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Recycling of Lithium-Ion Batteries: Effect of Hydrogen Peroxide and a Dosing Method on the Leaching of LCO, NMC Oxides, and Industrial Black Mass

Nathália Vieceli, Piamcheewa Benjamasutin, Raksina Promphan, Pia Hellström, Magnus Paulsson, and Martina Petranikova*



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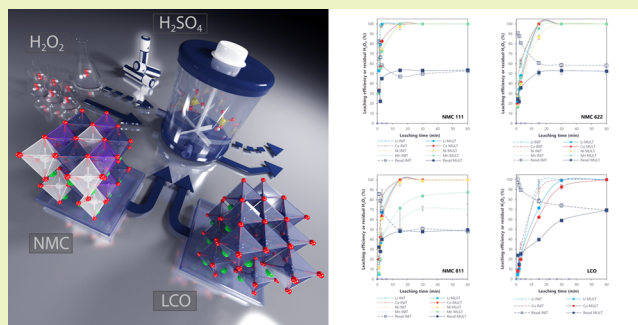
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ABSTRACT: The effect of hydrogen peroxide on the leachability of different reference cathode active materials used in lithium-ion batteries (LCO, NMC 111, NMC 622, and NMC 811) was investigated using 2 M H_2SO_4 . An innovative method was used to monitor the consumption and residual concentration of hydrogen peroxide, which can help optimize its addition and improve the economy and resource yield of the process. The reducing effect of hydrogen peroxide was compared using two methods of adding it. Leaching with hydrogen peroxide significantly improved the dissolution and 100% yield was reached within 15 min for the NMC oxides (except NMC 811) and within 30 min for LCO. Co and Ni were more easily leached from NMC 811 compared to other NMC oxides. The dissolution of metals from LCO was in general slower. Cu and Al can also act as reducing agents and their presence increased the leaching yield of the transition metals, especially Mn, and resulted in less residual hydrogen peroxide. The H_2O_2 addition method (all at once or charged at multiple occasions) did not influence the maximum leachability (except for Mn/NMC 811) or the amount of consumed hydrogen peroxide, but the rate of dissolution was slower when charging at multiple times. The optimal conditions determined for the reference NMC 111 cathode material [3% v/v H_2O_2 (59% w/w), S/L 1:20 g/mL, 2 M H_2SO_4 , 50 °C] were applied for an industrial black mass sample ($\text{Li}_{1.09}\text{Ni}_{0.31}\text{Mn}_{0.39}\text{Co}_{0.39}\text{O}_2$). The result was 100% yield for Li, Mn, and Ni, after 15 min and 100% yield for Co after 60 min.



KEYWORDS: battery recycling, waste valorization, hydrometallurgy, lithium-ion batteries, acid leaching, hydrogen peroxide

INTRODUCTION

The shift in demand for lithium-ion batteries (LIBs) to include larger batteries for electric vehicles (EVs) and stationary storage has boosted the supply chain of LIB materials¹ and it is expected that EVs account for 77% of the total installed capacity of LIBs in 2030.² From the resource conservation and environmental protection point of view, it is essential to adopt sustainable methods to recycle the consequent increasing flow of spent LIBs,³ which is also attractive from an economical perspective when considering their high content of metals when compared to the respective primary resources.⁴

When the mass production of LIBs started in 1995, LiCoO_2 (LCO) was by far the dominant cathode material; however, it started to be gradually replaced by other materials due to cost and resource availability issues.⁵ Thus, the chemical substitution at the Co site started to be explored, resulting in several combinations of cathode active materials with decreased production costs, increased safety, and enhanced energy densities.⁶ By 2010, the market share of LCO declined

to 40% and the use of the ternary system $\text{LiNi}_{1/3}\text{Mn}_{1/3}\text{Co}_{1/3}\text{O}_2$ (NMC 111) increased dramatically.⁵ Nowadays, NMC 111 chemistry has been replaced by $\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$ (NMC 622) and $\text{LiNi}_{0.8}\text{Mn}_{0.1}\text{Co}_{0.1}\text{O}_2$ (NMC 811), comprising less Co^{7,8}—one of the battery materials considered critical for the European Union.⁹ However, although alternative chemistries have been commercially adopted, most studies about LIB recycling focus on the obsolete LCO chemistry or the first generation of NMC (NMC 111).

Initially, pyrometallurgical processes were preferred for recycling of LIBs due to their existing use with Ni- and Co-based alloys.^{10–12} Nowadays, hydrometallurgical processes

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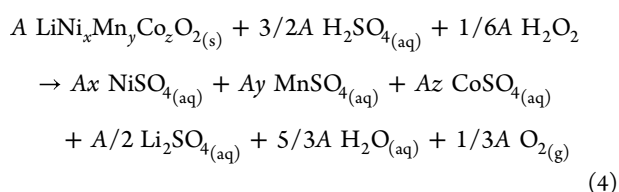
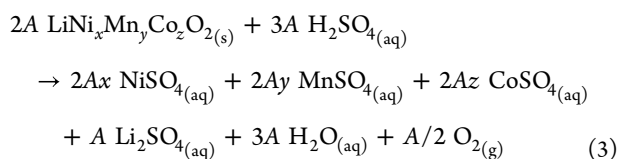
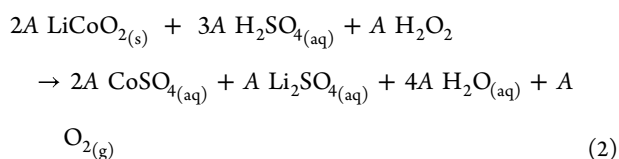
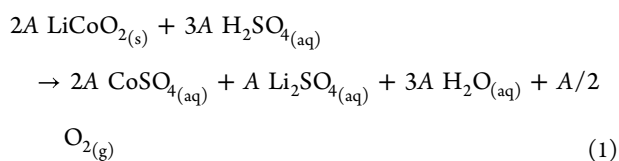
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have been widely investigated for the recycling of LIBs, and the largest LIB recycling market, China, is mainly covered by hydrometallurgical methods.¹³ Hydrometallurgical processes are more flexible and have advantages over pyrometallurgy, such as low energy requirement, high metal and material recovery, high product purity, and lower gas emissions^{14–17} and enables the recovery of metals that are lost in the slag in pyrometallurgical processes, such as lithium.¹⁸ Thus, hydrometallurgical processes will be essential to achieve the sustainability of the value chain of LIBs, as well as the recovery targets and shares of recovered materials that are expected to be required by the recent proposal to modernize the EU's legislation on batteries.¹⁹

