ANALYTICAL TECHNIQUES APPLIED TO THE CHARACTERIZATION OF CATHODE MATERIALS IN LITHIUM-ION BATTERY RECYCLING PROCESSES

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ABSTRACT: The growing use of lithium-ion batteries (LIBs) in stationary and vehicular applications demands sustainable recycling solutions. A key challenge is the proper characterization of cathode materials, essential for waste sorting and quality control of recovered products. This article reviews the main analytical techniques applied to physicochemical, structural, and morphological characterization in recycling, including XRD, ICP-OES, XRF, and SEM-EDS. Their fundamentals, applications, and limitations are discussed, with emphasis on phase identification, stoichiometry, and morphological integrity in direct recycling routes. Challenges of standardization, industrial validation, and integration with artificial intelligence are also addressed, reinforcing that advanced characterization is vital for efficient and scalable LIB recycling.

KEYWORDS: Lithium-ion batteries; material characterization; analytical techniques; recycling; cathode regeneration.

1. INTRODUCTION

The exponential growth in the production of lithium-ion batteries (LIBs), driven mainly by the sectors of electric mobility, portable electronic devices and renewable energy storage systems, has intensified the concern with the environmentally safe and economically viable destination of these devices at the end of their useful life. It is estimated that, by 2030, the amount of LIBs discarded annually will exceed 2 million tons, putting at risk the sustainability of the supply of critical elements such as lithium, cobalt, nickel and manganese (Lv et al., 2018; Abdalla et al., 2023).

In this context, LIB recycling processes emerge as central strategies to mitigate environmental impacts, reduce dependence on primary mining, and promote the circularity of materials (Lai et al., 2021). Among the reuse routes studied, the direct regeneration of cathodes stands out, which allows the functional recovery of the active materials without complete degradation of the crystalline matrix. This approach represents significant energy and reagent savings when compared to conventional pyrometallurgical and hydrometallurgical routes (Fan et al., 2020; Georgi-Maschler et al., 2012; Ding et al., 2024).

However, the effective adoption of direct regeneration on an industrial scale imposes a fundamental requirement: the accurate, reliable and comprehensive characterization of cathode materials throughout the entire process. Advanced analytical techniques are therefore indispensable for diagnosing the elemental composition of the waste, assessing the degree of degradation of the crystal structure, verifying the presence of contaminants, and validating the compatibility of the regenerated materials with industrial standards (Latini et al., 2022; Fan et al., 2020; Lv et al., 2018).

The absence of adequate characterization can compromise not only the efficiency of regeneration, but also the safety and performance of the new batteries produced. Therefore, methods such as X-ray diffraction (XRD), inductively coupled plasma optical emission spectroscopy (ICP-OES), X-ray fluorescence (XRF), scanning electron microscopy (SEM) coupled to energy dispersive spectroscopy (EDS) and Raman spectroscopy have been widely applied in recent studies, providing technical subsidies for the advancement of circular routes (Heelan et al., 2016; Fan et al., 2020; Lv et al., 2018).

Therefore, the present work aims to present a critical and updated review of the main analytical techniques used in the characterization of cathode materials from LIBs, with a special focus on their application in direct regeneration processes. It seeks to discuss the foundations, applications, limitations and perspectives of each method, highlighting its role in traceability, quality control and technical-economic feasibility of large-scale battery recycling.

2. THEORETICAL FOUNDATIONS OF ANALYTICAL TECHNIQUES

2.1 Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

Inductively coupled plasma optical emission spectrometry (ICP-OES) is widely used in the quantitative determination of metals in cathode materials of LIBs. The technique involves acid digestion of the sample and its introduction into an argon plasma, where the elements present are excited and emit characteristic radiation at specific wavelengths (Lv et al., 2018; Heelan et al., 2016).

This approach allows for the accurate quantification of lithium, cobalt, nickel, and manganese—central elements in the formulation of cathodes such as LCO, NMC, and LFP. ICP-OES stands out for its multi-element capacity, high sensitivity, and reproducibility, being fundamental both in the evaluation of residue stoichiometry and in the certification of the regenerated product (Fan et al., 2020; Ding et al., 2024).

However, the technique requires standardized digestion procedures to ensure the comparability of results between laboratories, which still represents a challenge in industrial validation (Fan et al., 2020; Latini et al., 2022).

