



Recycling of low value components using high purity pre-treatment, direct recycling and green hydrometallurgical approaches for recycling of Lithium Ion and Sodium Ion batteries.

D3.1 Requirements for hydrometallurgy

WP3 – Hydrometallurgy

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1. Introduction

Deliverable 3.1 focuses on chemical and physical characterization, including preliminary leaching test of feed material provided by WP1. WP1 works on the development of cost-effective pre-treatment process to produce high purity (high grade) material and low grade materials. High grade materials refer to pure separated cathode particle with trace anode particle and vice versa. High grade materials will be used as feed in direct recycling in WP2, while the low grade material will be hydrometallurgically processed in WP3. The characterization and preliminary leaching results will be used as input in the development of hydrometallurgical process and as feedback in separation and grading process performed by WP1. Hydrometallurgical works cover 3 tasks in WP3: T3.1 Lithium Extraction by Mild Hydrometallurgical Process, T3.2 Hydrometallurgical Recycling of Cathode Material and T3.3 Hydrometallurgical recycling of Na-Ion cathode material.

This report contains details of characterization of NMC black mass samples from partners within consortium and commercial black mass of Na-type battery, followed by preliminary leaching tests using different lixiviants. Based on the results, we propose hydrometallurgical flowsheet for inorganic leaching and subsequent hydrometallurgical operations for metal recovery.

2. Black mass characterization

2.1 Physical characterization

After the pre-treatment process in WP1, small batches of low grade NMC material were received in WP3 for characterization. In the project, it is planned to use this low grade material as input material for development of hydrometallurgical process. However, since such a hydrometallurgical process development requires high volume of material (> 1 kg scale) and the pre-treatment technology developed by WP1 is still in the up-scaling stage, we opted to use the black mass material provided by HYDRO ENERGI AS (HYDRO) to simulate the starting material for hydrometallurgical process development, namely NMC111, NMC622, NMC901. Their chemical characterizations being shown in Table 1. Based on chemical characterization results, it is estimated that the total Li and NMC content in HYDRO black mass is within 30.4-34%. In the case of impurities (Cu, Fe and Al), the content in HYDRO black mass is 2-3%. Impurities level is critical since it will add step(s) in separation and purification stage after the initial leaching step. Higher level of impurities in HYDRO black mass can be used as test case to determine the tolerance limit of impurities for hydrometallurgical process to be further developed. In the case of Na-battery, (Table 2) the starting material was obtained from commercial source, since end-of-life Na-ion batteries are not currently available in the market.

Table 1. Metal compositions on NMC black mass obtained from HYDRO

Black Mass	Weight (%)						
	Li	Ni	Mn	Co	Cu	Fe	Al
NMC 111	3.50 ± 0.21	10.92 ± 0.68	7.96 ± 0.52	9.49 ± 0.63	2.42 ± 0.23	0.11 ± 0.01	0.58 ± 0.04
NMC 622	3.49 ± 0.06	18.73 ± 0.51	6.10 ± 0.13	5.71 ± 0.21	2.08 ± 0.08	0.03 ± 0.00	0.81 ± 0.01
NMC 901	3.19 ± 0.02	24.03 ± 0.19	0.20 ± 0.00	3.02 ± 0.02	1.39 ± 0.04	0.01 ± 0.00	0.57 ± 0.00

Table 2. Metal composition in Na-type black mass, based on acid digestion and ICP-MS determination

Black Mass	Content (%)			
	Na	Mn	Fe	Ni
Na-black mass	20.7	15.2	14.4	16.3

2.2 Physical characterization

XRD characterization was done for all three types of black mass using Bruker D8 ADVANCE DaVinci at NTNU.. The XRD analysis indicates that the cathode active material for NMC 111, 622 and 901 appears, as $\text{LiNi}_{0.33}\text{Mn}_{0.33}\text{Co}_{0.33}\text{O}_2$, $\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$, and $\text{LiNi}_{0.806}\text{Mn}_{0.094}\text{Co}_{0.1}\text{O}_2$ respectively (Figure 1). SEM-EDS characterization of black mass was done using a Hitachi SU5000 Scanning Electron Probe provided scanning electron microscopy and energy-dispersive X-ray spectrometry (SEM-EDS) analysis by WCT. Figure 2 shows SEM image and elemental mapping of NMC622 black mass sample obtained by SEM-EDS. Distinctive features were observed differentiating the graphite components (C) from the nickel, manganese and cobalt in NMC as well as phases for aluminium. The details on SEM-EDS characterization of other black masses carried out by NTNU can be accessed in [Appendix SEM-EDS](#).