Both organic and inorganic leaching agents have been widely investigated for the hydrometallurgical treatment of LIBs, but also alkali and biological leaching and intensified leaching (combined with mechanochemical activation or ultrasonic technology) have been studied.^{16,20–27} For example, ultrasound helped decrease the process time by 50% and improved the leaching yield.²³ Inorganic leaching agents typically used to leach metals from LIBs include hydrochloric acid, nitric acid, sulfuric acid, and phosphoric acid.^{28,29} Additionally, hydrogen peroxide (H₂O₂) is generally used as a reducing agent during leaching.^{30–32} The purpose of adding H₂O₂ or other reducing agents during leaching is to reduce the transition metals present in spent LIBs (for example Co³⁺ to Co²⁺) to reach more favorably leachable valence states^{33,34} but also to decrease the need for more concentrated acid solutions to obtain comparable efficiencies.³⁵ The reactions of the cathode material from LIBs (LCO or NMC chemistries) with sulfuric acid without and with the addition of hydrogen peroxide are represented by eqs 1–4, where $x + y + z = 1$, while A represents the molar quantity of reagents and products in the reactions. It is possible to observe that oxygen is the main gas product obtained in the leaching process when sulfuric acid is the leaching agent and hydrogen peroxide is used as the reducing agent.



The use of alternative reducing agents has also been investigated, including, for example, sodium bisulfite,³⁴ sodium metabisulfite,³⁶ ascorbic acid, glucose, hydrazine sulfate,^{37,38} biomass waste,³⁹ and also iron ions.⁴⁰ However, hydrogen peroxide is still the most widely used reducing agent, even having the constrain to be decomposed by transition metals. Furthermore, although the use of hydrogen peroxide has been investigated in several studies, monitoring its consumption and remaining concentration during the leaching reaction has not been reported, although it can affect its efficient use.

In this context, considering that there is a lack of research on the recycling of new cathode chemistries, one of the main goals of this study is to compare the leaching behavior of different reference cathode active materials currently used in LIBs (LCO, NMC 111, NCM 622, and NMC 811). Two methods of adding H₂O₂ were compared during their leaching, which is a novel contribution from this study and could lead to improved use of chemicals and reduced costs. Another innovative approach tested in this work was to monitor the consumption of hydrogen peroxide in the leaching by following its residual concentration, which has not been reported in the literature, despite its widespread use as a reducing agent. This can help to optimize the use of hydrogen peroxide, increasing the resource efficiency of the process and reducing operational costs.

MATERIALS AND METHODS

Solid Samples: LCO, Reference Mixed NMC Oxides, and Spent LIBs from EVs. The cathode active materials (CAMs) used in this study are LCO (pure metal oxide, Sigma-Aldrich, powder, 99.8% trace metals basis) and reference mixed NMC oxides (NMC 111, NMC 622, and NMC 811) which were kindly provided by Uppsala University (Department of Chemistry—Ångström Laboratory) as fine powders in a pure form. Since only reference cathode active materials were used, the samples did not contain any organic coating from binders. Although the particle size of the oxides was not determined, similar products commercially available have a particle size in the range of <5 μm. The real NMC cathode material from spent LIBs of EVs was kindly provided by Volvo Cars AB (Sweden), dismantled by Stena Recycling International AB (Sweden), and mechanically treated at Akkuser Oy (Finland) using a two-stage crushing line followed by mechanical sieving and magnetic separation. The fine fraction was used in this work (92.8% w/w < 1 mm) and its elemental composition was determined by digestion in aqua regia for 5 h at 80 °C, followed by filtration and ICP-OES analysis (Inductively Coupled Plasma Optical Emission Spectrometry, iCAP 6000 Series, Thermo Fisher Scientific).

Leaching Tests. General Procedures. Acid solutions were prepared using sulfuric acid (H₂SO₄, 95–97%, Sigma-Aldrich) and Milli-Q water. The acid concentration was set the same for all tests (2 M), which is in the range considered optimal conditions.¹⁸ Leaching tests were performed in 100 mL plastic vessels with caps or 20 mL glass vials with caps, depending on the volume considered in the tests. Leaching temperatures around 60–80 °C are usually employed,¹⁸ but in this study, all the experiments were performed at 50 °C using a water bath with temperature control to lower the influence of temperature in the process. The leaching solution was heated, and the solid sample was added into it after reaching constant temperature, and then, the recording of the leaching time was started. Magnetic stirrers were used for agitation, and the stirring speed was set at 300 rpm. When the defined leaching time was reached, a sample was taken with a syringe and immediately filtered to stop the reaction (~200 μL of sample using a filter of 0.45 μm of pore size, 25 mm of diameter, polypropylene membrane, VWR). The leachates obtained after filtration were diluted in 0.5 M nitric acid (Sigma-Aldrich, 65%, for inorganic trace analysis) and analyzed by ICP-OES to determine the concentration of the metals leached during the tests. The leaching

yield was calculated based on the initial content of metals present in each sample.

Preliminary Tests without Hydrogen Peroxide. Preliminary tests were performed without reducing agents, and two solid to liquid ratios (S/L) were evaluated (1:10 and 1:20 g/mL) to obtain more concentrated leachates and reduce the volumes in subsequent operations. The volume of liquid was set at 10 mL to promote efficient mixing, which can influence the leaching yield, and samples were taken after 1, 5, 10, 15, 30, and 60 min of leaching. Additional preliminary tests were performed to evaluate the reduction capacity of current collectors by adding 10% w/w Al (commercial Al foils) and 10% w/w Cu (Merck, powder <63 μm , purity >99%), based on the weight of the cathode material.

Testing the Effect of Hydrogen Peroxide. The effect of hydrogen peroxide (59–59.5% w/w, EKA HP C59, Nouryon) as a reducing agent was investigated. In these tests, 40 mL of 2 M H_2SO_4 was used to decrease the effect of sampling, considering that larger volumes were required to monitor the consumption of hydrogen peroxide. The S/L ratio was set at 1:20 g/mL, and samples were taken after 1, 2, 3, 15, 30, and 60 min. The main goal of taking samples along the time was to observe the kinetic behavior of the different samples.

