

The Role of UV-Vis and ICP-OES Spectroscopy in Lithium-Ion Battery Recycling Methods

Supporting a one-step hydrometallurgical recycling method for leaching metals from cathode materials



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Abstract

The growing demand for lithium-ion batteries (LIBs) highlights the need for sustainable recycling solutions. This study explores a one-step hydrometallurgical process using contact-electro-catalytic (CEC) technology, which integrates pretreatment and leaching to recover critical metals such as Li, Co, Ni, and Mn. This eco-friendly approach employs ultrasonic-assisted leaching with SiO_2 as a recyclable catalyst, enhancing metal dissolution through electron transfer.

UV-Vis spectroscopy and inductively coupled plasma optical emission spectroscopy (ICP-OES) play a key role in optimizing and monitoring the process. UV-Vis enables real-time tracking of radical activity and metal dissolution, while ICP-OES ensures precise quantification of metal recovery and impurity levels. The results confirm that CEC-assisted recycling achieves high metal recoveries from spent cathode materials, offering a cost-effective and sustainable alternative to conventional methods.

Introduction

LIBs have powered portable electronics for decades. However, the growing demand from the electric vehicle and energy storage sectors has raised concerns about managing the increasing volume of batteries reaching the end of their functional life. While the disposal of LIBs presents significant environmental challenges, it also offers opportunities for recovering valuable materials, paving the way for more sustainable practices.

LIB manufacturers rely on a steady supply of critical elements such as lithium (Li), cobalt (Co), nickel (Ni), and manganese (Mn) for use in key battery components such as the cathode. Recycling spent LIBs and recovering metals helps meet the growing demand for these resources.

Traditional methods for recycling spent LIBs rely on multistep hydrometallurgical or pyrometallurgical processes that are costly, energy-intensive, and environmentally hazardous.^{1,2} A more sustainable and cost-effective hydrometallurgical recycling method has been developed, which simplifies the conventional multistep approach into a single step by integrating the pretreatment and leaching stages. This method employs contact-electro-catalytic (CEC) technology, a green catalytic process that uses ultrasonic-assisted metal leaching with silicon dioxide (SiO_2) as a recyclable catalyst.^{3,4} CEC processes use electron transfer during liquid-solid contact electrification to generate free radicals to catalyze chemical reactions.

Analytical techniques such as UV-Vis spectroscopy and ICP-OES provide valuable data that can be used to optimize and monitor the efficiency of the CEC process. These techniques are essential for measuring radical generation, monitoring metal dissolution, evaluating process performance, and verifying the purity of any recovered materials.

The CEC recycling process

The CEC recycling process first involves the separation of the main components from spent LIBs. Metals can then be leached from cathode materials such as lithium cobalt (III) oxide (LCO) or lithium nickel manganese cobalt oxides (NCM). As outlined in Figure 1, the cathode material is mixed with organic acids such as malic acid ($\text{C}_4\text{H}_6\text{O}_5$) or citric acid ($\text{C}_6\text{H}_8\text{O}_7$), a SiO_2 catalyst is added, and ultrasound is applied for approximately 6 hours of reaction time.^{3,4} UV-Vis spectroscopy is an ideal technique for monitoring CEC-generated radicals and optimizing leaching conditions. The extracted metals are then processed back into LiCoO_2 , NCM622, and NCM712 cathode powders using precipitation methods or sol-gel synthesis and high-temperature calcination at 850 to 900 °C, respectively. ICP-OES is widely used to determine metals and other elements in LIB battery materials, including regenerated cathode active materials.⁵ Finally, the SiO_2 catalyst can be recovered by filtration.