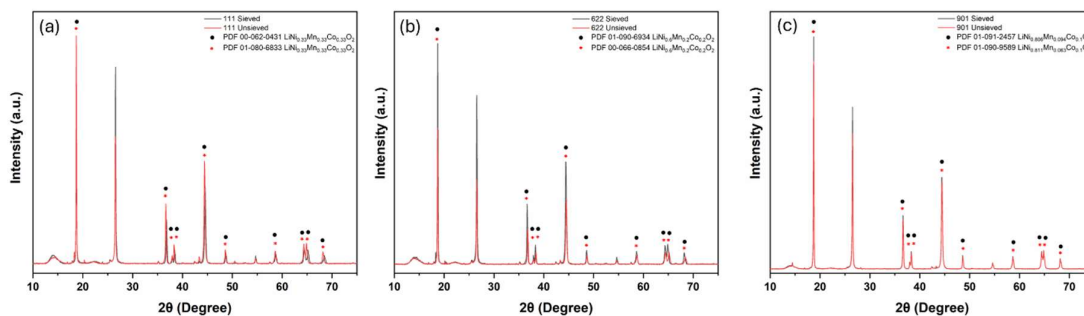


Figure 1 XRD pattern of BM (a) NMC 111 (b) NMC 622 and (c) NMC 901

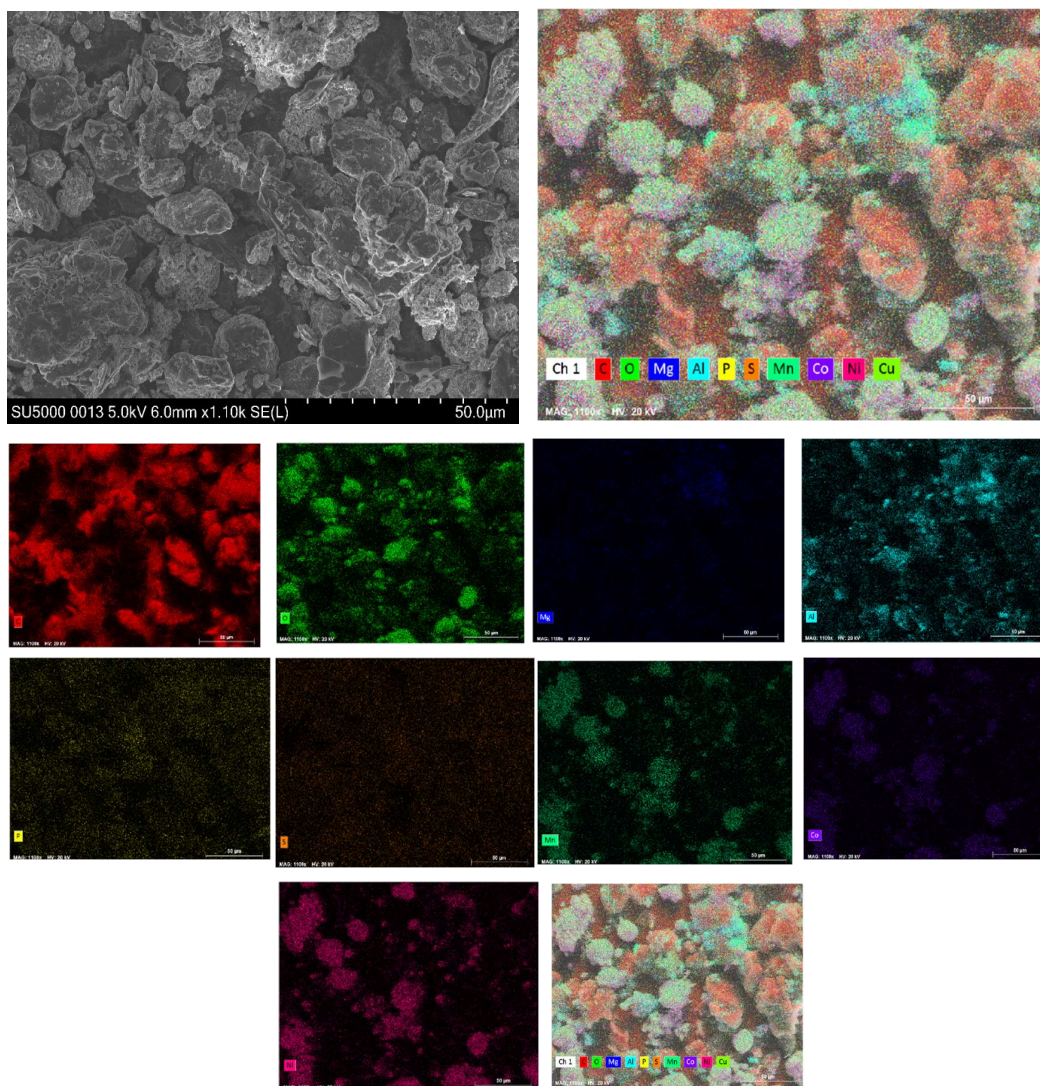


Figure 2. SEM image and EDS elemental mapping of NMC 622.

3. Preliminary leaching tests

3.1 Leaching test using Inorganic acid.

3.1.1 Effect of leaching parameters

Leaching tests were performed for all three black mass types using three kinds of inorganic acids: sulfuric acid, nitric acid, and hydrochloric acid. The detail protocol on inorganic leaching could be found in [leaching protocol](#). Metal leaching efficiency ($R, \%$) is calculated using equation (1), where C_m , V_l , w_m and c_m are total concentration of LiNMC in the leach solution, volume of leach solution, weight of black mass added and content of LiNMC in black mass, respectively.

$$R = \frac{C_m V_l}{w_m c_T} \times 100\%$$

Where;

$$C_m = C_{Li} + C_{Ni} + C_{Mn} + C_{Co}$$

$$c_m = c_{Li} + c_{Ni} + c_{Mn} + c_{Co}$$

The effect of leaching variables i.e. pulp density, temperature and acid concentration on LiNMC leaching efficiency in Table 3 It is generally shown that sulfuric acid has lower efficiency compared to nitric acid and hydrochloric acid (in terms of acid normality). Nitric acid and hydrochloric acid yields are almost twice of sulfuric acid recovery at the same leaching condition. The details on leaching test results can be accessed in [Appendix Inorganic Acid Leaching Test](#).