Considering that hydrogen peroxide decomposes in the presence of transition metals, two methods to add H_2O_2 were also tested—all added at the beginning or charging the same total amount but divided in multiple additions. The theoretical amount of H_2O_2 needed to fulfill the reactions (for an S/L ratio of 1:20 using 10 mL of 2 M H_2SO_4) was calculated based on eqs 1–4 for the different cathode materials (Table 1). However, since the consumption of H_2O_2 is

Table 1. Theoretical Amount of H_2O_2 Required when Leaching Different Reference Cathode Materials (at an S/L Ratio of 1:20) Was Calculated Based on Eqs 1–4

cathode material	H_2O_2 (59% w/w) (% v/v)	H_2O_2^a (g/L)	H_2O_2 (as 100%) ^b (mg/g cathode material)
LCO	1.2	8.7	174
NMC 111	0.4	2.9	59
NMC 622	0.2	1.8	35
NMC 811	0.1	0.9	17

^a H_2O_2 concentration in the leaching solution (S/L ratio of 1:20 using 10 mL of 2 M H_2SO_4). ^bTheoretical amount of H_2O_2 (as 100%) required.

expected to be higher than the stoichiometric amount due to the reduction/oxidation of other metals, the amount of H_2O_2 required for each cathode material was experimentally determined. In this case, the leaching was done on a small scale (10 mL of solution, 2 M H_2SO_4 , S/L 1:20 g/mL, 50 °C) and the H_2O_2 was added slowly (around 50–100 μL at a time) until the cathode material was totally dissolved (H_2O_2 amounts are given in Table 2). The effect of combining hydrogen peroxide with current collectors was also tested, and the

same amount of Al and Cu was used (10% w/w based on the weight of the cathode material).

The residual concentration of hydrogen peroxide was determined by iodometric titration, which is described by Schumb and co-workers⁴¹ and the titration was performed immediately after sampling. Although the method is well known, it has not been applied in studies focused on recycling of LIBs using hydrogen peroxide. Therefore, the use of this method represents an innovative approach of this work to monitor the consumption and efficient use of the reducing agent. Interfering substances present in the leaching solutions were not considered in the procedure; therefore, minor discrepancies may have occurred. All the tests were performed in triplicate to evaluate the experimental deviation.

Leaching of Industrial Black Mass. The best conditions determined for the reference cathode materials were applied in a leaching test using an industrial black mass sample from spent lithium-ion batteries. In this case, the leaching was performed using a titration vessel with a thermostat jacket covered with a lid. The temperature (50 °C) was controlled using a thermal bath connected to the vessel and the stirring speed was set to 300 rpm (overhead mechanical stirrer). After leaching the industrial sample, the solid residue was filtered (Whatman quantitative hardened filter paper, ashless, grade 542), abundantly washed with Milli-Q water to remove residual leachate, and then dried in a furnace at 80 °C for 24 h. The solid residue was characterized by X-ray powder diffraction (XRPD) using a Siemens D5000 X-ray diffractometer with an accelerator voltage of 40 kV and a current of 40 mA. A Cu K-radiation was used with a rotation speed of 15 rpm and a 2θ range from 15° to 100°, and the Powder Diffraction File (PDF) database from ICDD (International Center for Diffraction Data) was used to evaluate the results.

RESULTS AND DISCUSSION

Preliminary Tests without Reducing Agents: Effect of Solid to Liquid Ratio. Preliminary tests were performed comparing two solid to liquid ratios (1:10 and 1:20 g/mL). The total volume of the acidic solution was higher than the theoretical volume required according to eqs 1–4. In general, changing the S/L ratio did not promote a significant increase in the leaching yield, possibly because the amount of acid did not constrain the reaction in any case. When an S/L ratio of 1:20 was used, a slight improvement in the leaching yield of some metals was observed, except for Ni from NMC 111 and NMC 811, as can be observed in Figure 1. A decrease in the pulp density (lower S/L ratio) decreases the viscosity of the system, and as result, it decreases the mass transfer resistance in the liquid–solid interface, which can have a positive effect on the leaching yield.⁴²

The final leaching yield of Co from LCO was similar to NMC 111 (~32% after 45 min), while the lowest final yield for Li was from LCO (~63%). The leaching yield increased until

Table 2. Amount of H_2O_2 Needed To Fully Dissolve the Cathode Materials and Addition Time of H_2O_2 (59% w/w)

cathode material	H_2O_2 (59% w/w) (% v/v) ^a	H_2O_2 (g/L) ^{a,b}	time of addition of H_2O_2 (min of reaction)
LCO	7	51	0, 3, 6, 15, 22, 27, 35
NMC 111	3	22	0, 3, 6
NMC 622	4	29	0, 3, 6, 15
NMC 811	3	22	0, 3, 6
LCO + Al and Cu ^c	8	59	0, 3, 6, 15, 22, 27, 35, 45
NMC 111 + Al and Cu ^c	3	22	0, 3, 6
NMC 622 + Al and Cu ^c	6	43	0, 3, 6, 15, 20, 27
NMC 811 + Al and Cu ^c	3.5	26	0, 3, 6, 15, 20, 27

^aAmount experimentally determined and corresponding to the amount required to fully dissolve the cathode materials using an S/L ratio of 1:20 g/L (tests using 0.5 g cathode materials). ^b H_2O_2 concentration in leaching solution. ^cAddition of 10% w/w Cu (powder, <63 μm) and 10% w/w Al (foils), based on the weight of the cathode material.

