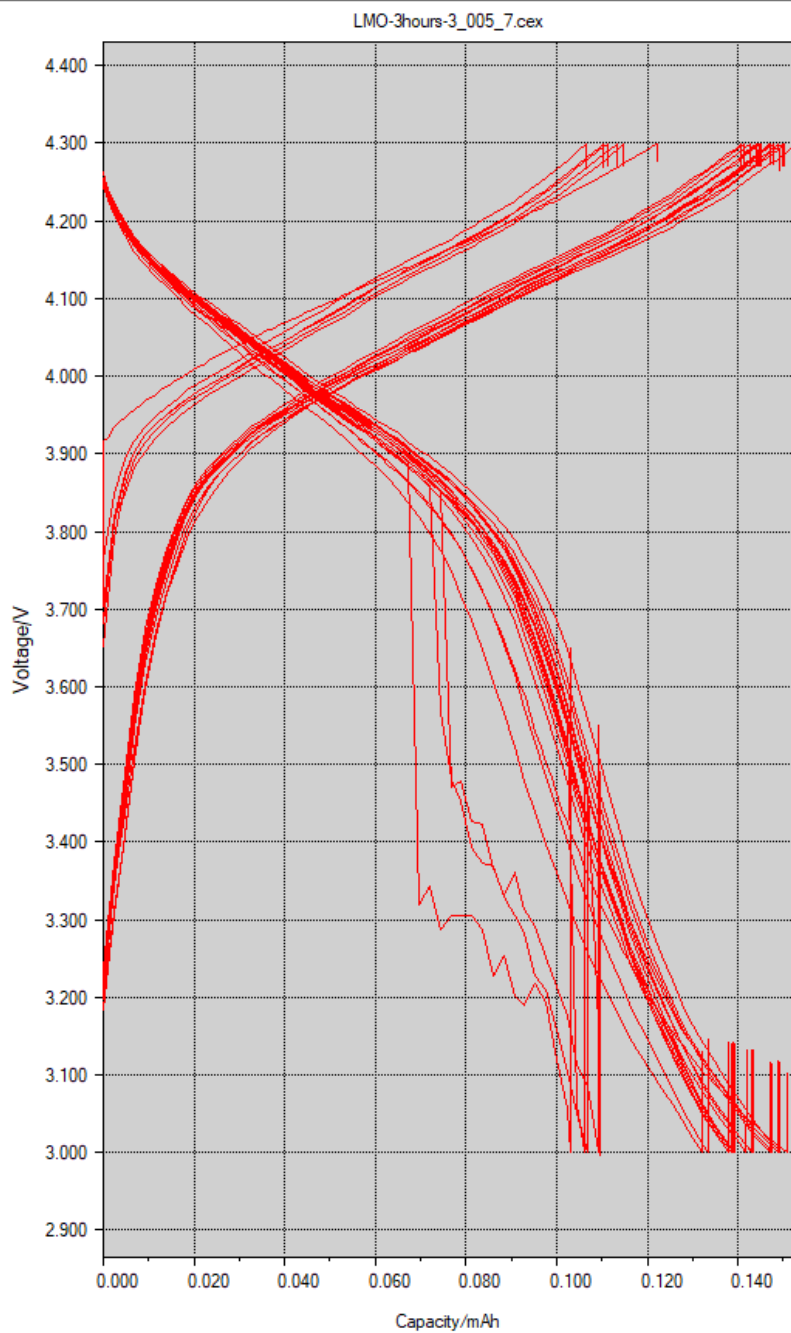


Figure 15: Charge/Discharge graph for 3-Hour De-Lithiated $\text{Li}_{1-x}\text{Mn}_2\text{O}_4$ Coin Cell

