

Separation of manganese from cobalt by solvent extraction: Application to the recycling of spent cathode material from lithium-ion batteries

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Lithium-ion batteries are becoming ubiquitous as mobile power sources, particularly as the global adoption of electric vehicles intensifies. Currently batteries typically use a lithium nickel manganese cobalt oxide as the cathode and graphite as the anode. Recycling of the valuable components of spent electrode materials at the end of product life is imperative for closing the resource loop. This paper evaluates the solvent-extraction separation of manganese from cobalt from synthetic leach liquor simulating the sulfuric acid dissolution of spent cathode material. Most testwork on this separation to date has focussed on dilute leach solutions that arise from primary hydrometallurgical applications; in this case, however, the leach solutions contain valuable metals at close to their solubility limit, which influences both the chemical and physical nature of the reaction systems. Batch testwork results for manganese separation from cobalt using di(2-ethylhexyl)phosphoric acid are presented. The effects of extractant concentration, temperature, and neutralising agent were evaluated. Preliminary staging requirements, based on McCabe–Thiele isotherm data, are discussed.

INTRODUCTION

With the global focus on reducing energy use and creating sustainable technology solutions, many parts of the world are moving towards increased use of electric vehicles (Barton & Schütte, 2016). These are powered by lithium-ion batteries. The compound growth in demand for lithium-ion batteries (in GWh) is predicted to increase by 11% pa from 2019 to 2024 (Zhao *et al.*, 2021). The most widely used battery formulation comprises a lithium nickel manganese cobalt oxide ($\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$) as the cathode and graphite as the anode (Buchmann, 2017). A recent prediction indicated that cobalt demand is expected to rise fourfold and nickel by more than an order of magnitude for the decade to 2028, as illustrated in Figure 1. Owing to the long lead times required for exploration and mine development, new primary production of these metals is unlikely to meet this demand. It is therefore imperative to consider alternative sources of these valuable metals. One approach is to recycle spent cathode material from spent lithium-ion batteries.

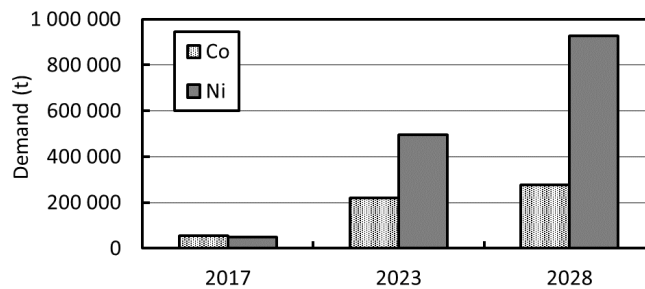


Figure 1. Predicted demand for cobalt and nickel for use in lithium-ion batteries (from Rawles, 2017).

Original cathode formulations used a 1:1:1 molar ratio of Ni:Mn:Co, the so-called NMC chemistry. About 70% of world primary cobalt supply originates in the Democratic Republic of Congo (Garside, 2022), of which about 20% is sourced from individual artisanal miners (Brown, 2018). Owing to unpredictable political and policy stability and to reduce this heavy reliance on a single country (Shedd *et al.*, 2017), cathode chemistry is moving towards use of lower proportions of cobalt. More recently, 5:3:2, 6:2:2, and 8:1:1 NMC formulations have been commercialised (Buchmann, 2017), and 9:1/2:1/2 chemistry is under development. This evolving battery chemistry means that the composition of the cathode material will change with time. When the batteries reach the end of their useful life, the spent cathode material, the so-called black mass, will have a similarly evolving composition. Recycling of the valuable components of the spent cathode material at the end of product life is imperative for closing the resource loop.

Any process route for recycling of black mass associated with different battery chemistries must be able to handle different feed compositions with minimal process disruption. One way to achieve this is to separate the individual major components of the spent battery material into pure chemicals and then reformulate these according to current battery specifications.