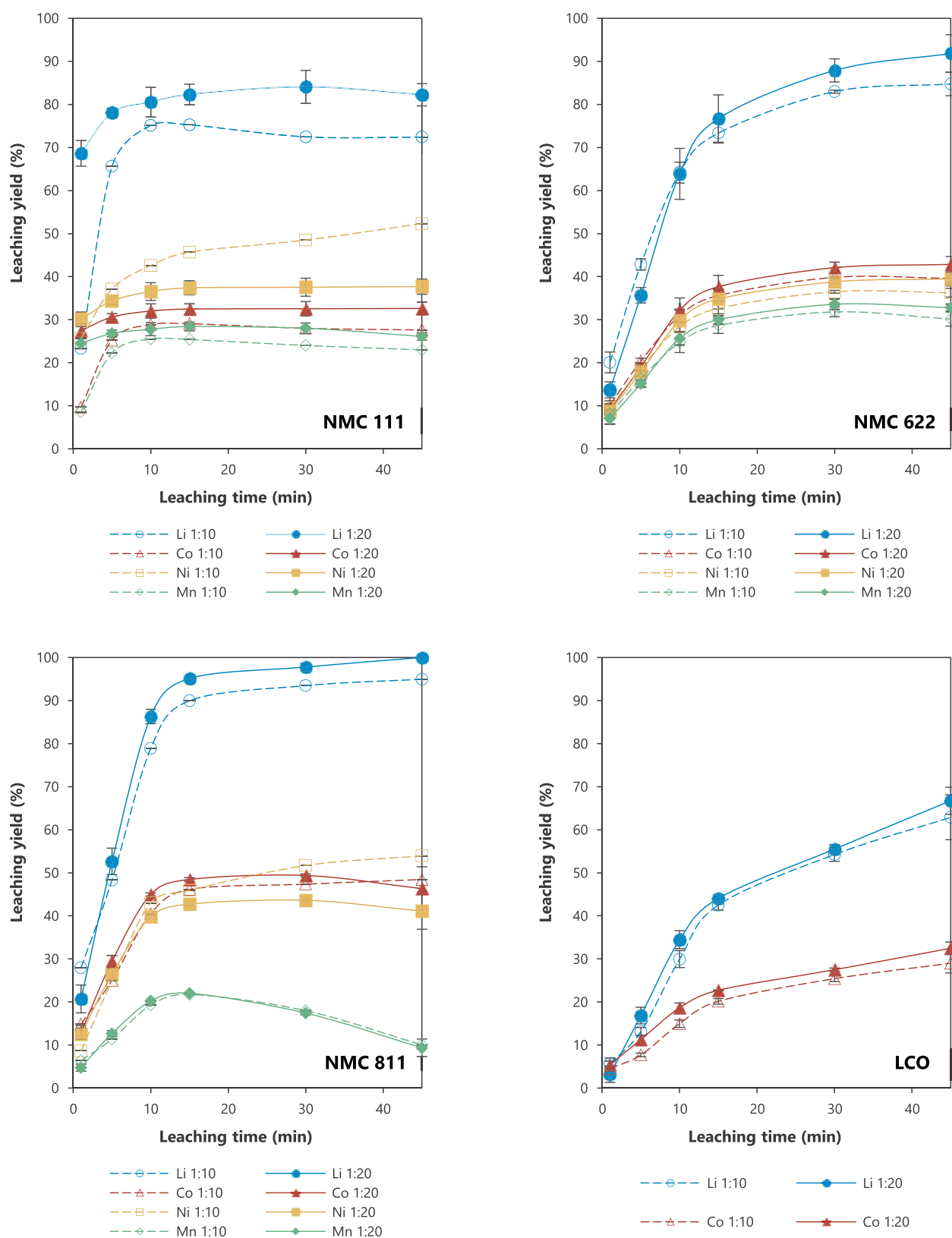


Figure 1. Leaching yield of metals from different cathode materials at 50 °C, 10 mL 2 M H₂SO₄, and two different solid to liquid ratios—1:10 g/mL (dotted lines) and 1:20 g/mL (solid lines). Standard deviation for triplicates.

around 15 min in most cases and for Ni, Mn, and Co, it remained in general lower than 50%, which is expected when leaching without a reducing agent since the leaching yield is enhanced at lower valence states. The leaching behavior of Co

and Ni was similar to each other for NMC 622 and NMC 811. Mn had the lowest leaching yield among the transition metals, especially from NMC 811, from which the dissolution of Mn increased until 15 min and then decreased. The leaching yield

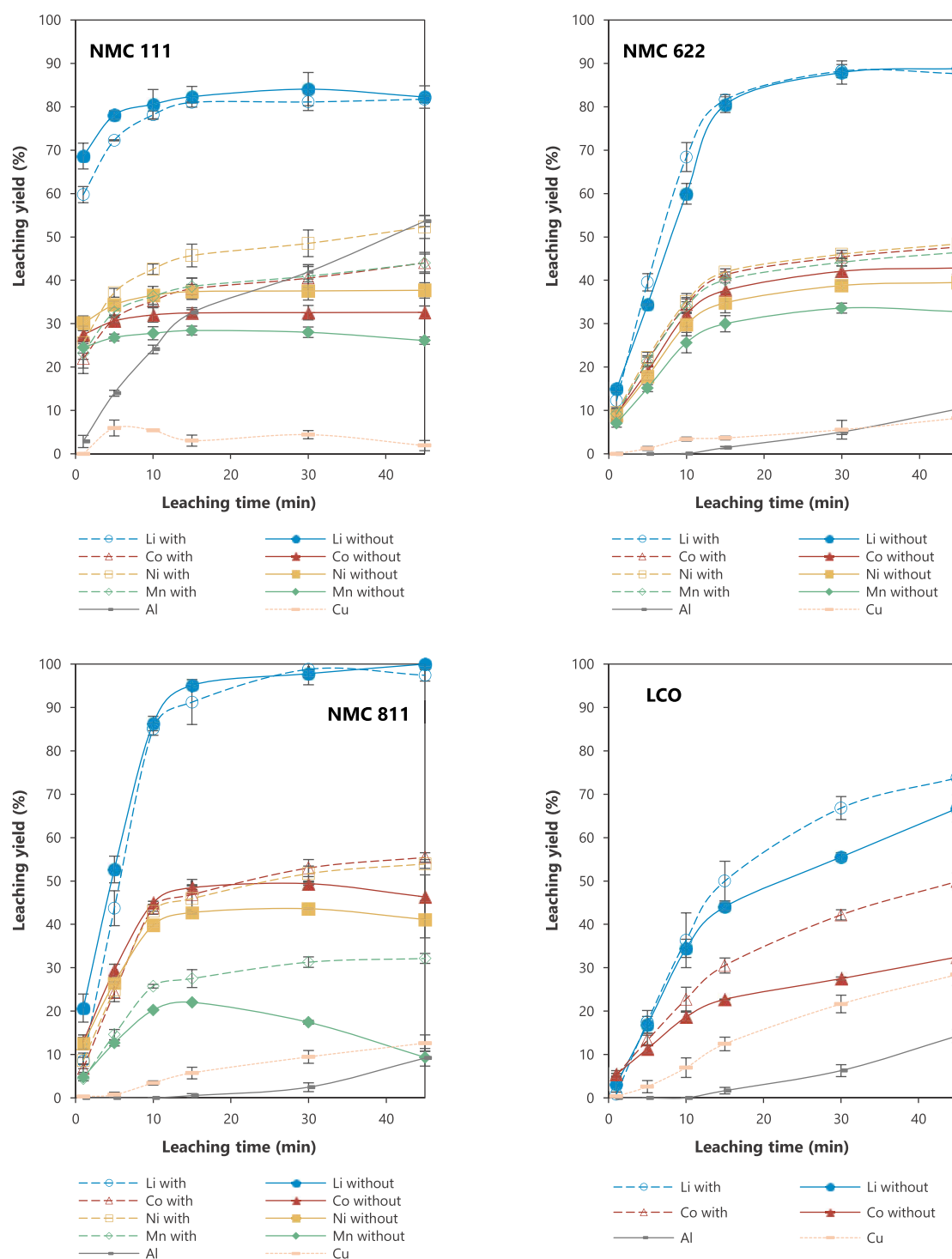


Figure 2. Leaching yield of metals from different cathode materials with (dotted lines) and without current collectors (solid lines). Leaching conditions: 50 °C, 10 mL 2 M H₂SO₄ and S/L ratio of 1:20 g/mL. Addition of 10% w/w Al (foils) and 10% w/w Cu (powder). Standard deviation for triplicates.

after 45 min of Li, Co, and Ni from NMC 811 was slightly higher than that for the other CAMs.