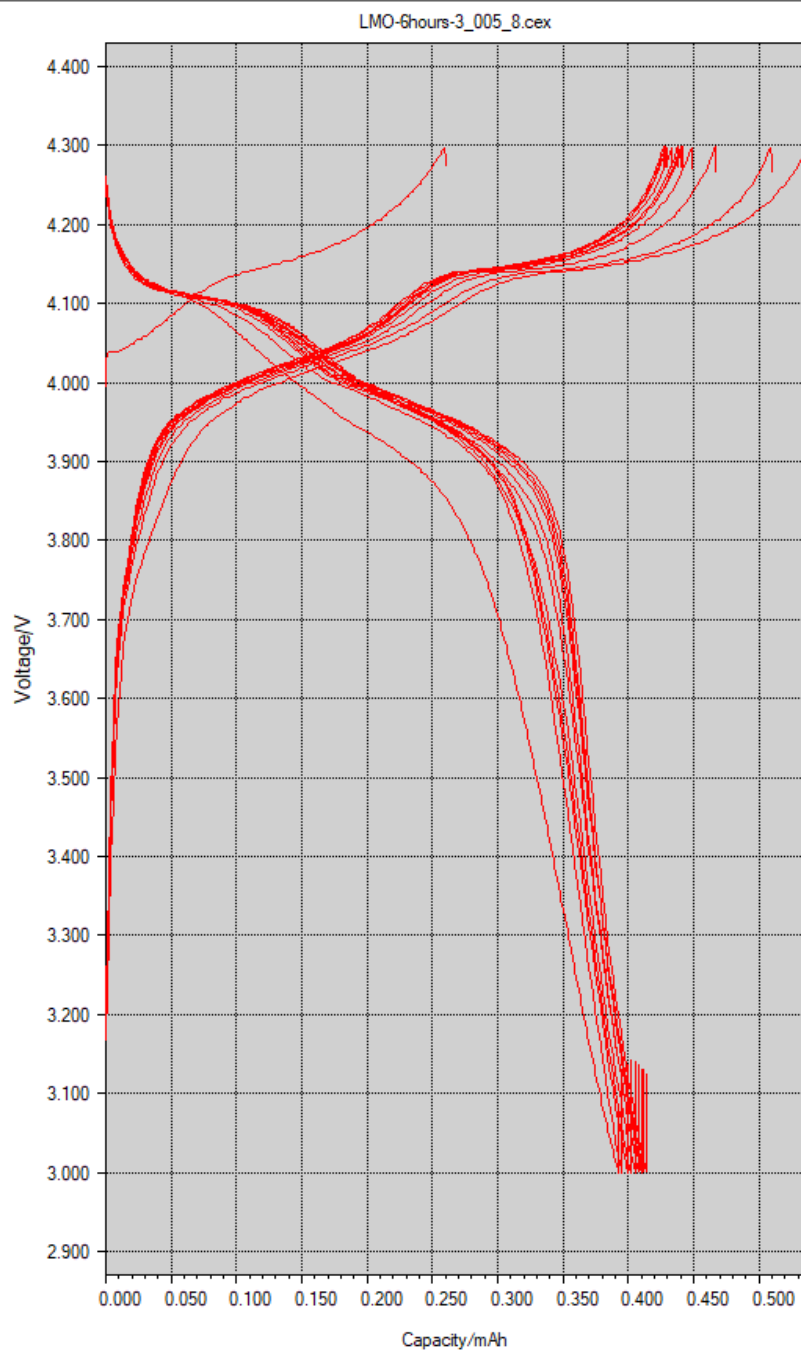


Figure 16: Charge/Discharge graph for 6-Hour De-Lithiated $\text{Li}_{1-x}\text{Mn}_2\text{O}_4$ Coin Cell

XRD Results of De-Lithiated Powders

Following the de-lithiation of the lithium manganese oxide powder, both the 3-hour powder and 6-hour powder were analyzed using an XRD to determine whether the de-lithiated structure was consistent with the original powder. Upon examination, both the 3-hour powder and the 6-hour powder were found to be consistent with the original lithium manganese oxide. As seen in Figure 17, the de-lithiated structure (D-LMO) has all of the same peaks as the original structure (O-LMO), just with a small phase shift that demonstrates the lesser lithium content of the de-lithiated structure. Since the structure was found to be consistent with the original, the de-lithiated powders were deemed usable for the later electrochemical testing.