Table 3 Total Leaching efficiency for LiNMC based on different acid and black mass type

Pulp Density (g/L)	Temp (°C)	Acid Conc (M)	NMC111			NMC 622			NMC 901		
			H ₂ SO ₄	HNO ₃	HCl	H ₂ SO ₄	HNO ₃	HCl	H ₂ SO ₄	HNO ₃	HCl
100	75	1	58.95	54.97	53.99	60.45	41.25	52.37	75.69	58.98	64.56
250	25	1	37.78	21.60	22.06	37.67	16.69	18.22	43.41	21.14	33.82
100	50	3.5	41.34	53.89	91.80	56.22	45.09	98.99	88.20	66.43	110.17
250	75	1	38.00	24.55	22.18	44.66	16.71	20.94	62.59	22.19	27.82
175	75	3.5	61.76	73.33	85.61	81.98	65.56	99.45	130.77	100.88	101.62
250	25	6	46.95	48.79	66.99	47.04	66.61	60.52	69.00	24.47	96.85
175	50	1	54.81	31.22	30.96	44.78	30.84	30.57	61.76	39.22	21.08
250	50	3.5	66.46	59.89	59.99	55.62	43.62	48.21	32.29	25.34	56.53
100	25	6	56.51	73.60	76.70	25.27	40.44	86.44	30.47	70.14	55.17
175	50	6	73.67	75.91	95.07	39.83	47.25	76.52	84.77	92.77	54.27
175	25	3.5	49.72	64.83	60.00	21.73	39.33	46.90	62.86	62.08	39.96
100	75	6	110.34	93.37	106.76	42.24	80.94	89.68	114.80	113.89	109.70
250	75	6	90.26	94.15	93.16	37.04	81.25	97.03	112.93	122.54	109.21
100	25	1	50.58	38.85	40.55	17.62	36.90	36.58	55.72	24.67	42.53
175	50	3.5	17.50	61.57	75.94	28.23	69.27	25.05	71.36	69.30	97.16
175	50	3.5	27.79	61.89	73.41	6.97	50.00	51.46	75.56	69.03	102.64