Xuan et al.⁴³ also investigated the leaching behavior of different reference NMC oxides using 4 M HCl at 54 °C, and a faster dissolution rate was observed for NMC 811, followed by NMC 622, which was possibly related to the higher stability of manganese-rich NMC electrodes. The leaching yield of Li was much higher than that for the transition metals, whose yield did not exceed 60%. This is in accordance with the results

observed by Billy et al.⁴⁴ who investigated the dissolution mechanism of NMC 111 in sulfuric acid (1 M at 30 °C). Their results suggest a dissolution mechanism governed by a delithiation process, which controls the leaching driving force and the transport properties. According to them, lithium depletion occurs at the beginning of the leaching process and induces charge compensation by the transition metals, which release electrons. The electrons can recombine inside the particle to reduce transition metals and promote oxide

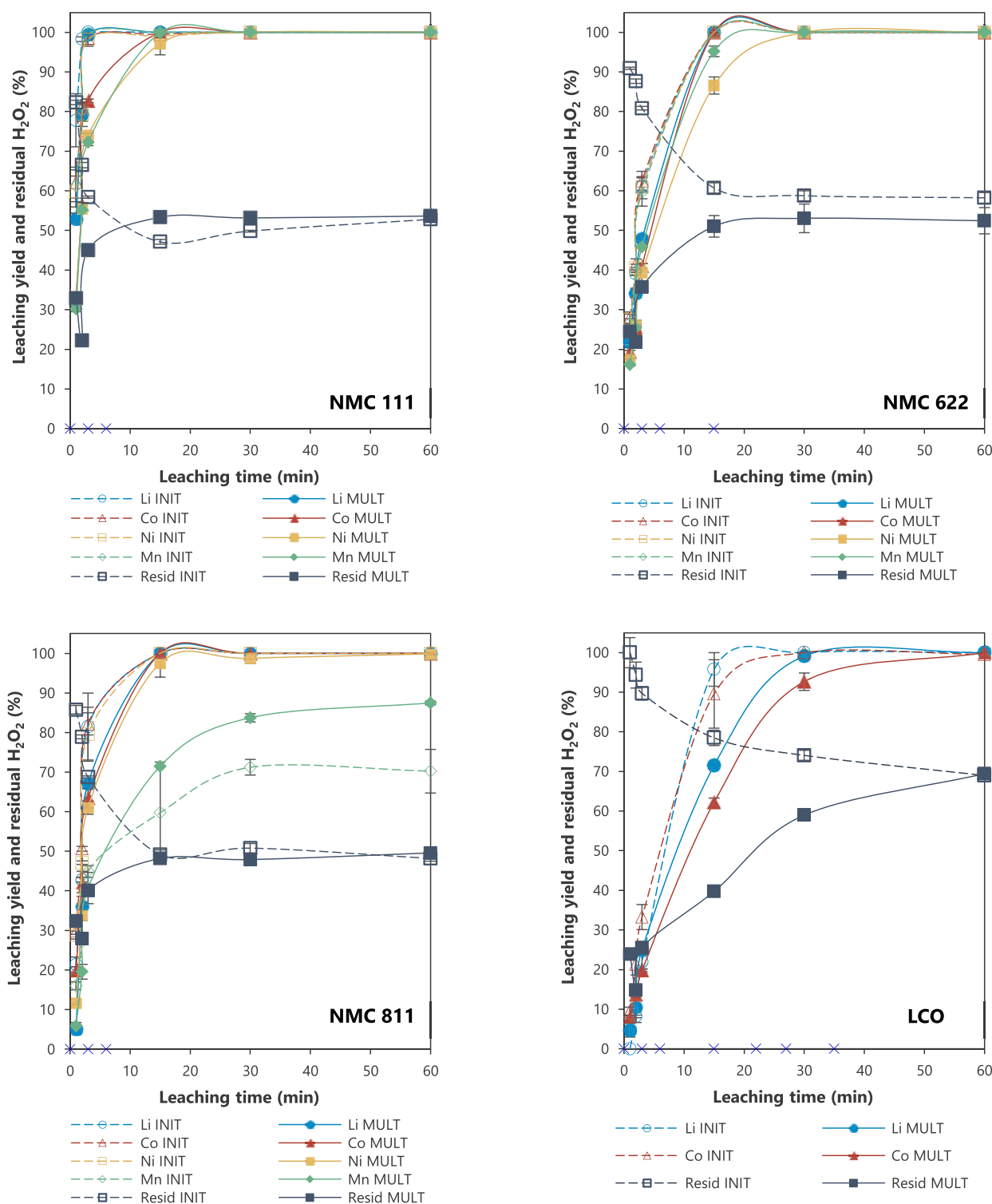


Figure 3. Leaching yield of metals from different cathode materials using two different methods to add hydrogen peroxide (H₂O₂) and the residual amount of H₂O₂ (%). INIT—total volume of H₂O₂ added at the beginning (dotted line) and MULT—multiply additions of H₂O₂ (solid line). The adding time is identified by blue crosses on the x-axis (the H₂O₂ charge is given in Table 2). The residual content of hydrogen peroxide was calculated considering its total final concentration, despite the method of addition. Leaching conditions: 50 °C, 40 mL 2 M H₂SO₄ and S/L ratio of 1:20 g/mL. Standard deviation for triplicates.

dissolution during the removal of lithium ions.⁴⁴ In addition, covalent bonds connect oxygen and transition metals in the layered LiNi_{1/3}Co_{1/3}Mn_{1/3}O₂ material, while lithium and oxygen are connected through ionic bonds, which are more fragile than covalent bonds⁴⁵ and could also explain the more

favorable leachability of Li when compared to transition metals.

Preliminary Tests: Effect of Copper and Aluminum.

The leaching of transition metals from LIBs generally requires reductive conditions, which are normally reached using

Table 3. Accumulated Consumption of Hydrogen Peroxide (in mmol) for Different Cathode Materials when Adding all Hydrogen Peroxide at the Beginning of Leaching (INIT) and when Charging the Same Total Amount of H₂O₂ but Divided in Multiple Additions (MULT)

CAM sampling time (min)	LCO		NCM 111		NMC 622		NMC 811	
	INIT	MULT	INIT	MULT	INIT	MULT	INIT	MULT
1	0.0		1.5	0.1	3.4	0.3	3.7	0.2
2	5.7	0.0	8.7	2.9	4.6	1.2	5.5	1.4
3	8.5	1.9	10.8	5.6	6.9	5.0	8.1	6.9
15	15.3	10.6	13.7	12.1	13.7	17.0	13.2	13.4
30	17.9	16.3	13.0	12.2	14.4	16.3	12.7	13.5
60	21.0	18.6	12.2	12.0	14.6	16.6	13.4	13.0

hydrogen peroxide. The alternative use of Cu and Al was tested considering that these metals are used in LIBs as current collectors and therefore are present in industrial samples (black mass) to be leached. The standard reduction potential for Cu and Al is +0.34 and −1.66 V, respectively, compared to +1.92 V for Co³⁺. Thus, due to their lower electrochemical potential, Al and Cu could act as reducing agents in the leaching system, decreasing the oxidation state of Co³⁺, for example, and promoting the formation of CoSO₄ in sulfuric acid media.

