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Re-synthesis of Cathode Precursors from End-of-Life Lithium-Ion Battery Electrode Powders: Effect of Leaching Conditions on the Co-precipitation of Nickel-Manganese-Cobalt Mixed Oxalate

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The lithium-ion battery (LIB) market is experiencing rapid growth due to the increasing interest in electric and hybrid vehicles. LIBs are categorized by their cathodic materials into four main types: LCO (cobalt-based), NMC (nickel, manganese, cobalt), NCA (nickel, cobalt, aluminum), and LFP (iron phosphate). The widespread production and use of LIBs will inevitably result in a significant increase in end-of-life LIBs, which have to be recycled. Hydrometallurgical recycling processes offer an innovative solution for metal recovery from end-of-life LIBs. In particular, processes that include the resynthesis of cathodic materials can enhance sustainability by directly recovering mixed precursors of cathodes from leach liquors. The chemical composition of leach liquors, specifically the concentrations of Ni, Mn, and Co, as well as the levels of impurities such as Fe, Cu, and Al, can influence the final properties of the mixed precursors. In this study, two different leach liquors and one synthetic solution were used for precursor production via oxalate precipitation. The results indicate that according to the leaching composition the stoichiometry and conversion efficiency of the precipitation reaction can very, while no significant difference is observed in morphology and size of particles.

1. Introduction

The energy transition is one of the most urgent challenges of recent years. Many promising technologies are under development (Solomon and Krishna 2011). Short-range energy storage systems are key components of this transition, particularly concerning near-future perspectives. A significant portion of the field is dominated by batteries (Gallo et al. 2016). These batteries fall into several categories, among which Lithium-Ion Batteries (LIBs) are the most established and promising (Li et al. 2018; Whittingham 2004). Almost all electronic devices rely on this technology. Their popularity is mainly driven by the increasing interest in electric vehicles (EVs) and hybrid vehicles (HVs). LIBs consist of a cathode and an anode, where lithium ions intercalate and de-intercalate during charge-discharge cycles; this mobility allows circuit closure and electron transfer (Korthauer 2018; Whittingham 2004). However, cyclic intercalations stress the electrodes, causing deterioration. The goal is to use materials that enhance ion mobility while improving durability.
The anode is typically made of graphite, although alternative materials are being explored in current research (Cheng et al. 2021; Liu, Yu, and Lee 1999). For the cathode, several materials have been proposed (Fergus 2010), but four dominate the market: NMC (nickel, cobalt, and manganese), LCO (lithium cobalt oxides), NCA (nickel, cobalt, and aluminum), and LFP (lithium iron phosphate) (Julien et al. 2014). Cathode materials exhibit greater variability than anode materials due to their lower specific capacity (electric capacity per weight or volume), and no single material has been identified as superior. Improving cathode materials is essential for enhancing LIB performance. Consequently, lithium-ion batteries on the market often feature different cathodes, each with unique characteristics (Chen et al. 2012). Socio-economic factors also play a role. While transition

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metal oxide cathodes have higher capacities than LFPs, they require raw materials identified as critical or strategic (Lebrouhi et al. 2022).
The projected growth in demand for lithium-ion batteries (Bajolle, Lagadic, and Louvet 2022) will result in a substantial volume of depleted materials. If improperly managed, these materials could harm the environment. To support a future reliant on these technologies, recycling is imperative, particularly to recover the substantial quantities of critical materials within them (Thompson et al. 2020).
LIB recycling proposals fall into two categories: pyrometallurgical and hydrometallurgical methods (Wei et al. 2023). Hydrometallurgical methods are more promising due to higher efficiency and lower energy consumption and environmental impacts. However, these methods often involve strong inorganic acids (e.g., nitric acid, sulfuric acid, or hydrochloric acid) and hydrogen peroxide as reducing agents, which increase their environmental impact. To mitigate this, studies have explored alternatives using organic acids (Chaudhary et al. 2024; Golmohammadzadeh, Faraji, and Rashchi 2018) and organic reducing agents (Pagnanelli et al. 2014). In all cases, the final product of the treatment is a metal-rich solution, known as leach liquor. This solution can be used to precipitate metals into new chemicals or directly synthesize new cathodic materials through co-precipitation of mixed metals precursors of cathodes, the method used for industrial production of mixed cathodic materials from synthetic salt solutions (Dong and Koenig 2020). Co-precipitation involves two steps: precipitating the electrode precursor via co-precipitation and thermally activating the precursor to incorporate lithium into its crystalline structure. Using leach liquor as raw material for the co-precipitation reaction directly links end-of-life LIB recycling to new battery production. However, the solution's purity limits its direct use. End-of-life batteries contain additional materials (e.g., copper, aluminum, and iron) from current collectors and external casings, which dissolve alongside target metals into the leach liquor. To reuse the solution for new cathodes, many studies employ purification steps, increasing the pH to precipitate impurities (Chen et al. 2015). This process requires adding NaOH, which dilutes the leach liquor and reduces the final metal concentration, but prevents impurities from affecting the final product. Other studies (Jeon et al. 2021; Medvedeva et al. 2022) suggest that impurities, in limited quantities, can act as doping agents, stabilizing capacity retention during cycling. Achieving optimal percentages is key. This study examines the effect of leach liquor composition on the characteristics of the NMC precursor when transitioning from hydrometallurgical treatment directly to co-precipitation. Given the environmental concerns of traditional leaching methods using inorganic acids and hydrogen peroxide, this study investigates innovative leaching procedures. One method uses LFP as a reducing agent (Pagnanelli et al. 2024), while another employs citric acid and glucose. The resulting materials will be characterized for morphology, stoichiometry, and dimension and compared to analogous products derived from synthetic solutions. Additionally, the study will analyze content of impurities in precursors.