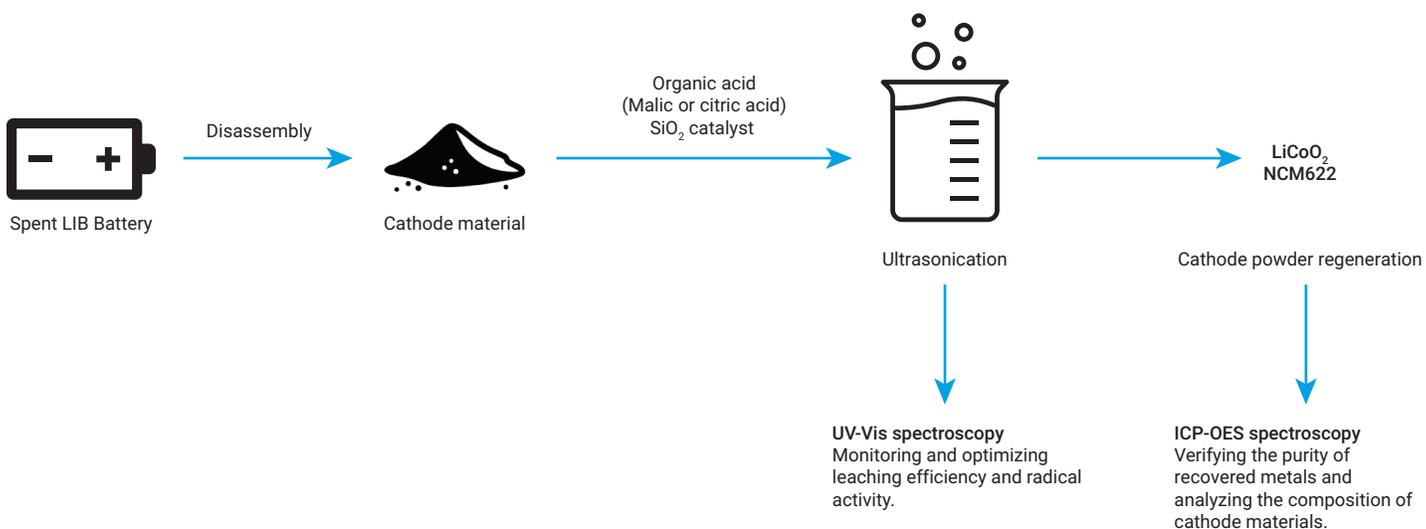


Figure 1. Process flow diagram: illustrating the roles of UV-Vis and ICP-OES in the CEC-recycling workflow.

Experimental

Materials and sample preparation

Waste cathode sheets of NCM were collected separately from battery recycling plants in China. The NCM cathode sheets were cut into 4 × 4 cm pieces. One piece of sheet was added to a glass bottle with 99.99% SiO₂ (Macklin, China) and 40 mL of 99.5% malic acid (Macklin). The bottle was placed in an ultrasonic bath (40 kHz, 300 W) for 6 hours at 80 ± 2 °C facilitating simultaneous metal leaching and degradation of the organic binder.

UV-Vis spectroscopy analysis

An Agilent Cary 3500 Multicell Peltier UV-Vis spectrophotometer (Figure 2) with Agilent Cary UV Workstation was used to monitor metal-ion release and radical generation. Spectral analysis focused on the detection of metal ions and free radicals that facilitate metal dissolution. The following specific absorption peaks were tracked to confirm leaching: 390 nm for Ni²⁺, 508 nm for Co²⁺, and 660 nm for Mn²⁺.

Radical scavenger tests (using *p*-benzoquinone, silver nitrate (AgNO₃), and *tert*-butanol ((CH₃)₃COH)) confirmed the presence of superoxide, and electron and hydroxyl radicals, which are crucial for binder oxidation and metal reduction. UV-Vis spectroscopy provides real-time monitoring of radicals that promote leaching efficiency. Its non-destructive nature makes it ideal for continuous process control.

ICP-OES analysis

An Agilent 730 ICP-OES, which had been superseded by an Agilent 5800 Vertical Dual View (VDV) ICP-OES, was used to evaluate metal recovery efficiency. ICP-OES enables the detection of multiple elements with high sensitivity, providing precise quantification of metal concentrations. A high level of accuracy is needed to ensure that recovered materials meet industry standards. The ICP-OES instrument was calibrated using certified standards to ensure accurate quantification. The concentrations of Li, Ni, Co, and Mn in the leachates were measured to assess leaching performance and impurity levels.



Figure 2. An Agilent Cary 3500 Multicell Peltier UV-Vis with the Multizone software add-on; up to four independent temperature zones can be configured, allowing simultaneous experiments at different temperatures within each cuvette pair.

Results and discussion

UV-Vis spectroscopy: monitoring leaching efficiency and radical activity

To recycle spent LIBs and separate metals from other components such as organic binders like polyvinylidene difluoride (PVDF), superoxide (O₂^{-•}) and hydroxyl (OH•) radicals need to be generated. These reactive species play a key role in organic degradation and metal reduction. Hydroxyl radicals can recombine to form hydrogen peroxide (H₂O₂), further facilitating metal leaching and PVDF oxidation.