The addition of Cu and Al promoted an increase in the leaching yield of the transition metals from all the different chemistries tested (Figure 2). As expected, the leaching yield of Li was not much affected by the presence of Al and Cu since it is less dependent on reductive conditions.^{40,46} The highest dissolution of Al was from NMC 111 (54%), and Cu was most easily solubilized from LCO (28%). The leaching yield of Li from LCO was also lower than that from the other CAMs, and the maximum leaching yield was not reached within the leaching time examined (45 min).

The effect of Al and Cu scrap on the acid leaching of an industrial processed cobalt-rich waste battery concentrate was recently investigated by Chernyaev et al.⁴⁷ and both Al and Cu had a positive effect on the leaching of Co. The authors predicted using a model that either 0.75 Cu/Co mol/mol, 0.7 Al/Co mol/mol, or a combination of both are required for full recovery of cobalt. According to them, Cu would be more efficient than Al; however, the effectiveness of Cu would be associated with side reactions with Fe, which was not evaluated in the present study. Moreover, the addition of such large amounts of Al or Cu could bring some challenges to the further purification of the leachate. The presence of Al, Cu, and Fe in the leachate may, for example, hinder the separation of Co in the solvent extraction step using the dialkyl phosphinic acid extractant Cyanex 272.⁴⁸ The removal of these elements is normally required and is generally accomplished by precipitation.¹⁸ Thus, high content of Al and Cu could require high amounts of precipitation agents and produce large quantities of solid residues that require proper treatment and disposal, negatively affecting the sustainability of the process.

Effect of Hydrogen Peroxide and Addition Method.

Hydrogen peroxide is normally added once at the beginning of the acid leaching operation as a reducing agent, but the effectiveness of this approach has not been reported in the literature. Considering that hydrogen peroxide is decomposed by transition metals, multiple additions during the leaching operation could be a more efficient alternative. Thus, the effect of adding hydrogen peroxide once at the beginning of the leaching operation was compared to adding the same total amount through multiple additions. The amount of hydrogen peroxide used (Table 2) was higher than the theoretical

amount (Table 1) and was experimentally determined as described in Materials and Methods. For this reason, different addition times and quantities were used, considering that the required amount of hydrogen peroxide experimentally determined varied for different cathode active materials, but its consumption was monitored for all different samples.

It is possible to observe that the use of hydrogen peroxide substantially improved the dissolution rate of metals from different CAMs when compared to the previous results. After 60 min, both ways of adding hydrogen peroxide resulted in similar leaching efficiencies for all CAMs (except for Mn, NMC 811), as can be seen in Figure 3. The residual content of hydrogen peroxide was calculated considering its total final concentration, despite the method of addition. The accumulated consumed amount of H₂O₂ after leaching, i.e., when the leaching yield is almost 100%, is not significantly affected by the addition method (Table 3). However, as can be seen in Figure 3, the reaction rate is higher when the total amount of hydrogen peroxide is added initially. The results suggest that since the reduction reaction is fast and for most metals the leaching yield reached almost 100% after only 15 min, it would be more favorable to provide the total amount of hydrogen peroxide once at the beginning of the process.

When the residual concentration of hydrogen peroxide using both addition approaches is compared (Figure 3), it is possible to observe that after 60 min of leaching, above 40% of hydrogen peroxide is left and the residual amount was even higher for the LCO chemistry (above 60%). Jha et al.²⁹ reported a similar efficiency for LCO (99.1% Li and 70.0% for Co); however, the hydrogen peroxide addition was 5% v/v. The leaching yield for all elements and chemistries was 100%, except for Mn in NMC 811 which was about 87%. The hydrogen peroxide amount used in this study seems to be high enough to reduce the oxides to more leachable forms, but it could probably be lowered and optimized in future studies. Moreover, it is important to highlight that an increase in the concentration of H₂O₂ promotes an increase in the metal dissolution up to a certain limit, from which a higher hydrogen peroxide concentration changes the reductive role of this agent to act as an oxidant.^{49,50}

Effect of Copper, Aluminum, and Hydrogen Peroxide. Tests were performed adding Al and Cu to simulate the leaching behavior of real samples, where the active material is supported on Al (cathode) and Cu (anode) foils. The total amount of hydrogen peroxide added was higher than the amount used in the previous test (without current collectors), and it was experimentally determined as discussed above (cf. Materials and Methods and Table 2). The total volume of hydrogen peroxide was added once when the leaching test was started. The presence of Cu and Al did not negatively affect the