3.1.2 Data analysis

To understand the influence of leaching variables (pulp density, temperature, acid concentration, and BM type) on total LiNMC leaching efficiency using different acids. Design of Experiments (DoE) was used and the results were analysed using multilinear regression statistical model. Table 4 list the combinations of input variables used in DoE for NMC111 leaching using HCl. Similar DOE was followed for NMC 622 and 901 while using HCl, HNO₃ and H₂SO₄ respectively. Details of DoE results for other black mass leaching experiments using different inorganic acids can be accessed in [Appendix Inorganic Acid Leaching Test](#).

Table 4. Design of experiments using HCl and NMC111

Test	Leaching Condition		
	Pulp density (g/L)	Temp. (°C)	Acid Concentration (M)
1	100	75	1
2	250	25	1
3	100	50	3.5
4	250	75	1
5	175	75	3.5
6	250	25	6
7	175	50	1
8	250	50	3.5
9	100	25	6
10	175	50	6
11	175	25	3.5
12	100	75	6
13	250	75	6
14	100	25	1
15	175	50	3.5
16	175	50	3.5

p-test was carried out on the dataset to evaluate the significance of each leaching variable on output total LiNMC leaching efficiency, while regression coefficients were used to evaluate the goodness of fits. A complete test analysis (144 experiments based on the DOE shown in Table 4) showed that all the leaching variables (pulp density, acid concentration, temperature, BM type and inorganic acid type), have a significant effect on total leaching efficiency of LiNMC (p-value < 0.05) as shown in Table 5, albeit a low regression coefficient of 0.57. Therefore, the predictive power of this multilinear model is not accurate within the studied design space. This could either be because of increase in the number of input variables (from 3 to 5) which the initial DOE did not consider or due to experimental/random errors arising from five involved personnel performing the experiments.

Table 5. RSq value of the model and P-Values of all the factors analysed

	P-Values				
RSq	Pulp density (g/L)	Temperature (°C)	Acid Concentration (M)	BM Type	Acid Type
0.57	0.002	0.000	0.000	0.000	0.030

In order to assess how individual acid has performed in total LiNMC leaching, we sorted the data into 3 subsets based on acid type, meaning that the model fitting was done considering 4 input variables. The significance of the input variables along with the regression coefficients are shown in Table 6.

Table 6. RSq value of the model and P-Values of factors analysed on Acid Based model

	RSq	P value			
		Pulp density (g/L)	Temperature (°C)	Acid Concentration (M)	BM Type
H ₂ SO ₄	0.53	0.556	0.000	0.031	0.000
HNO ₃	0.70	0.017	0.000	0.000	0.043
HCl	0.67	0.006	0.001	0.000	0.195

From Table 6 it was found that for H₂SO₄, pulp density is not a significant factor, where as for HCl, the BM type is not an important factor. This means that HCl will leach LiNMC efficiently regardless of the BM type/ composition, as represented by p-value > 0.05. Temperature and acid concentration are the two major factors which have statistical significance for all three acid types. From the data in Table 6, it can be observed that for HNO₃ model all the factors are significant and the model has a high regression coefficient of 0.70, whereas the RSq values for HCl and H₂SO₄ models are 0.67 and 0.53 respectively. It can be hypothesized that the Rsq value (0.57) for the model when all 5 factors were considered was less due to the contributions arising from low Rsq value (0.53) for the model in case of H₂SO₄, where 4 factors were considered.

We have also analysed the data based on the DOE shown in Table 4 where we made 9 models in total to understand the effect of variables (Pulp density (g/L), temperature (°C) and acid concentration (M)) and the p-values for the factors and regression coefficients for the models can be seen in Table 7. For the analysed data, we found that in case of H₂SO₄, pulp density is not a significant factor for all 3 types of BM while for NMC622, acid concentration is also not a significant factor. For NMC 111 and 622 models (in case of H₂SO₄), the Rsq values were 0.43 and 0.28 respectively, indicating that the models do not fit the experimental data accurately. This is, however, not the case for NMC 901 model (in case of H₂SO₄), which has a far higher RSq value of 0.58. This further explains why the previous models fitted with 5 parameters or 4 parameters (in case of H₂SO₄) also showed low Rsq values.