2. Materials and methods

2.1 Materials and methods

The first leaching system (leaching system 1) used sulfuric acid and LFP as reducing agent (Pagnanelli et al. 2024). The reactor was initially loaded with the same mass of the two solids: NMC black mass provided by S.E.Val. (Colico, Italy) and LFP scraps kindly furnished by FAAM (Teverola, IT). 1.5 M sulfuric acid (96% for analysis, Carlo Erba) solution was fed while the reactor agitation was fixed at 200 rpm using the AM20-D stirrer by ARGOLAB. The volume of the solution used was chosen to reach a solid to liquid ratio (S/L) of 100 (g/L). The procedure was undertaken at 60°C by controlling the temperature with a thermostatic bath (ECO RE 620, Lauda). The leaching test lasted 3 hours. The slurry obtained from the leaching was filtered under vacuum to separate the residual solid from the liquid one (leach liquor solution with extracted metals). The leach liquor pH is increased up to 2.5 adding a solution 1 M NaOH (EmsureSupelco, Merck) to precipitate iron phosphate, separated by solid/liquid separation. The purified leach liquor is characterized with Inductively-Coupled Plasma Optical Emission Spectrophotometer (ICP-OES AVIO 220 Max, PerkinElmer) to determine the concentrations of metals in solution. The solution is later corrected by adding synthetic metal salts ($NiSO_4$ hexahydrate 98%, Thermo Scientific and $CoSO_4$ heptahydrate, VWR Chemicals) in order to obtain a solution with atomic percentages of 80% nickel, 10% manganese and 10% cobalt. The obtained solution was used to undertake a co-precipitation reaction using 1.5 M oxalic acid (Anhydrous 98%, Thermo Scientific) as precipitating agent. The volume used was measured to have an excess of 10% of oxalic acid with respect to the quantity needed to precipitate all the target metals (nickel, cobalt and manganese). Each precipitation was done starting from 50 ml of leach liquor. The reaction lasted 3 hours. The precipitate and the supernatant were separated after being centrifuged using NEYA 16 centrifuge. The precursor is later dried at 60°C. It will be addressed as P1. The supernatant was analyzed to determine the % metal precipitation yield as oxalate computed as:

$$Me\ precipitation\ (\%) = \frac{m_0 - m}{m_0} * 100 \quad (1)$$

Where m_0 is the initial mass of metals in solution and m is the final. The solid was digested using 4 ml of nitric acid 65% ww (VWR Chemicals, Radnor), 4 ml of hydrochloric acid 37%ww (VWRChemicals) and 2 ml of hydrogen peroxide 30%ww (Merck). Aqua regia was not directly used due to its prolonged operating times and susceptibility to oxidation. The resulting solution was analyzed using ICP-OES to determine the atomic percent composition of the precursors and quantify impurities. Particle size of precursor samples was determined by using laser particle analyzer (Mastersizer 3000, Malvern Panalytical). Particle morphology was analyzed by SEM Phenom Pro X G6.