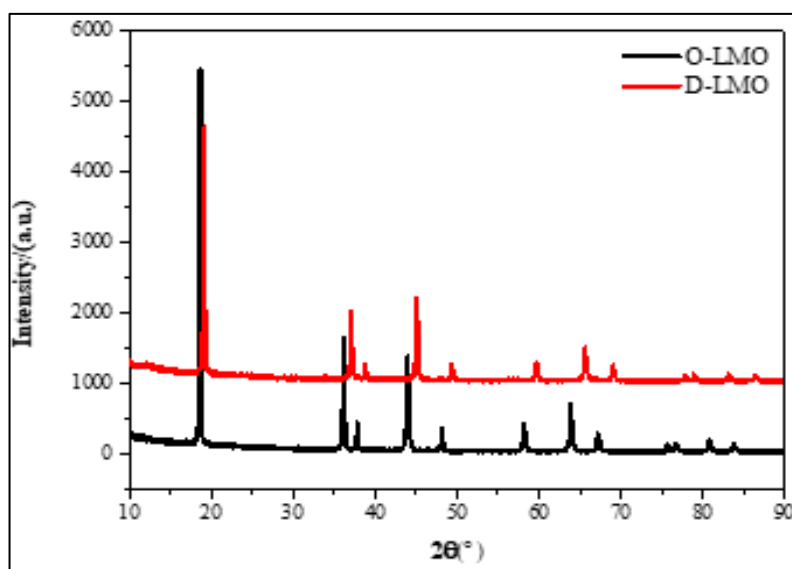


Figure 17: XRD Graph of Original and De-Lithiated LiMn_2O_4 Powder

Electrochemical Capacity Calculations

During each electrochemical test, current-time graphs were obtained over the course of both two-hour trials, which could be used for capacity calculations. Three of the most relevant current-time graphs can be seen in Figures 18, 19, and 20, which utilized combinations of the

KCl recovery solution, battery solution, and the 3-hour and 6-hour de-lithiated electrodes. All of these graphs come from the second two-hour trial of each of their tests. As each graph demonstrates, the current starts off at a relatively high value in mA, then steadily drops off to close to 0 by the end of the two hours. This indicates the large rate of reaction that happens initially as the voltage is applied to the system, followed by the decreased rate of reaction that occurs over the course of the rest of the time as the reactions and lithium transfer reach their limits.

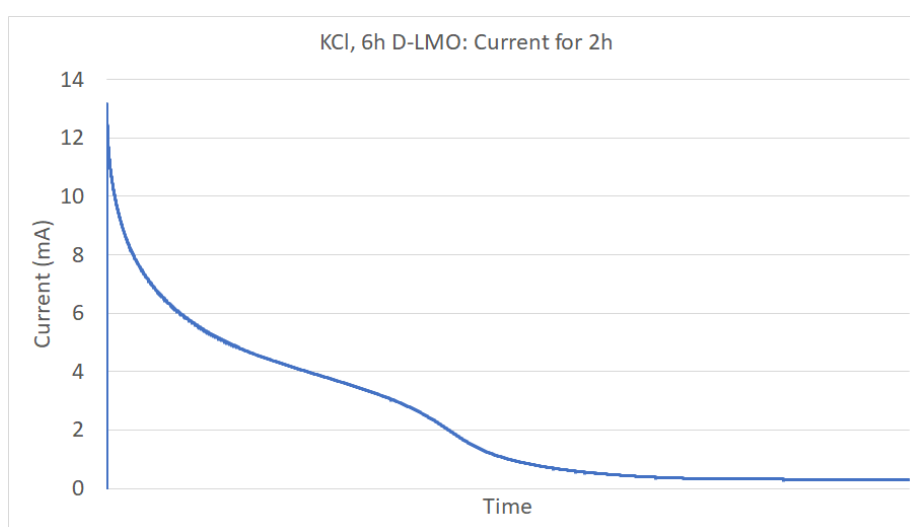


Figure 18: Current-Time Graph of Test Using KCl and 6-Hour De-Lithiated Powder

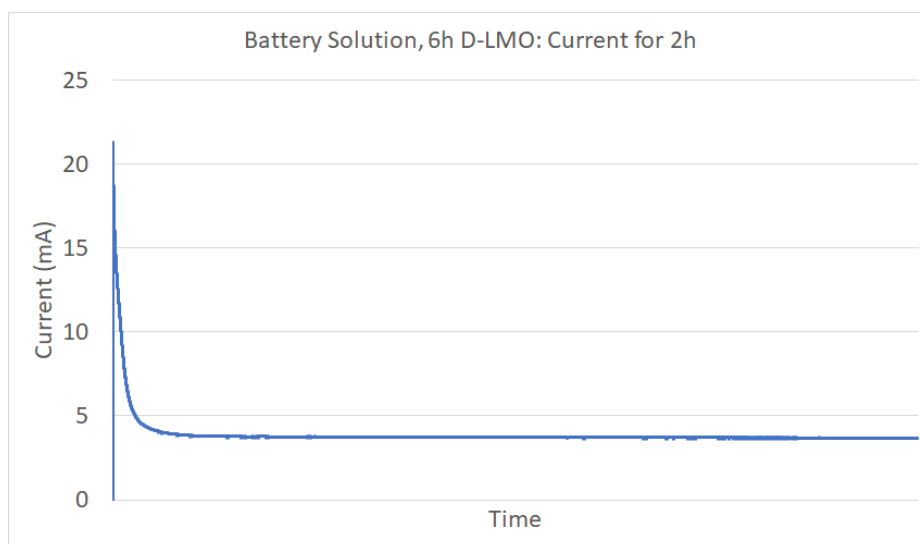


Figure 19: Current-Time Graph of Test Using Battery Solution and 6-Hour De-Lithiated Powder