Reactive species such as superoxide can be detected using nitro blue tetrazolium (NBT), which changes color from yellow to blue/purple upon reaction, forming formazan. The intensity of this color shift correlates with the amount of superoxide present, making UV-Vis spectroscopy an ideal technique for monitoring these reactions and optimizing leaching conditions.

To analyze the metal-leaching efficiency of the NCM CEC workflow, UV-Vis was used to investigate the following key parameters:

- **SiO₂ catalyst:** As shown in Figure 3A, SiO₂ significantly improves leaching efficiency by facilitating electron transfer under ultrasound, leading to the formation of active species such as H₂O₂ that promote metal reduction. The optimal SiO₂ amount was 80 mg, as excess catalyst led to ultrasound scattering and reduced efficiency (Figure 3B).
- **Acid concentration:** Increasing the concentration of malic acid from 0.2 to 1 M boosted efficiency by enhancing the reaction gradient (Figure 3C). Higher acid concentrations reduced efficiency due to ion screening effects.
- **Temperature:** Since the reaction is endothermic, higher temperatures favor leaching. As shown in Figure 3D, 80 °C was identified as the optimal temperature.
- **Reaction time:** Peak intensities for Mn²⁺ and Co²⁺ stabilized after 5 hours, while Ni²⁺ peaked at 6 hours (Figure 3E), making this the ideal reaction duration.
- **UV-Vis monitoring:** Peaks at 390, 508, and 660 nm correspond to Ni²⁺, Co²⁺, and Mn²⁺, respectively, allowing real-time tracking of metal leaching.

Under optimized conditions (80 mg SiO₂, 1 M malic acid, 10 g/L solid-liquid ratio, 80 °C for 6 hours), the leaching efficiency achieved for NCM was 99.6% (Li), 98.3% (Ni), 99.4% (Co), and 97.4% (Mn). These results demonstrate the effectiveness of the one-step CEC method for LIB recycling.

The results also demonstrate the flexibility of the Cary 3500 Multicell Peltier UV-Vis spectrophotometer for the thorough study of reaction kinetics under different conditions. Benefits of the instrumentation for this application included:

- A "multizone" capability that streamlined data collection, allowing faster and more efficient analysis compared to single-cuvette instruments. The Cary 3500 Multizone enabled rapid analysis of seven samples in approximately 15 seconds, significantly accelerating the evaluation of leaching effectiveness across multiple samples.
- A simplified experimental design that enabled the identification of optimal conditions and timings of complex species formation.
- Accurate temperature control from –5 to 110 °C without water, noise, or messy cables, thanks to a fully integrated air-cooled Peltier system. This system maintains stable temperatures across eight cell positions within four zones for consistent and reliable performance.