The second leaching system was made up of citric acid and glucose as reducing agent (leaching system 2). The leaching test was performed using the same solid/liquid ration as before (100 g/L) and NMC black mass as the only solid input material . Then 1.5 M of citric acid solution (citric acid monohydrate 96%, VWR chemicals) was added and stirred at 300 rpm at 90°C. The leaching lasted 1 h, then the slurry was filtered to separate the leach liquor from the solid residue. In this case the leach liquor was directly used to precipitate the NMC precursor after the stoichiometry correction up to a total metal molarity of 2 with 8:1:1 ratio as Ni, Mn and Co. The precipitating agent was oxalic acid fed in the reactor with the same procedure used for the other leach liquor. The material collected was analyzed with the same procedures and it will be addressed as P2.

A third precursor named P3 was produced following the same procedures but starting from a solution 2 M made with synthetic salts of metals.

3. Results and discussion

3.1 The leach liquors

Table 1 shows the compositions of the two leach liquors obtained from NMC-LFP mixture leached with H_2SO_4 after iron phosphate removal (1), and from NMC leached with citric acid and glucose (2).

Table 1: Chemical composition of purified leach liquor using H_2SO_4 and LFP (1) and of the leach liquor using citric acid and glucose (2)

Leaching system	Ni (mmol/L)	Mn (mmol/L)	Co (mmol/L)	Li (mmol/L)	Cu (mmol/L)	Fe (mmol/L)	Al (mmol/L)
1 (H_2SO_4 + LFP)	26.5	30.5	10.6	130.5	1.7	0.4	2.6
2 (Citric acid + glucose)	166.6	55.74	39.3	404.2	4.1	4.3	12.9

The purified leach liquor coming from NMC-LFP mixture (1) presents lower metal concentrations in comparison with NMC leach liquor (2). This is due to the fact that using NMC-LFP mixture with the same solid liquid ratio than using only NMC as solid feed, determined a reduced metal concentration (Ni, Mn, Co) in the solid and then in solution. In addition, the precipitation of iron phosphate was made by adding NaOH solution determining a further metal dilution in the purified leach liquors (1). Finally, in the leach liquor coming from the treatment with LFP, the iron concentration in the solution is reduced by an order of magnitude due to the precipitation step for iron phosphate recovery.

After concentration adjustment, total metal molarity (as the sum of nickel, cobalt and manganese moles divided the volume) is 0.28 M for the purified leach liquor from NMC-LFP leaching, and 2 M in the acid citric-glucose leaching.

3.2 Co-precipitation reaction

Table 2 shows the metal contents (as atomic percentages) in the produced precursors. Precursors from synthetic solution presented the desired stoichiometry NMC 811 with a slight deficiency of manganese. Both precursors from leach liquors of real samples presented some impurities with comparable amounts.

The observed concentrations are in line with doping values proposed in the literature for such metals (Jeon et al. 2021; Medvedeva et al. 2022).

Comparing Ni, Mn and Co concentrations in precursor P2 and P3, there is an effect of the leach system 2 on this variable. The stoichiometry (Ni/Mn/Co) of precursors P1 and P2, eliminating the contribution of impurities, turns out to be: 7.8/1.1/1.1 for sample P1 and 8.3/0.7/1 for sample P2. While P1 has a stoichiometry close to the desired one, P2 turns out to be the precursor with the highest manganese deficiency.

Table 2: Composition of the NMC precursors synthesized.

NMC precursor	Ni (%at)	Mn (%at)	Co (%at)	Li (%at)	Cu (%at)	Fe (%at)	Al (%at)
P1	76.3	10.64	11.33	5.58	0.74	0,03	0.97
P2	81.21	6.93	9.58	1.42	0.16	0.09	0.62
P3	81.25	8.7	10.06	-	-	-	-