2.2 X-ray Fluorescence (XRF))

X-ray fluorescence (XRF) is a non-destructive technique that relies on the secondary emission of X-rays by atoms excited by incident radiation. It is widely applied for the qualitative and semi-quantitative analysis of solids, pressed pellets and cathode powders from discarded LIBs (Georgi-Maschler et al., 2012; Lai et al., 2021).

Although it has lower sensitivity for light elements, such as lithium, XRF allows direct analysis of the sample, without the need for chemical digestion, being advantageous for rapid screening, in-line control, and compositional mapping of mixed batches (Latini et al., 2022). The use of certified standards and rigorous calibration are essential to increase the reliability of results in quantitative applications (Fan et al., 2020; Abdalla et al., 2023).

With the growing demand for rapid analysis in industrial plants, XRF has established itself as a first-response tool in the characterization of LIBs at the beginning of the repurposing process.

2.3 X-ray Diffraction (XRD)

The X-ray diffraction (XRD) technique is essential for the identification of crystalline phases present in cathode materials and for the monitoring of structural transformations during regeneration processes. The interaction of X-rays with the crystalline planes of the sample generates diffraction patterns that enable the identification of phases such as LCO ($LiCoO_2$), NMC ($LiNi_xMn\gamma CozO_2$), and LFP ($LiFePO_4$), as well as byproducts such as spinels, metal oxides, or carbonates (Fan et al., 2020; Ding et al., 2024).

In addition to the identification of phases, XRD allows estimating the degree of crystallinity, detecting secondary phases, and monitoring crystal restructuring after thermal processes, such as sintering and relitiation (Tian et al., 2024). In industrial settings, this technique has been combined with artificial intelligence algorithms to speed up the interpretation of diffractograms and allow for real-time adjustments (Heelan et al., 2016).

XRD is therefore considered essential to validate the structural integrity of regenerated materials and ensure their compatibility with electrochemical performance requirements.

2.4 Scanning Electron Microscopy with EDS (SEM-EDS)

Scanning electron microscopy (SEM), combined with energy dispersive spectroscopy (EDS), is an indispensable tool in the morphological and compositional evaluation of cathode materials. SEM provides high-resolution images of the surface of the particles, allowing the observation of characteristics such as agglomeration, porosity, crack formation, and degradation of the active layer (Lv et al., 2018; Latini et al., 2022).

When coupled to EDS, the system allows the identification of the elemental distribution along the particles, albeit in a semi-quantitative way, helping to map contaminants or loss of active elements after regeneration (Heelan et al., 2016; Ding et al., 2024). This analysis is particularly useful for diagnosing the uniformity of relitiation, evaluating the efficiency of sintering, and identifying structural flaws that may compromise electrochemical performance.

In addition, recent advances have integrated MEV-EDS into automated platforms, with artificial intelligence analysis, optimizing the screening and statistical quality control of regenerated batches on an industrial scale (Ascend Elements, 2025; ReCell Center, 2025).

2.5 Complementary Techniques: Raman spectroscopy, FTIR and TG/DSC

Spectroscopic and thermal techniques complement the analysis of regenerated materials by providing information on chemical bonds, the presence of residues, and thermal stability. Raman spectroscopy, for example, is sensitive to changes in local structure and chemical bonds, and is effective in detecting residual carbonates, amorphous species, and doping effects (Fan et al., 2020; Heelan et al., 2016).

FTIR (Fourier transform infrared spectroscopy) contributes to the identification of functional groups from the electrolyte or degraded organic ligands, allowing the tracking of impurities that impact the electrochemical behavior of the regenerated powder (Latini et al., 2022; Lai et al., 2021).

TG/DSC (thermogravimetry and differential scanning calorimetry) thermal analyses provide data on thermal stability, solvent removal, and endothermic or exothermic transitions associated with the decomposition of ligands and volatiles (Tian et al., 2024). These analyses are critical to validate the material's performance before reintegration into new LIB production cycles (Abdalla et al., 2023).

The combined use of these techniques offers a comprehensive view of the physicochemical integrity of the regenerated materials and strengthens the reliability of the process, especially when combined with quality certification protocols.