Many flowsheets have been proposed in recent years for treating black mass to produce battery-grade nickel, manganese, and cobalt chemicals (for example: Chen *et al.*, 2015; Gaines, 2018; Xu *et al.*, 2008; Yang *et al.*, 2017; Zeng *et al.*, 2014; Zubi *et al.*, 2018). A simplified typical generic flowsheet is given in Figure 2. Following separation of the spent cathode material from the used battery by physical and mechanical means, the black mass is subjected to sulfuric acid leaching under oxidative or reducing conditions (or both), depending on the chemistry and oxidation states of the various components. Copper can selectively be removed from the pregnant leach solution (PLS) by solvent extraction (SX) using conventional techniques. Species such as iron and aluminium can be removed by precipitation, as in many conventional sulfate-based hydrometallurgical processes. Subsequent steps are required to separate the Ni, Mn, Co, and Li, and recover these as pure chemicals. Using the well-known orders of selectivity of commercially available SX reagents, Mn can first be extracted using di(2-ethylhexyl)-phosphoric acid (D2EHPA), followed by Co extraction using di(2,4,4-trimethylpentyl)phosphinic acid (TMPPA; exemplified by CYANEX 272 or Ionquest 801), and then Ni extraction using Versatic 10 carboxylic acid, leaving Li in the final raffinate to be recovered as Li_2CO_3 or $\text{LiOH}\cdot\text{H}_2\text{O}$.

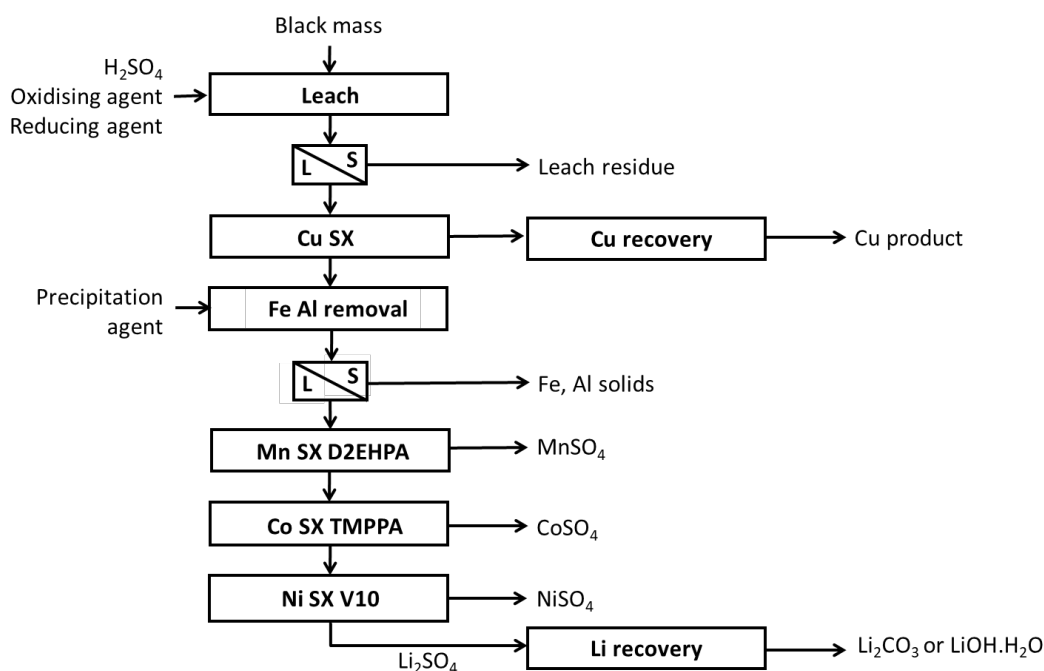


Figure 2. Simplified generic flowsheet for recovery of nickel, manganese, cobalt, and lithium from spent lithium-ion battery cathode material.

This study evaluated the SX separation of manganese from cobalt in a synthetic leach liquor simulating typical concentrations of these metals resulting from the sulfuric acid dissolution of spent cathode material. Most testwork on the Mn/Co separation to date has focussed on relatively diluted leach solutions that arise from primary hydrometallurgical applications (1–5 g/L) and the leaching of ores (see for example: Baba *et al.*, 2014; Cheng, 2000; Darvishi *et al.*, 2011; Feather *et al.*, 2000; Hoh *et al.*, 1984; Zhang and Cheng, 2007); in this case, however, the leach solutions contain the valuable metals at close to their solubility limit (~ 100 g/L), which changes both the chemical and physical nature of the reaction systems. Testwork results and preliminary staging requirements for manganese separation from cobalt using D2EHPA are presented for this application.