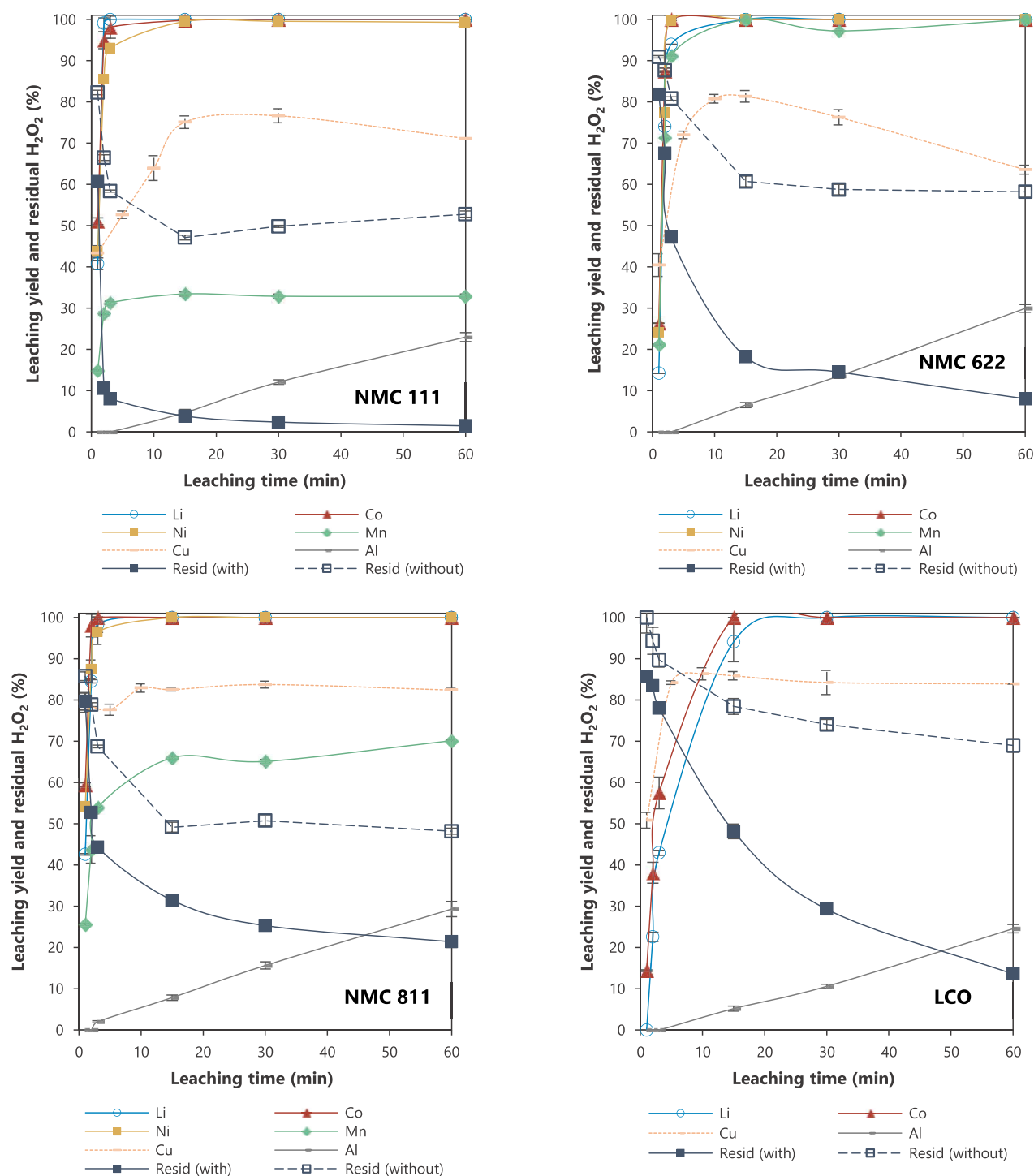


Figure 4. Leaching yield of metals from different cathode materials with hydrogen peroxide (H_2O_2) and current collectors 10% w/w Al (foils) and 10% w/w Cu (powder) present. The total amount of H_2O_2 (cf. Table 2) was added at the beginning of the test. Resid (with)—residual amount of H_2O_2 with the foils (solid line); resid (without)—residual amount of H_2O_2 without Al/Cu (dotted line). Leaching conditions: 50 °C, 40 mL 2 M H_2SO_4 and S/L ratio of 1:20 g/mL. Standard deviation for triplicates.

leaching yield of Li, Ni, and Co, which reached $\sim 100\%$ after less than 15 min (Figure 4). The leaching yield of Mn was lower for NMC 111 in the presence of current collectors (Figure 4). The leaching yield of Cu increased at the beginning of the leaching and then tended to decrease again for NMC 111 and NMC 622. The leaching yield of Al had a slower increase, which can be related to the fact that it was added as foils (while copper was added in a powder form). Additionally,

the passivation of Al foil surfaces during the acid leaching can constrain the reaction.⁵¹ The residual concentration of hydrogen peroxide was also monitored throughout the operation, and it was lower than the previous tests without Cu and Al, as expected (the residual amount of H_2O_2 without Cu and Al was plotted in the graphs for comparison). For the NCM 111, similar conditions were used by Wang and Friedrich⁵² (25 g/L H_2O_2), who observed that a further

Table 4. Elemental Composition of the Black Mass Sample from Spent EV LIBs (Results Based on Triplicate)

result/element	Li	Mn	Co	Ni	Cu	Al	Mg	Zn
mass fraction (%)	3.4 ± 0.1	7.5 ± 0.1	10.5 ± 0.3	8.2 ± 0.2	7.8 ± 0.1	3.5 ± 0.1	0.20 ± 0.01	0.20 ± 0.01
limit of detection	<0.1	<0.1	<0.1	<0.1	<0.1	0.2	<0.1	<0.1

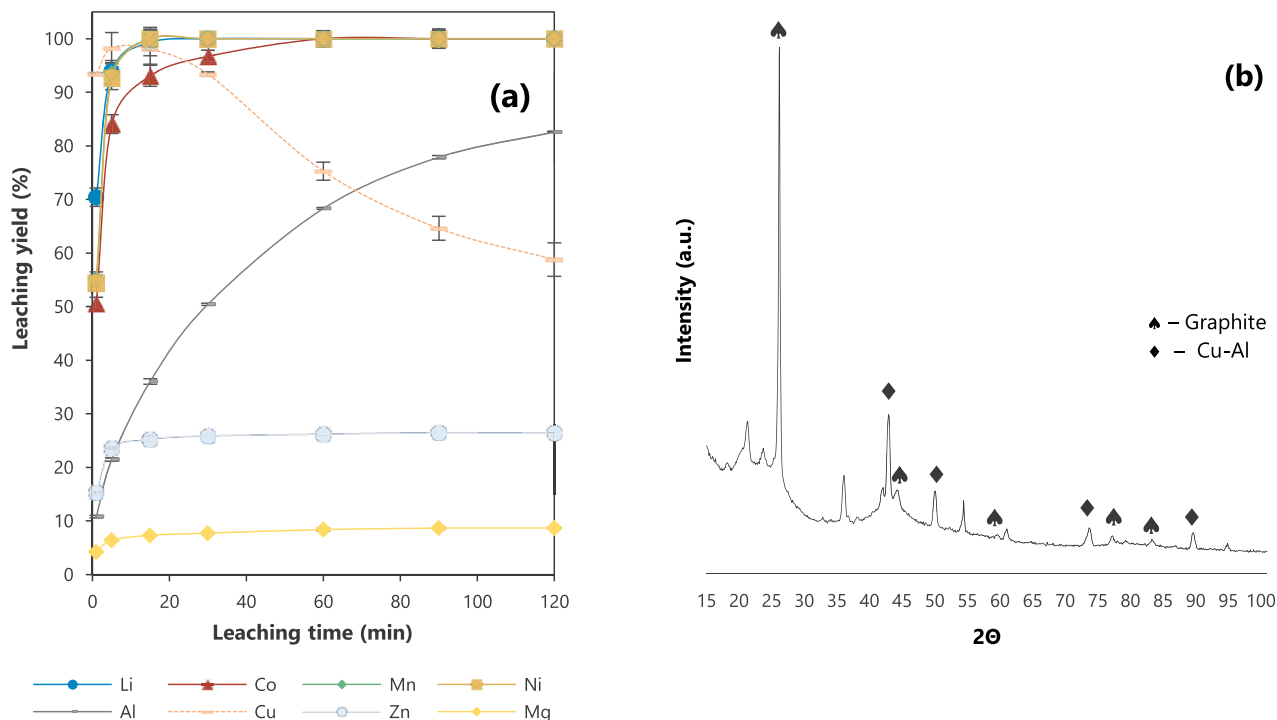


Figure 5. (a) Leaching of metals from a black mass sample ($\text{Li}_{1.09}\text{Ni}_{0.31}\text{Mn}_{0.39}\text{Co}_{0.39}\text{O}_2$) from spent lithium-ion batteries. Leaching conditions: 50 °C, 120 min, 60 mL 2 M H_2SO_4 , S/L 1:20 g/mL, and 3% v/v H_2O_2 (59% w/w). Standard deviation for triplicates. (b) Diffractogram of the leaching residue, where spade: graphite and diamond: $\text{Cu}_{0.92}\text{Al}_{0.08}$.