For HNO₃, it can be observed that all the 3 models have a high RSq > 0.65. Analysed data shows that the pulp density is the least significant factor in all the black mass models for HNO₃ while temperature can be considered as non-significant for HNO₃ - 622 model.

For HCl NMC 111 model, it was concluded that all the factors have a significant role in influencing total LiNMC leaching, whereas in HCl NMC 622, acid concentration has the highest significance, followed by pulp density (p-value 0.052) and temperature (p-value 0.068). For HCl 901 model, acid concentration is the most significant factor while pulp density and temperature are insignificant based on p-values. Further, this model has a low RSq value (0.50) as compared to higher RSq values for NMC 111 (0.88) and NMC 622 models (0.71).

Based on the above analysis, it may be safely inferred that the low Rsq values in case of H₂SO₄ models stems from experimental errors rather than how the DOE has been customized for our experimental matrix.

Table 7. RSq value of the model and P-Values of factors analysed on Acid and BM based model

	RSq	P-Values		
		Pulp density (g/L)	Temperature (°C)	Acid Concentration (M)
H ₂ SO ₄ NMC 111	0.43	0.542	0.077	0.043
H ₂ SO ₄ NMC 622	0.28	0.720	0.055	0.807
H ₂ SO ₄ NC 901	0.58	0.504	0.003	0.011
HNO ₃ NMC 111	0.83	0.058	0.012	0.000
HNO ₃ NMC 622	0.65	0.644	0.062	0.001
HNO ₃ NMC 901	0.75	0.069	0.003	0.001
HCl NMC 111	0.88	0.007	0.012	0.000
HCl NMC 622	0.71	0.052	0.068	0.001
HCl NMC 901	0.5	0.494	0.104	0.014

To validate the performance of our statistical models and to find the optimum conditions predicted by the models to achieve highest total LiNMC leaching efficiency, we performed three experiments using HCl as the acid of choice and these results are summarized in Table 8. The results show that all the models can well predict the total LiNMC leaching efficiency, except for NMC 901, which may be attributed to low Rsq value (0.50) for this model compared to the other two..50).

Table 8 Validation leaching results of three black mass using HCl, pulp density in g/L, temperature in celcius and acid concentration in M.

Optimum Leaching condition	LiNMC	
	Predicted	Actual
NMC 111, Pulp Density 166, Temp 70, Conc. 6	101.7	96.1
NMC 622, Pulp Density 250, Temp 75, Conc. 6	83.0	88.1
NMC 901, Pulp Density 249, Temp 58, Conc. 4.7	80.0	110.7

3.1.3 Impurities dissolution during leaching

Table 9 lists the impurities concentration in leach solution at optimum leaching conditions and its ratio to total target metals (LiNMC). In the case of NMC 111, the impurities constitutes of 18% of all metals in the leach solution (impurities level), while NMC 622 and NMC 901 are 8.15 and 7.14% respectively. The impurities i.e. Cu, Fe and Al hypothetically can be removed from the stream using solvent extraction or precipitation method to purify target metal Li and NMC in leach solution. The impurities concentrations is within 0.75-5.39 g/L which is suitable concentrations for solvent extraction method. As reference, previous research on hydrometallurgical flowsheet development done at NTNU for battery recycling could successfully handle impurities level 2.46%. The level of impurities in black mass sample in current studies is Cu < 4%, Fe < 0.1% and Al < 1% (total < 5.1%). Sieving can be recommended to separate and decrease the impurities level as shown in Table 10. It is demonstrated that impurities tend to concentrate at coarser fraction.

Table 9 Impurities concentration in leach solutions obtained at optimum leaching conditions and its ratio to target metals.