EXPERIMENTAL

Synthetic aqueous phases were prepared using chemically pure sulfate salts of Co and Mn. Metal concentrations were selected assuming quantitative leaching of the valuable metals from a 1:1:1 NMC black mass at a solids' content of 10%. The molar masses of the two elements are very similar, so the aqueous phases were made up to 50 g/L Co and 50 g/L Mn. The initial acidity was adjusted to pH 1 using H₂SO₄. The organic phases were made up using D2EHPA, supplied by Chemquest, in BP mining solvent, a partially aromatic (~ 18%) diluent, supplied by Petronex.

Extraction was determined as a function of pH by contacting 100 mL of the aqueous phase with 200 mL of the organic phase by magnetic stirring in a water-jacketed beaker with temperature control using a water bath. The pH of the emulsion was adjusted by dropwise addition of 10 M NaOH, 10 M KOH, or 5 M LiOH. Once equilibrium was achieved at a defined pH, the phases were allowed to separate and 5 mL of the aqueous phase was removed for analysis. A 10 mL organic sample was also removed to maintain the same organic-to-aqueous (O:A) phase ratio throughout the experiment. The remaining volumes of the two phases were again contacted by magnetic stirring and the equilibrium pH of the emulsion adjusted to a new value. Samples were collected at values ranging from pH 1 to pH 5 in increments of approximately 0.5 pH units. The extraction at each pH value was determined from the difference between the feed and aqueous sample concentrations.

Once appropriate pH conditions were selected to maximise Mn extraction and minimise Co co-extraction, an extraction isotherm was prepared to determine the number of stages that would be required to achieve a target Mn concentration in the raffinate. A range of O:A values from 10 to 0.1 were selected, using different relative volumes of the aqueous and organic phases. These were contacted at a defined pH to achieve equilibrium distribution of the two metals. The phases were then separated: the aqueous and organic phases were assayed.

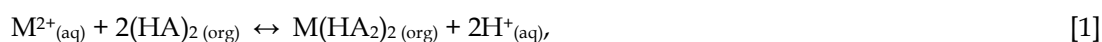
All aqueous- and organic-phase samples were analysed for metal content by inductively coupled plasma optical emission spectroscopy by CM Solutions.

RESULTS

Extraction as a function of pH

Effect of extractant concentration

Extraction of divalent base-metal cations (M^{2+}) by D2EHPA (HA) is assumed to take place according to:



where D2EHPA is considered to exist in dimeric form in these systems. For qualitative extraction of 50 g/L Mn, 0.34 mol/L D2EHPA is required on a stoichiometric basis. To ensure that sufficient D2EHPA was available to fully load all Mn, an O:A of 2 was used for the batch extraction experiments.

Figure 3 shows the effect of D2EHPA concentration on the extraction of Mn and Co as a function of pH. It is evident that extraction of both Mn and Co shifted to lower pH (or increased at a given pH) with increasing D2EHPA concentration, although there was no significant difference in performance above 2.26 M D2EHPA. At the higher extractant concentrations, phase separation was severely affected, so such concentrations would be impractical in practise. It would be better to employ a lower extractant concentration in the organic phase and higher O:A to provide sufficient extractant capacity for quantitative extraction of Mn.

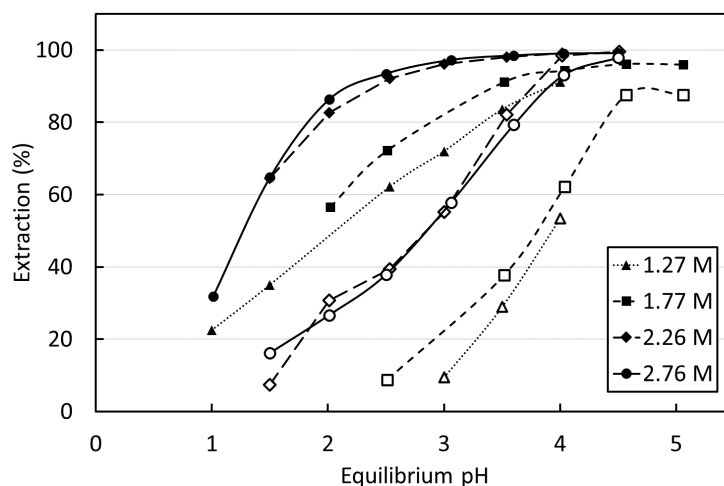


Figure 3. Effect of D2EHPA concentration on pH dependence of Mn (closed symbols) and Co (open symbols) extraction at O:A = 2 at 25°C.