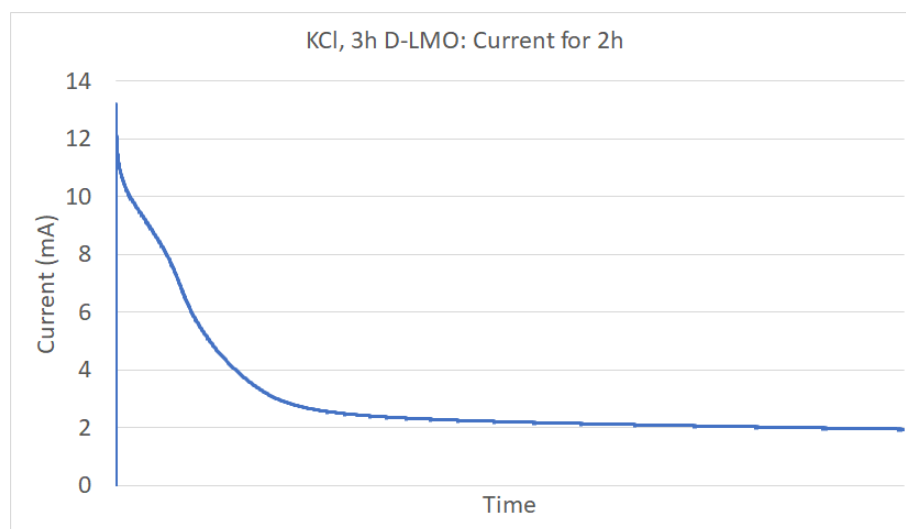


Figure 20: Current-Time Graph of Test Using KCl and 3-Hour De-Lithiated Powder

To obtain a capacity value from each of these three tests, the area under the graph was taken in mAh, which was then divided by the amount of active material cast onto the electrodes used in the test. To obtain a value for the amount of active material on the electrodes, each carbon paper electrode was weighed before and after the slurry casting process, and the difference of the masses (in grams) was then multiplied by 0.8, the proportion of lithium

manganese oxide in the slurry. The highest capacity was obtained from the KCl recovery solution with the 6-hour de-lithiated electrode, which was a value of 49.6 mAh/g. This test capacity was then compared to the results obtained from the coin cell tests, which serve as a theoretical or optimal value. A summary of this comparison can be found in Table 1.

Table 1: Test Capacity vs. Coin Cell Capacity

| Powder Type | Capacity (mAh/g) | Efficiency |
|--------------------|-------------------------|-------------------|
| Original | 106 | 47% |
| 6h de-lithiated | 83 | 60% |

Compared to the coin cell with the original cathode and the coin cell with the 6-hour de-lithiated cathode, the efficiency of the 49.6 mAh/g testing capacity is about 47% and 60%, respectively. This relatively low efficiency is likely a result of electrical impedance within the electrochemical testing setup or a result of degradation of the electrodes while being submerged in the solutions and subjected to hours of voltage.

ICP Results

To determine the amount of lithium transfer from the LiCl source solution to the KCl recovery solution or battery solution, samples taken from before and after the testing were analyzed by ICP. The results of the testing in terms can be found in Table 2.

Table 2: Results of ICP Testing

| Solution | Original (ppb) | Post-experiment (ppb) | Change |
|------------------|-----------------------|------------------------------|---------------|
| LiCl | 816ppb | 781ppb | -4.29% |
| KCl | 1ppb | 148ppb | N/A |
| Battery Solution | 2877ppb | 1858ppb | -35.41% |

As seen in Table 2, the electrochemical testing resulted in a 4.29% decrease in lithium content from the LiCl source solution, while the KCl recovery solution experienced a 147-ppb increase. Since the KCl solution should not have contained any lithium before the testing, which is reflected by the 1 ppb value, this difference is not given in terms of a percent change. For the battery solution, the lithium content was observed to have decreased by 35.41% despite it being used as a recovery solution alongside the LiCl source solution. A possible reason for this peculiarity is the presence of lithium within the battery solution. Since the battery solution already contained lithium before the electrochemical testing, it is difficult to obtain a definite amount of lithium transfer. It is also possible that lithium from the battery solution also transferred to the LiCl solution during the electrochemical testing.