Leaching condition	Impurities (g/L)			Total Target Metals (g/L)	Impurities/Total Metals (%)
	Cu	Fe	Al		
NMC 111, Pulp Density 166 g/L, Temp 70 °C, Conc. 6 M	5.39	3.24	1.47	53.38	18.92
NMC 622, Pulp Density 250 g/L, Temp 75 °C, Conc. 6 M	2.94	1.96	1.77	81.88	8.15
NMC 901, Pulp Density 249 g/L, Temp 58 °C, Conc. 4.7 M	1.89	3.22	0.75	82.09	7.14

Table 10 Effect of sieving on metal content distribution for NMC111

Fraction	Content (%)						
	Li	Ni	Mn	Co	Cu	Fe	Al
Unsieved	3.50±0.21	10.92±0.68	7.96±0.52	9.49±0.63	2.42±0.23	0.11±0.01	0.58±0.04
>106 micron	3.09±0.05	9.54±0.20	7.17±0.14	8.51±0.15	12.57±0.60	0.24±0.01	3.69±0.36
<106 micron	3.59±0.06	10.92±0.13	8.24±0.20	9.75±0.11	1.24±0.00	0.09±0.00	0.16±0.00

3.2 Leaching test using organic acid

The use of three organic acids was explored for the leaching of black mass NMC 622. Initial tests were performed with acetic acid, oxalic acid, and citric acid experimental procedures for which are listed in Table 11. Samples of 5mL were extracted using a syringe and the samples were filtered with 0.22µm PVDF filters prior to analysis by Turbospec e400 benchtop NMR and ICP-MS. The experimental results for leaching of NMC622 with 1.5 M of acetic, citric and oxalic acid can be seen in Table 2. These data represent the absolute amount of cations in the original black mass and in the leach solution after 2 and 4 hours of leaching experiment for acid concentrations of 1.5M at 80°C.

Table 11 Key experimental details of organic acid leaching

Leaching Agents	Concentration and Temperature:	Solid/Liquid ratio
Acetic, oxalic, and citric acid are good organic acids for metal leaching. Each has distinct properties: 1. Acetic acid is a weaker acid, often used as a selective leaching agent. 2. Oxalic acid can form metal oxalates, particularly for lithium and cobalt, which may impact downstream processing. 3. Citric acid is known for its chelating ability, which can aid in extracting transition metals like cobalt and nickel.	Using 1.5M concentration at 80°C is a reasonable approach for maximizing metal recovery without overly aggressive leaching. Higher temperatures accelerate dissolution, though they can also lead to unwanted side reactions or impurities dissolving alongside target metals.	20 g/L S/L ratio and 2 g of black mass

Table 12 Absolute mass of cations for leaching of NMC 622 using acetic acid, citric acid and oxalic acid

	Li	Ni	Mn	Co	Cu	Fe	Al
1.5 M Acetic Acid							
Conc in leaching t=0h (%)	0.00	0.00	0.00	0.00	0.02	5.61	1.49
Conc in leaching t=2h (%)	37.38	24.47	29.32	24.96	105.02	54.24	95.79
Conc in leaching t=4h (%)	42.66	26.66	32.45	27.65	114.87	81.95	136.34
1.5 M Citric Acid							
Conc in leaching t=0h (%)	0.00	0.00	0.01	0.00	0.01	11.08	0.73
Conc in leaching t=2h (%)	66.82	71.65	86.27	71.31	43.77	135.24	76.40
Conc in leaching t=4h (%)	71.46	77.93	95.22	78.19	67.57	147.79	95.92
1.5 M Oxalic Acid							
Conc in leaching t=0h (%)	0.01	0.01	0.00	0.00	0.00	12.00	1.94
Conc in leaching t=2h (%)	56.60	0.46	14.15	0.85	6.60	164.74	114.43
Conc in leaching t=4h (%)	63.85	0.25	17.93	0.75	12.92	188.05	126.05

The metal recoveries (calculated using equation 1) after 4 hours for acid concentrations of 1.5M at 80°C are reported in Figure 4. Acetic acid showed the lowest leaching efficiency with metal recoveries <43% for nickel, manganese, cobalt and lithium. The poor leaching efficacy of acetic acid is attributed to its high pKa of 4.76 making it a weak acid. The highest leaching efficiency was measured for citric acid with primary constituents nickel, manganese, cobalt and lithium reported at 78%, 96%, 78% and 72%, respectively. Aluminium recovery was 100% for acetic acid and 96% for citric acid. Citric acid, with a pKa of 3.1, has a higher acidity than acetic acid, resulting in enhanced leaching efficiency for black mass. Oxalic acid reported a lower lithium recovery of 64%, manganese recovery of 18%, copper recovery of 13% of Al. Oxalic acid showed no leaching capability for nickel and cobalt.