A quantitative means to evaluate the extent of Mn/Co separation is by comparing the difference in pH at 50% extraction of each metal (ΔpH_{50}). The data, shown in Table I, indicate that the best separation was achieved at 2.26 M D2EHPA, although 1.76 M D2EHPA would also be suitable.

Table I. ΔpH_{50} for Mn/Co separation as a function of D2EHPA concentration in the organic phase

[D2EHPA] (M)	1.27	1.76	2.26	2.77
pH_{50} Mn	2.20	1.72	1.20	1.24
pH_{50} Co	3.8	3.8	3.33	2.58
ΔpH_{50}	1.60	2.07	2.13	1.34

Effect of temperature

The effect of temperature on the Mn/Co separation is shown in Figure 4. Mn extraction was essentially unaffected by temperature, but there was a small effect on Co, with higher extraction at a given pH with higher temperature. Lower temperature therefore favoured extraction of Mn while minimising Co co-extraction.

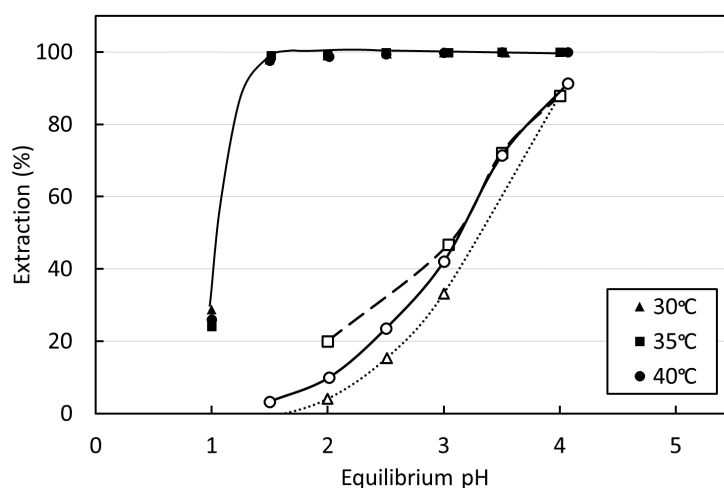


Figure 4. Effect of temperature on pH dependence of Mn (closed symbols) and Co (open symbols) extraction by 2.26 M D2EHPA at O:A = 2

Effect of neutralising agent

As shown by Equation [1], protons are generated by the extraction reaction when using the extractant in the acidic form. Because extraction decreases with a decrease in pH, it is necessary to neutralise these protons to avoid losing extraction capacity. In most hydrometallurgical applications, NaOH (or sometimes NH_4OH) is used for this purpose. The cation of the neutralising agent departs to the SX raffinate. In a flowsheet such as shown in Figure 2, Na^+ would ultimately depart with the Li product, from which Na_2SO_4 would be generated as a byproduct. There is currently a global excess of this chemical and it would not easily be sold, so alternatives were considered. In this work, KOH and LiOH were evaluated as alternative neutralising agents. K_2SO_4 can be sold into the fertiliser industry, while Li_2SO_4 can contribute to the final lithium product and would not introduce an additional impurity element into the system.

Figure 5 compares the extraction curves for Mn and Co using NaOH, KOH, and LiOH as neutralising agents. Mn extraction showed a slight dependence on neutralising agent, but Co extraction, particularly above 70% extraction, showed a negligible difference.