Discussion, Conclusions, and Recommendations

As shown above in Figure 6, the apparatus that was used to carry out electrochemical testing was two identical L-shaped glass tubes joined by a clamp with an ion exchange membrane between them. One side of the apparatus was then filled with recovery solution and the other was filled with source solution. The electrodes had nickel foam attached to them with silver epoxy so that alligator clips could connect the electrodes to the equipment providing the current. The electrodes were then suspended in the solutions and a 1V current was applied. A number of variables that possibly altered the results could have arisen from this experiment setup.

The area where the two tubes were clamped together was also prone to leaking solution over time. This leak caused there to be less solution within the apparatus to transfer/receive lithium ions through the exchange membrane.

One possible point of interest is the ion exchange membrane that was clamped between the two halves of the apparatus. This membrane had a tendency to allow lithium ions to transfer between the halves even when a current was not present, which was proven by leaving LiCl and KCl in the apparatus for a week without any electrical current and covering both open ends of the tubes. The result of this was the lithium ion concentration of the KCl increased by 60.549 ppb, meaning that the membrane was not functioning correctly. If the membrane was not functioning correctly it means that the solutions could have been contaminated with excess ions during the testing process.

The electrical equipment that was used during electrochemical testing could have introduced some error into the data as well. This is because the connection between the

equipment and the electrodes was made using alligator clips, which could have been corroded or not fully connected to the nickel foil on the electrodes. By having a variable connection, the capacity and current of the experiment may have been taken incorrectly and thus given incorrect data.

Another possible source of contamination could be the silver epoxy used to bond the carbon paper to the nickel foil. This is because if the electrodes were placed too far into the solution or if the solution soaked up to the top of the electrode, where it would be level with the epoxy, it could lead to the solution becoming contaminated with ions from the conductive epoxy, thus giving inaccurate results from the ICP testing.

In between the first and second trials during a test, the electrodes are switched from one solution to the other. This switch can cause contamination as the electrodes have soaked up some of the solution from the first trial while they were submerged, and the solution can concentrate the other side with ions from the other. This can lead to inaccuracies in the ICP test results. To combat this, the electrodes were rinsed off with deionized water and then dried between trials.

When the LiMn_2O_4 powder was put through the de-lithiation process it was heated to 80°C for either three or six hours in order to remove the lithium ions from the powder. This may have caused degradation of the properties of the LiMn_2O_4 because it starts to degrade at temperatures above 60°C . This possible degradation could be the cause of the difference in theoretical and experimental results for the capacity of the electrodes.

A possible point of further experimentation concerns the slurry ratio used to create the electrodes. Although a 7:2:1 (cathode powder : binder : carbon) ratio was used in the early stages of testing, an 8:1:1 ratio was eventually the chosen ratio to include more of the active cathode material. This was done to increase the amount of reactions occurring during the electrochemical

testing, which was intended to increase the amount of lithium transfer. However, slurry ratios that include other proportions of the cathode powder could be tested to either increase the amount of active material on the electrode or to increase the amount of the slurry that is able to bind to the electrode, since a higher proportion of the binder increases the ability of the slurry to stick to the carbon paper.

Future Work

Overall, based on the ICP results, the electrochemical testing was successful in transferring lithium from the LiCl source solution to the KCl recovery solution. This demonstrates that this electrochemical system can work as intended to recover lithium from relatively simple solutions and can be effective on a small scale. However, since the application of this system to the battery solution yielded unfavorable results, more research and testing would need to be done to make this system effective for solutions with more complex chemistries. It is also important to note that since the battery solution already contained Li before testing, it was already not an ideal candidate for a recovery solution. Therefore, major changes to the experimental setup may be necessary to account for solutions such as the battery solution.

Alongside the ICP lithium transfer results, the efficiency of the test capacity was relatively low. This may have been due to electrical impedance from the experimental setup, considering that the electrical connections consisted of connecting leads from the potentiostat, alligator clips, nickel foil, conductive epoxy, and finally the electrodes themselves to get the voltage to the solutions. A more direct way to connect the potentiostat to the electrodes may improve the efficiency of the reactions. However, the capacity value obtained from the current-time graphs may inevitably be lower than the coin cell testing, considering that the electrodes

used in the electrochemical testing are submerged in liquid solutions and subjected to a voltage for a total of four hours. These testing factors may lead to the degradation of the electrodes over the course of the testing, which would likely lead to inefficiencies regarding the capacity calculations.

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