The lowest pKa of 1.3, is associated with oxalic acid, which signifies that it is more acidic than citric acid, despite not all components in the black mass being extracted. Oxalic acid depicted selective leaching capabilities for lithium. The difference in solubility limit between lithium oxalate (6.6g/L at 18°C) and other metal oxalates favoured the separation of lithium from other metals in NMC 622.

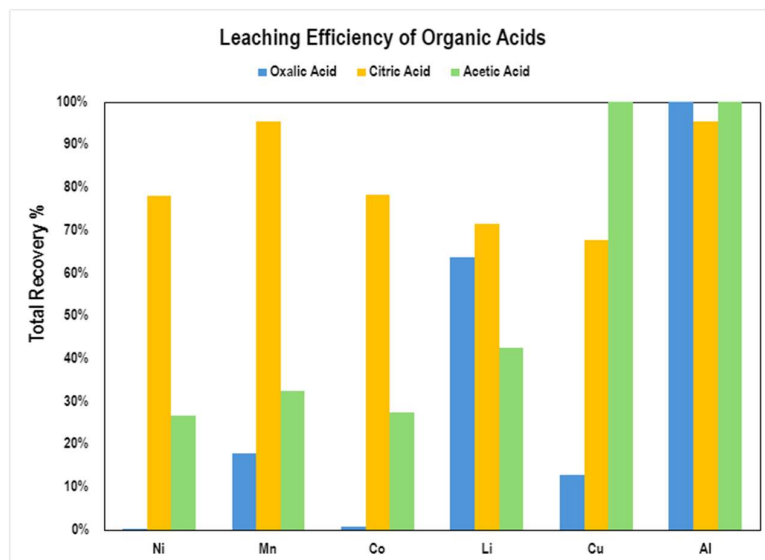


Figure 4 Leaching efficiency of 1.5 M acetic, oxalic and citric acid for NMC 622 after 4 hours at 80°C, S/L of 20g/L

Further experiments were conducted to determine the effect of decreasing oxalic acid concentration on NMC 622 leaching. The absolute amount of ions and the recovery data for these experiments are shown in Table 13 and Figure 5. Increased leaching efficiency was observed with reduced oxalic acid concentration for lithium. Reducing the molar concentration from 1.5M to 0.5M improved lithium recovery from 57% to 60%, respectively, whilst metal recoveries for competing cations decreased. The leaching efficiency decreased from 100% to 93% for aluminium, from 14% to 10% for manganese and from 7% to 5% for copper.

Table 13 Absolute mass of cations for leaching of NMC622 with 0.5M oxalic acid at 80 C	0.5 M Oxalic Acid						
	Li	Ni	Mn	Co	Cu	Fe	Al
Conc in leaching t=0h (%)	0.00	0.01	0.00	0.00	0.00	5.71	0.47
Conc in leaching t=2h (%)	59.62	0.43	10.00	0.69	5.47	117.74	93.34

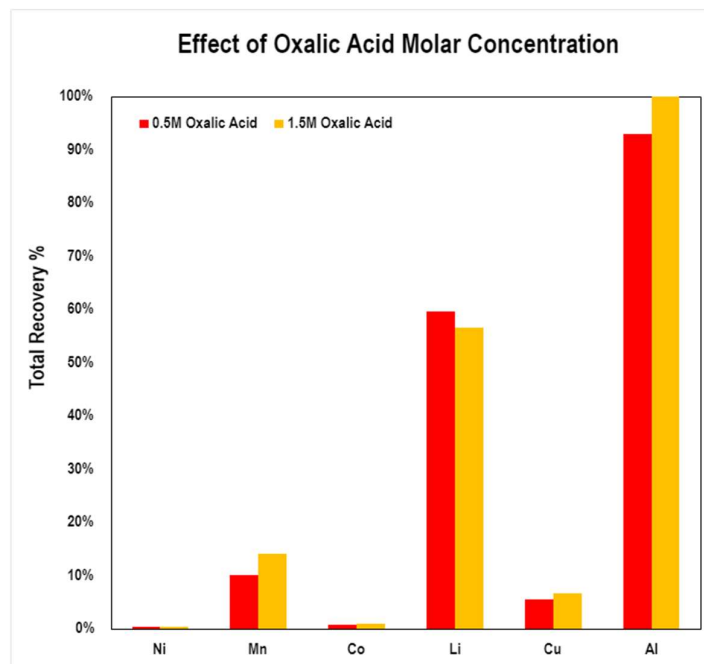


Figure 6 Leaching efficiency of oxalic acid at decreased molar concentration with S/L 20g/L at 80°C for two hours