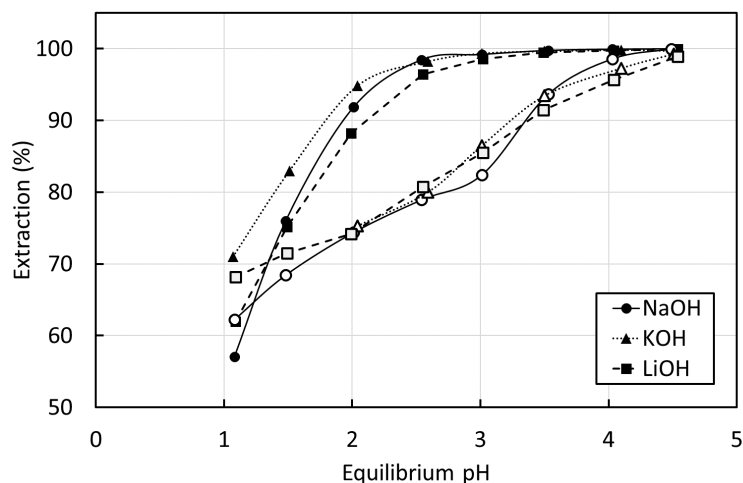


Figure 5. Effect of neutralising agent on pH dependence of Mn (closed symbols) and Co (open symbols) extraction by 3 M D2EHPA at 25°C and O:A = 2.

To confirm whether there was any difference in neutralisation capacity of these three reagents, standardised solutions were used to titrate the acidic proton on D2EHPA (and the two acidic protons on the trace mono-acid impurity present in the as-received chemical), as shown in Figure 6 and Equation 2:



where R = 2-ethylhexyl.

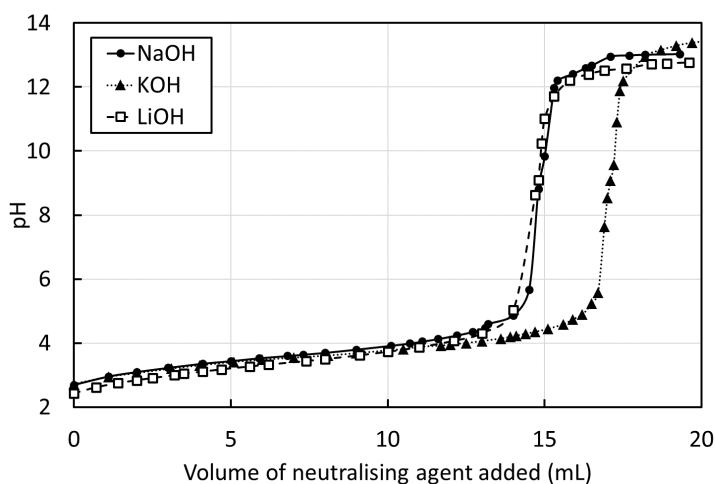


Figure 6. Potentiometric titration of 1.00 g D2EHPA by 0.1 M NaOH, KOH, and LiOH in 1:1 propanol:deionised water.

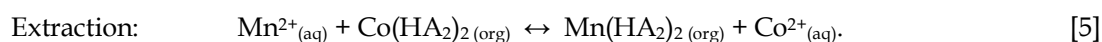
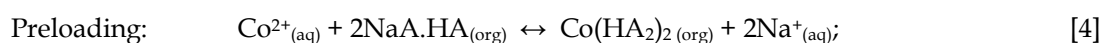
As shown in Figure 6, LiOH displayed very similar behaviour to NaOH; KOH proved to be weaker, requiring a greater volume of reagent to achieve the same extent of neutralisation. The relevant pK_b

values are listed in Table II. The order of basicity for the same aqueous molarity increases in the order of atomic mass of the cation: LiOH < NaOH < KOH.

Table II. pK_b values for bases used as neutralising agents in D2EHPA extraction

Base		LiOH	NaOH	KOH	NH ₄ OH
pK_b		-0.36	0.2	0.5	4.7

These results showed that LiOH or KOH could be used to replace NaOH as the neutralising agent, albeit with slight dilution of the raffinate. Both reagents are, however, more costly than NaOH. Another approach to minimise/eliminate the requirement for a neutralising agent is to preload the extractant with an appropriate less-selective cation, which would then be displaced by the target cation during the main extraction reaction. In this case, Co could be preloaded onto the partially saponified extractant, and would be displaced to the raffinate by the more-selective loading of Mn, as shown in the simplified reactions below. This does not introduce any foreign cations into the aqueous raffinate that then becomes the feed to the downstream Co SX circuit (Figure 2).