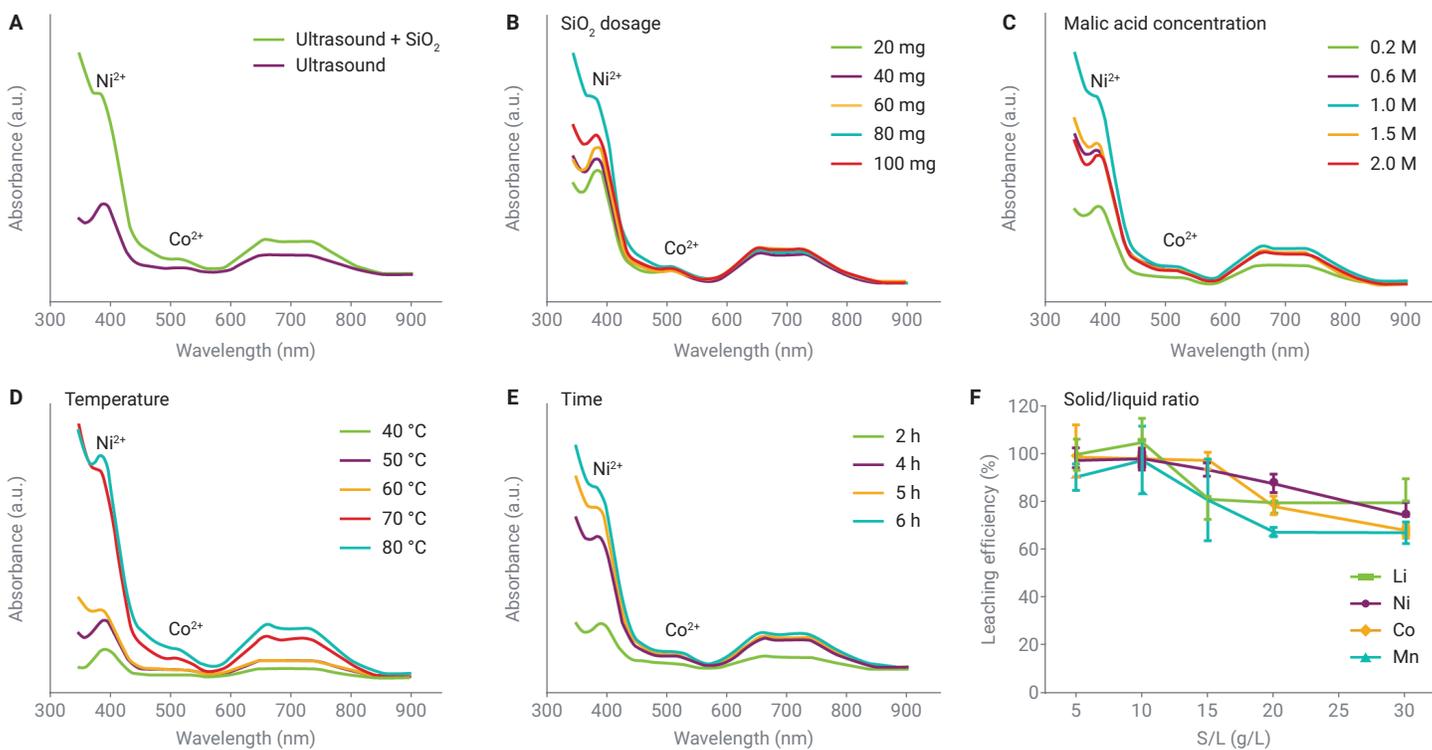


Figure 3. UV-Vis spectra illustrating metal-ion absorption peaks under varying conditions for optimizing CEC leaching efficiency, including (A) SiO₂ catalyst addition, (B) SiO₂ dosage, (C) malic acid concentration, (D) temperature, and (E) time. (F) The solid-liquid ratio, which was optimized using ICP-OES.³

ICP-OES: High recovery rates and purity assessment

ICP-OES analysis demonstrated high metal recovery efficiencies under optimized conditions, as follows: 99.6% (Li), 98.3% (Ni), 99.4% (Co), and 97.4% (Mn). Additionally, to further confirm that the synthesized electrode powder corresponds to NCM622, the elemental ratio of lithium, nickel, manganese, and cobalt in the synthesized powder was analyzed. As shown in Table 1, the molar ratio of Li:Co:Ni:Mn was 10.1:2.1:6:2, which aligns with the compositional requirements for NCM622.

Table 1. ICP-OES data. Concentration analysis of the regenerated NCM.

Metal	Li	Co	Ni	Mn
Concentration (mmol)	5,036.8	1,067.3	2,992.3	997.4

Trace-level analysis was performed using ICP-OES to measure the concentrations of Al, Ca, Mg, Na, Li, Co, Ni, and Mn in the leachate, as shown in Table 2. The results indicate that the concentrations of metal impurities such as Al, Ca, Mg, and Na were below the standard values (0.03%). These results highlight the efficiency and eco-friendly nature of the CEC recycling process, as well as its ability to recover high-purity materials suitable for reuse in the production of new LIB materials.

Table 2. Quality analysis of regenerated NCM622 using ICP-OES.

Metal	Mass fraction (wt%)
Al	0.009700
Ca	0.002267
Mg	0.003405
Na	0.000533
Li	0.062680
Co	0.102300
Ni	0.293200
Mn	0.090700

The ability of ICP-OES to measure multiple elements simultaneously streamlines quality control testing, ensuring that recovered materials meet battery-grade specifications.

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Conclusion

This study highlights the critical role of UV-Vis spectroscopy and ICP-OES in optimizing and monitoring contact-electro-catalytic (CEC)-assisted one-step hydrometallurgical recycling processes, supporting a circular economy for lithium-ion batteries. UV-Vis provided real-time tracking of metal dissolution and radical activity, while ICP-OES ensured precise quantification of metal recovery efficiency and metal impurity levels. The results confirm that eco-friendly, cost-effective CEC recycling methods can achieve high metal recoveries from spent cathode active materials (CAMs) such as LiCoO₂ and NCM, ensuring material purity of regenerated CAMs.

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Further information

- Cary 3500 Multicell UV-Vis spectrophotometer
- Cary UV Workstation software
- UV-Vis Spectroscopy and Spectrophotometer FAQs