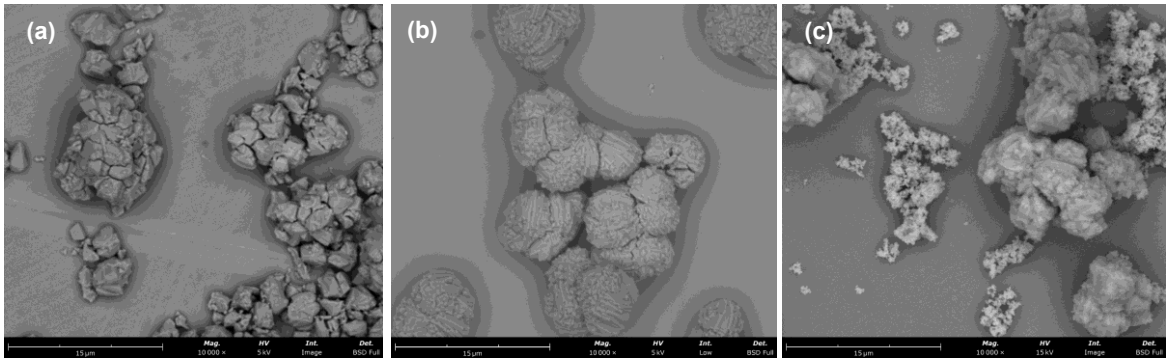

Figure 1: SEM images showing the picture of the three NMC precursors. (a) P1 ; (b) P2; (c) P3.

The main difference between leaching 2 and leaching 3 is the presence of citrate in solution which form highly stable chelates with all target metals (He et al. 2023; Matzapetakis et al. 2000). Chelate formation can negatively affect metal precipitation and according to the experimental findings further depresses the precipitation of the most soluble metal, i.e. manganese. This is in agreement with results from synthetic solution, showing that Mn is slightly under stoichiometric and with thermodynamic speciation in solution confirming that Mn among target metals is the most soluble as oxalate.
Table 3 shows the precipitation yields obtained from analyzing the supernatants. The supernatant from leach liquor 1 exhibits the lowest precipitation percentages, which can be attributed to the low metal concentrations caused by the dilution from NaOH addition during iron phosphate removal. In contrast, leach liquor 2 has initial concentrations equivalent to those of the synthetic solution. This enables the complete precipitation of nickel and cobalt; however, the presence of manganese citrate limits manganese precipitation to 75%.

Table 3: Metal precipitation (%) as oxalate using the different leach liquors

Leach liquors	Ni (%)	Mn (%)	Co (%)
1	55	33	67
2	99	75	97
3	100	100	100

Figure 1 reports SEM micrographs of the three precursors produced from the three solutions. P1 precursors evidenced a distinct morphology (Figure 1a) probably due to the lower initial concentration in solution negatively affecting nucleation and then depressing further aggregation phenomena (Mullin 2001). P2 and P3 denote similar structures characterized by aggregates of plate-like substructure with spherical shape typical of such precipitates (Dong et al. 2018). This finding evidenced that even in presence of citrate the mechanism of nucleation, growth and aggregation followed the same path giving the same morphology of particles.
Particle size was further addressed by laser size analyzer showing that particle precursors produced with leaching 1 (at low initial metal concentration) denoted reduced dimensions and heterogeneous distribution of

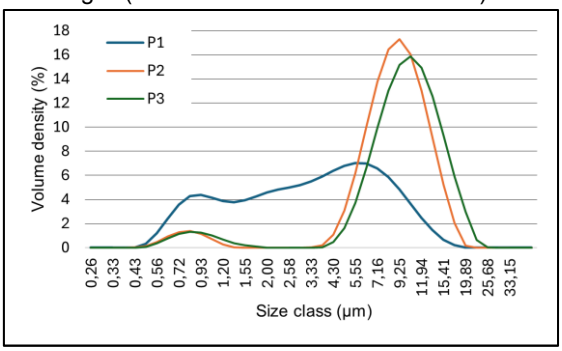

Figure 2: Measurement of the particle size distributions taken with laser size analyzer.

size. According to particle size again no relevant difference is denoted for leaching 2 in comparison with synthetic solution denoting that neither citrate nor impurities negatively affect the size and homogeneity of particles.

4. Conclusions

The study highlighted how metal composition of the initial solution used for precursor precipitation can affect precipitation yield, chemical composition, morphology and size of precursors.
Metal composition in precursors can be affected by the presence of citrate in solution depressing the precipitation of the most soluble metal, Mn.
Target metal initial concentration can affect particle size distribution and morphology: both these characteristics strongly affect cathodic performance in batteries reducing tap density of particles and then energy density of the final equipment.
Metal impurities in the precursors are in line with concentration studied as doping one in cathodic production but further characterization are required to confirm the effect of residual Cu, Fe and Al in the cathodes.
Further studies are needed to improve both processes. Leaching with LFP must achieve higher metal concentrations before precipitation, while for citric acid and glucose, the issue of citrate complexation must be resolved to prevent excessive manganese deficiency.

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