Extraction isotherm and McCabe–Thiele construction

A loading isotherm was prepared using the optimum conditions identified from the batch testwork, as shown in Figure 7. A temperature of 30°C and equilibrium pH of 2.5 were employed. The organic phase comprised 2.26 M D2EHPA in BP mining solvent.

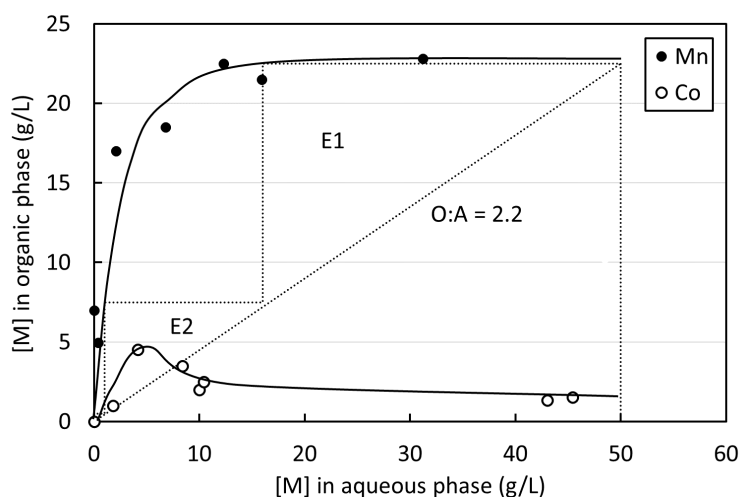


Figure 7. Extraction isotherm for loading of 50 g/L Mn (closed symbols) and 50 g/L Co (open symbols) by 2.26 M D2EHPA at pH 2.5, 30°C. McCabe–Thiele construction uses an advance O:A of 2.2.

Displacement of Co from the extractant as the loading of Mn increases is apparent. It is notable that the maximum organic-phase loading obtained in this work was ~ 22 g/L Mn (0.37 M). The D2EHPA:Mn molar ratio is therefore approximately 6:1. This is in contrast to the 4:1 ratio expected from Equation 1, which has been postulated for low Mn PLS and extractant concentrations. It is possible that different

complex stoichiometry occurs under these high metal and extractant conditions. The high viscosity of the organic phase under these conditions may also influence the ease of complex formation and transfer of Mn^{2+} cations across the aqueous–organic interface. Further experimentation is required to clarify the reasons for this observation.

A McCabe–Thiele construction is superimposed on the isotherm, based on an initial aqueous phase containing 50 g/L Mn and an advance O:A flowrate ratio of 2.2. The construction shows that at least three extraction stages will be required to recover the bulk of the Mn. Detail of the low-concentration region was not available from this testwork; however, it is likely that additional stages will be required to ensure that the Mn in the raffinate does not exceed the target value for the downstream Co product: Mn will be co-extracted with Co when using TMPPA in the Co SX circuit (Figure 2). An indicative typical specification is < 20 g/t Mn in $CoSO_4 \cdot 7H_2O$ (Sole, 2018).

This isotherm was prepared with the equilibrium pH controlled to pH 2.5. In actual practise, it would be more typical to use a varying pH profile across the extraction circuit, with a higher pH in the last extraction stage (where the stripped organic phase enters the circuit) to promote loading of residual Mn from the raffinate, and a lower pH in the first extraction stage, where high loading of Mn is driven by the high Mn concentration in the incoming PLS and to reduce co-extraction of Co on the loaded organic phase that leaves the extraction circuit. This work has demonstrated that it is difficult to achieve completely selective Mn removal from Co under these experimental conditions: it is likely that some Co will deport on the loaded organic phase. A series of pH-controlled scrubbing stages are also required to remove this co-loaded Co to ensure that there are no Co losses from the circuit with the Mn product stream.