increase from 50 to 100 g H_2O_2 /L did not promote an increase in the leaching yield. The residual amount of H_2O_2 was higher for LCO and NMC 811 than that for the other CAMs but lower than that in the tests where Cu and Al were not added. Based on the results, the removal of current collectors by physical processing before the leaching operation could reduce the consumption of hydrogen peroxide with a positive effect on the resource yield of the process.

Leaching of Cathode Material (Black Mass) from Spent LIBs. The leaching behavior of a real black mass sample from spent EV LIBs was evaluated. The elemental composition of the sample is presented in Table 4, and its empirical formula was $\text{Li}_{1.09}\text{Ni}_{0.31}\text{Mn}_{0.39}\text{Co}_{0.39}\text{O}_2$, where the fraction of Ni, Mn, and Co is close to the NMC 111 chemistry. Therefore, the optimal conditions previously obtained for NMC 111 were used in these leaching experiments (3% v/v H_2O_2 , S/L of 1:20 g/mL, 60 mL of 2 M H_2SO_4 , 50 °C). The Al and Cu content in industrially processed black mass will vary depending on the physical pretreatment used. In the black mass samples used in this study, the content of Al and Cu was lower than that in the previous tests with reference CAMs (Preliminary Tests: Effect of Copper and Aluminum and Effect of Copper, Aluminum, and Hydrogen Peroxide).

The leaching yield of Li, Mn, and Ni reached 100% within 15 min of leaching, while Co was completely leached after 60 min (Figure 5a). The leaching yield of Al increased more slowly and reached 68% after 60 min. The dissolution of copper increased very fast and reached 98% after 5 min and

then decreased to around 60% after 120 min. The leaching yield of Mg and Zn leveled off after 15 min and reached 7 and 25%, respectively. Thus, under the tested conditions in the presence of hydrogen peroxide, it is possible to efficiently leach valuable metals from spent lithium-ion batteries with very high leaching yields even after short leaching times. The diffractogram pattern for the solid residue is presented in Figure 5b and the main peaks in the pattern correspond to graphite and Cu–Al, indicating that the main metals present in the sample were leached under the tested conditions.

CONCLUSIONS

The leaching of different reference cathode active materials used in lithium-ion batteries (LCO, NMC 111, NMC 622, and NMC 811) was assessed in this work, which represents an innovative aspect of this study since their recycling has not been often reported in the literature. A slight improvement in the leaching yield of some metals was observed when the solid to liquid ratio was 1:20 g/L compared to 1:10. Co and Ni were more easily leached from NMC 811 compared to the other NMC reference cathode materials and about half of these elements could be leached without a reducing agent. The dissolution of metals from LCO was in general lower than that of other cathode chemistries and reached 92% for Li and 43% for Co after 60 min.

Leaching in the presence of Cu powder and Al foils, which are used as current collectors in industrial samples and may function as reducing agents, increased the leaching yield of the

transition metals, especially for Mn, for all NMC chemistries. Maximum leaching efficiencies obtained were for NMC 811 (100% Li, 54% Ni, 45% Mn, and 54% Co) and LCO (74% Li and 50% Co) after a leaching time of 60 min. Leaching in the presence of hydrogen peroxide, used as a reducing agent, significantly improved the dissolution of Co, Ni, and Mn, and 100% yield was reached within 15 min for the mixed NMC oxides (except for NMC 811 where 60% of the Mn was dissolved) and within 30 min for LCO. Two different methods of adding hydrogen peroxide were compared (all at once or charged on multiple occasions). The H₂O₂ addition method did not influence the maximum leachability (except for Mn from NMC 811) or the amount of consumed hydrogen peroxide, but the rate of dissolution was slower when adding H₂O₂ on multiple occasions. An innovative approach was used to monitor the consumption of hydrogen peroxide and evaluate the yield of its use. This method can help to decrease the consumption of chemicals and costs. Leaching in the presence of Cu and Al (to simulate industrial samples) resulted in a lower amount of residual hydrogen peroxide. Thus, the removal of current collectors by physical processing before leaching is important to reduce the H₂O₂ consumption and improve the resource yield of the operation.

Leaching of an industrial-produced black mass sample (Li_{1.09}Ni_{0.31}Mn_{0.39}Co_{0.39}O₂) using the optimal conditions for the NMC 111 reference cathode material [3% v/v H₂O₂ (59% w/w), S/L 1:20 g/mL, 2 M H₂SO₄, 50 °C] led to 100% yield for Li, Mn, and Ni, after 15 min, and Co was completely leached after 60 min (93% after 15 min). The dissolution of Cu, Al, Zn, and Mg was 98, 36, 25, and 7%, respectively, after 15 min, and 59, 83, 26, and 9% after 120 min. Optimization of the leaching conditions, also considering the leachability of noncathode active elements, will be important to not negatively affect subsequent process steps.

AUTHOR INFORMATION

Corresponding Author

Martina Petranikova – Department of Chemistry and Chemical Engineering, Industrial Materials Recycling and Nuclear Chemistry, Chalmers University of Technology, Gothenburg SE-41296, Sweden; orcid.org/0000-0002-0957-7768; Email: martina.petranikova@chalmers.se

Authors

Nathália Vieceli – Department of Chemistry and Chemical Engineering, Industrial Materials Recycling and Nuclear Chemistry, Chalmers University of Technology, Gothenburg SE-41296, Sweden

Piamcheewa Benjamasutin – Department of Chemistry and Chemical Engineering, Industrial Materials Recycling and Nuclear Chemistry, Chalmers University of Technology, Gothenburg SE-41296, Sweden

Raksina Promphan – Department of Chemistry and Chemical Engineering, Industrial Materials Recycling and Nuclear Chemistry, Chalmers University of Technology, Gothenburg SE-41296, Sweden

Pia Hellström – Nouryon, Bohus SE-44580, Sweden

Magnus Paulsson – Nouryon, Bohus SE-44580, Sweden

Complete contact information is available at:

<https://pubs.acs.org/10.1021/acssuschemeng.3c01238>

Notes

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