3.3 Leaching test of Na-type battery

In order to determine the best leaching agents, a first screening was developed. The leaching agents selected provided excellent extraction yields, on LIBs chemistries. This information can be found on previous EURECAT project reports and in literature. The ones selected are summarised in the following Table 14:

Table 14 Leaching agents selected for the screening tests

1	Citric Acid
2	P-Toluene Sulfonic Acid (PTSA)
3	Deep Eutectic Solvent based on Choline chloride and P-Toluene Sulfonic Acid (DES ₃ PTSA:H ₂ O:ChCl)
4	Deep Eutectic Solvent based on Choline chloride, Lactic Acid and Ascorbic Acid (DES ₄ ChCl:LA:AA)
5	Sulfuric acid

The protocol regarding the methodology and experimental procedure can be found on the leaching reagents screening [protocol](#). The first leaching results are summarised in Table 15.



Figure 7. Thermomixer used on the leaching experiments.

Table 15. Metal leaching efficiency

Sample	Na (%)	Mn (%)	Fe (%)	Ni (%)
Citric Acid	94	100	98	91
PTSA	95	58	84	90
DES ₃ PTSA:H ₂ O:ChCl	41	85	86	85
DES ₄ ChCl:LA:AA	85	85	86	81
Sulfuric acid	85	1	83	92

Citric acid is the reagent that extracts almost all the metals in the CAM, achieving almost complete dissolution. Politholuene sulfonic acid, achieved similar extraction yields for sodium and nickel, but lower in the case of iron and manganese. The deep eutectic solved based on PTSA, achieved the lower results, and the DES based on organic acids, was also not better. Finally sulfuric acid obtained the best results in nickel extraction but wasno so good in iron and sodium extraction, and it was particularly bad for manganese recovery.

4. Recommendations

4.1 Inorganic acid leaching

Based on the inorganic acid leaching results including its statistical analysis, hydrochloric acid based flowsheet is proposed for hydrometallurgical process of battery recycling as shown in Figure 8. The advantages of proposed flowsheet compared to existing hydrometallurgical flowsheet for black mass recycling include 1. Robust flowsheet to treat various chemistries within NMC spectrum (no reducing agent needed), 2. Close loop process (HCl regeneration), 3. Better separation and purification of metals from leach solution in later stages especially Li, in this case it is possible to recover Li first prior to other metals, and solve persisting significant loss of Li in battery recycling, 4. Compact process (less stages required compared to conventional recycling flowsheet). Sieving combined with impurities removal stage is advised to reduce the impurities Cu, Fe and Al.



Based on the organic acid leaching results there are several recommendations:

- **Selective Recovery:** Depending on the target metal, we may consider sequential leaching (e.g., starting with oxalic acid for aluminum and phosphorus, followed by citric acid for transition metals) to optimize recovery.
- **Optimization of Conditions:** To improve lithium recovery, additional factors like extended leaching time or increased agitation could be explored, particularly when using oxalic acid.
- **Complex Formation:** Since citric acid works through both proton attack and chelation, understanding how citric-metal complexes form during leaching might give further insights into tuning the process for different metal recoveries.

Since some ions could remain trapped in the solid, it might be useful to:

- Perform additional washes of the solid after leaching, as suggested, to ensure full recovery of leached metals.
- Optimize the contact time and agitation conditions, since 250 rpm may not be sufficient for thorough mixing in all cases.

- Look into the formation of metal-organic complexes or precipitates in the solution with oxalic or citric acids, which could complicate metal recovery. This can be analyzed by techniques like XRD or SEM on the final dried solid.

4.3 Na-battery leaching

Regarding the Na-ion metal recovery, the task will continue:

- Optimizing the solid-liquid extraction stage..
- Developing the purification stage to recover valuable metal precursors, and marketable products.