DISCUSSION

The well-known order of selectivity for extraction of relevant cations by D2EHPA is: $Fe(III) > Al \sim Zn > Ca > Mn > Cu > Co > Ni > Mg > Li$. It is therefore necessary to remove cations that extract more strongly than Mn from the black mass PLS prior to recovery of Mn if a pure Mn salt product is required. It is also necessary to remove Cu from the PLS, which can be readily achieved using conventional extractants and SX operating conditions. Target specifications for battery-grade Mn, Co, and Ni chemicals require typically < 1 g/t of most base-metal impurities (Sole, 2018). SX is usually not able to achieve such tight specifications owing to carryover of impurities by both chemical co-loading and physical entrainment. Depending on the system, some selectivity may be achieved in a crystallisation process. The need for ion exchange for final polishing of these liquors is likely. In this particular case, for example, quantitative Cu removal in an upstream SX circuit cannot be reliably achieved: removal of residual Cu from the Mn stream by ion exchange, typically using an iminodiacetic-acid resin functionality, is one approach to achieving the required product purity.

This work focussed only on separation of two major components of the spent cathode material. It is important to note, however, that these battery materials are hugely complex and contain trace amounts of a wide range of other elements, including rare-earths and various amphoteric elements that are likely to form anionic oxy-species in acidic sulfate media. Depending on the source of the batteries for recycling, there may also be contamination of the feed by other non-lithium battery types, which could introduce elements such as Zn, Cd, and Pb into the leach liquor. Deportment of all of these species must be considered in design of a flowsheet to achieve battery-grade chemicals from this secondary source.

CONCLUSIONS

In a generic hydrometallurgical flowsheet for the leaching of spent lithium-ion battery NMC cathode material followed by SX separation and recovery of the valuable components, Mn can be extracted from Co (and other valuable and impurity elements) using D2EHPA. Under the high metal concentrations (total ~100 g/L) generated in these systems, the ease of chemical separation and physical phase disengagement is more difficult than in conventional primary applications, in which the metal concentrations are approximately an order of magnitude lower. This batch testwork, using synthetic solutions containing only Mn and Co, yielded the following conclusions:

- Mn is more selectively extracted (i.e., extracted at lower pH) than Co by D2EHPA and can be quantitatively extracted even at concentrations as high as 50 g/L Mn, provided sufficient extractant is available;
- As expected from the equilibrium of the extraction reaction, extraction of both metals shifts to lower pH with increasing extractant concentration; however, Mn extraction is less dependent on extractant concentration than Co;
- Extraction of Co is slightly enhanced by an increase in temperature to 40°C, indicating that the extraction reaction is endothermic;
- Use of NaOH as a neutralising agent can be substituted by KOH or LiOH, to produce a more saleable sulfate byproduct or avoid contaminating downstream process streams by foreign cations, albeit at increased reagent cost;
- At least four stages of counter-current extraction are required to achieve a raffinate containing < 1 mg/L Mn. Cobalt is squeezed off the extractant as Mn loading increases.

This work demonstrates that leach solutions with high metal concentration can be effectively processed using conventional extractants, provided optimum operating conditions are ensured. In treatment of real leach solutions, the behaviour of other trace impurities that may also co-extract with Mn (such as Fe, Al, Cr, Zn, Ca, Zr, Ti, Cu, etc.) should also be taken into account: it may be necessary to process the purified Mn loaded strip liquor by ion exchange or selective crystallisation to ensure that the final product meets battery-grade specifications. In addition to use of a relatively high number of extraction stages and controlled pH of extraction, it is likely that pH-controlled scrub stages will also be necessary to fully remove any co-extracted Co from the organic phase before stripping, to avoid loss of valuable Co with the less-valuable Mn product.

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Kathy obtained her PhD from the University of Arizona (USA) and holds a part-time appointment in the Department of Materials Science and Metallurgical Engineering at University of Pretoria. Her main areas of professional expertise are in solvent extraction, ion exchange, and electrowinning, and she has worked across a wide range of commodities, including copper, cobalt, nickel, zinc, uranium, titanium, vanadium, platinum-group metals, and gold. Her current research focusses on producing high-purity lithium-ion battery salts from both primary and recycled sources, and purification of locally produced electrolyte for use in vanadium redox flow batteries.

Kathy received the 2019 Milton E. Wadsworth award for contributions in non-ferrous chemical metallurgy from the Society of Mining, Metallurgy and Exploration (USA). She represents Southern Africa on international committees for solvent extraction, copper, and ion exchange, and chairs the Copper Cobalt Africa conference series. Kathy is a Fellow of SAIMM and the South African Academy of Engineers.