3. IMPORTANCE OF CHARACTERIZATION IN DIFFERENT STAGES OF RECYCLING

The recycling of LIBs, particularly by direct regeneration, relies on analytical characterization to guide decisions in sorting, process optimization, and product certification (Fan et al., 2020; Latini et al., 2022).

3.1 Screening and Initial Diagnosis

At the initial stage, techniques such as XRF and XRD identify chemical composition and crystalline phases, while SEM-EDS evaluates particle degradation and ICP-OES quantifies stoichiometry and metal losses. These analyses define whether materials are suitable for regeneration and help avoid processing unviable waste. (Lai et al., 2021; Latini et al., 2022; Georgi-Maschler et al., 2012; Lai et al., 2021).

3.2 Monitoring During Direct Regeneration

In regeneration, XRD tracks crystal restructuring and detects secondary phases, while ICP-OES and XRF confirm stoichiometry recovery. Complementary methods (Raman, FTIR, TG/DSC) identify organic residues and impurities, ensuring structural integrity and stable electrochemical performance (Fan et al., 2020; Tian et al., 2024; Ding et al., 2024; Heelan et al., 2016).

4. DISCUSSION: CHALLENGES AND REQUIREMENTS FOR INDUSTRIAL USE

The industrial adoption of cathode characterization in LIB recycling faces challenges related to standardization, costs, and traceability (Fan et al., 2020; ReCell Center, 2025).

4.1 Standardization

The lack of unified protocols for sampling and analysis compromises reproducibility, especially in direct regeneration, where small variations affect electrochemical performance. International standards with validated methods and certification criteria are urgently needed (Ding et al., 2024).

4.2 Costs and Accessibility

High costs of advanced techniques (SEM-EDS, XRD, ICP-OES) and long response times hinder real-time control. Portable and benchtop solutions (e.g., XRF, Raman) offer adaptability, but wider adoption depends on cost reduction, modular equipment, and supportive public policies (Lv et al., 2018; Steward et al., 2019; Lai et al., 2021; ReCell Center, 2025).

4.3 Traceability and Certification of Materials

Manufacturers demand certified regenerated materials, but digital tracking systems integrating analytical data are still lacking. Centralized databases linked to blockchain or ERPs are promising to ensure transparency and market acceptance (Heelan et al., 2016; Ding et al., 2024).

5. CONCLUSION AND PERSPECTIVES

The technical and economic feasibility of recycling lithium-ion batteries (LIBs), especially through direct regeneration routes of cathode materials, is intrinsically associated with the ability to characterize accurate, fast and reliable analytical characterization. This work presented a critical review of the main techniques used in the physicochemical, structural and morphological analysis of these materials, focusing on their application throughout the reuse cycle.

It was observed that each technique fulfills a specific and complementary role: XRF and XRD are essential for rapid screening and identification of phases; ICP-OES provides accurate diagnostics on stoichiometry; SEM-EDS allows the evaluation of morphological integrity and elemental distribution; while Raman, FTIR and TG/DSC elucidate the presence of chemical residues and the thermal stability of regenerates (Fan et al., 2020; Ding et al., 2024; Latini et al., 2022).

The strategic integration of these methodologies, from sorting to final certification, is critical to ensure the traceability, reproducibility, and electrochemical performance of recycled cathodes. However, important challenges persist, such as the lack of analytical standardization, high laboratory infrastructure costs, and the absence of integrated digital tracking systems (ReCell Center, 2025; Steward et al., 2019).

In this scenario, the following emerge perspectives stand out:

The development of automated platforms for the analysis of regenerated LIBs, using artificial intelligence to interpret spectral data and control processes in real time (Tian et al., 2024; Heelan et al., 2016). The formulation of international technical-normative guidelines, involving minimum criteria for the validation of regenerated materials (Latini et al., 2022; Abdalla et al., 2023);

The deployment of digital traceability systems based on analytical data, connecting recyclers, refiners, and manufacturers through transparent and auditable circular chains (Ascend Elements, 2025; Lai et al., 2021).

As a continuation of this study, it is recommended the investigation of hybrid methodologies (e.g. portable Raman + AI) and in situ techniques, which allow real-time monitoring of regenerated materials, reducing response time and operational costs. Such innovations will be decisive in accelerating the transition to circular industrial models based on secondary supply of critical metals and the high traceability green economy.

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