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Original research article

CHARACTERIZATION OF ELECTRODE MATERIALS FROM SPENT BATTERIES IN THE PROCESS OF RECOVERING VALUABLE METALS

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ABSTRACT – The chemical and phase analysis of electrode materials from spent batteries is essential for identifying recyclable components and optimizing metal recovery processes. In this study, the electrode material from a spent cell of an unknown manufacturer was analyzed using inductively coupled plasma mass spectrometry (ICP-MS) and powder X-ray diffraction (XRD). XRD results revealed that the anode material consists of three crystalline phases: NiO, $LaCoO_3$, and CeO_2, while the cathode material contains $Ni(OH)_2$ and Ni. ICP-MS analysis confirmed the presence of lanthanides in the anode material. Given that lanthanide deposits are concentrated in only a few countries, their recovery from spent batteries presents an opportunity for nations without natural reserves to secure their supply through recycling. This study highlights the potential for sustainable resource management by recovering metals from electronic waste.

Keywords: Recycling, Spent batteries, Characterization, Lanthanide.

INTRODUCTION

The increasing demand for battery materials has led to growing concerns about the depletion of natural resources and the environmental impact of waste disposal. Spent batteries, including lithium-ion (LIBs) and nickel-metal hydride (NiMH) batteries, contain valuable metals such as lithium (Li), cobalt (Co), nickel (Ni), and lanthanides, making them an important secondary source of these critical raw materials [1,2].

Among these critical materials, lanthanides play a key role in battery electrodes and are classified by the European Union (EU) as critical raw materials (CRMs) due to their strategic and economic importance. However, the global supply of rare earth elements (REEs) is highly concentrated in a few countries, particularly China, raising concerns over supply security. To address this issue, the recovery of lanthanides and other valuable

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[#] corresponding author: dmedic@tfbor.bg.ac.rs metals from spent batteries offers an opportunity to reduce dependence on primary sources and promote a circular economy. Hydrometallurgical, pyrometallurgical, and other methods were explored to enhance the efficiency of metal recovery [3].

Given the growing need for sustainable resource management, modern materials science is shifting from merely improving material properties to optimizing the reuse of secondary raw materials. The complexity of these multi-component materials requires detailed characterization to facilitate their reintegration into new applications. Understanding their chemical and structural composition is crucial for developing efficient recycling strategies and ensuring the quality of recovered materials [4].

To evaluate the quality and applicability of recycled materials, several analytical techniques are employed. Inductively coupled plasma mass spectroscopy (ICP-MS) is widely used to determine the concentrations of metals, while X-ray Diffraction (XRD) plays a critical role in determining the phase composition of the samples [5].

By integrating sustainable recycling strategies with advanced material characterization techniques, the recovery process can contribute to a more efficient and environmentally friendly battery supply chain. In this study, the recovered materials were analyzed using XRD to determine their phase composition and ICP-MS to assess their chemical composition, providing essential insights into their suitability for reuse.

EXPERIMENTAL

Sample Preparation

The analyzed sample was a spent battery from an unknown manufacturer. After opening the battery, six individual cells were extracted and fully discharged to prevent short circuits before disassembly. Using a hacksaw, the cell terminals were removed, followed by a longitudinal cut to separate the plastic casing, metal casing, anode, cathode, and separator (Figure 1). The anodic material was thermally treated at 580°C for 10 minutes to detach it from the current collector. Both anodic and cathodic materials were then ground into a fine powder using an agate mortar and pestle.

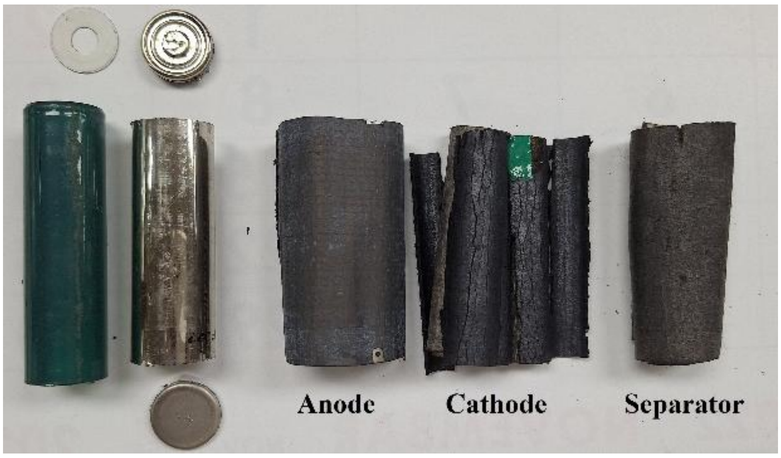

Figure 1 Basic components of a battery

Analytical Techniques

The metals from the recovered anode material were extracted using acid digestion. A 0.375 g sample was treated with 15 mL of aqua regia (a mixture of HCl and HNO_3), leaving behind a small portion of undissolved residue. The resulting solution was diluted to 100 mL with deionized water in a volumetric flask, then further diluted 25-fold in a 25 mL volumetric flask. Additional dilutions of 10-, 100-, and 10,000-fold were prepared to ensure that element concentrations fell within the linear range of the calibration curve before analysis using ICP-MS (PerkinElmer NexION 1000). Multi-element calibration standards (AccuStandard PE-MECAL2-ASL-1 and MES-21-1) were used for calibration and quality control of the measurements.

To accommodate the wide range of element concentrations, multiple dilutions were necessary, as their levels spanned several orders of magnitude. However, extensive dilution can introduce greater analytical errors, particularly for elements present in higher concentrations, potentially affecting measurement accuracy. The standard deviations between different dilutions ranged from 1.0% to 28.4%, indicating variable repeatability for different elements. Additionally, the obtained element concentrations do not represent the total elemental content of the sample, as an undissolved residue remained after digestion. Therefore, the determined values should be interpreted as the fractions extracted by aqua regia, rather than the total elemental concentrations.

The phase composition of the cathode and anode materials was characterized using powder X-ray diffraction (XRD). Measurements were carried out on a Rigaku MiniFlex 600 diffractometer equipped with a high-speed D/teX Ultra 250 detector and a copper anode X-ray tube. Data were collected over a 3–90° 2θ range with a step size of 0.02° and a scan speed of 10°/min. The instrument operated at 40 kV and 15 mA. Phase identification was performed using PDXL 2 software (Version 2.4.2.0), with diffractograms compared with reference patterns from the ICDD PDF-2 2015 database.

RESULTS AND DISCUSSION

Chemical Composition Analysis

The elemental composition of the anode material was analyzed to assess the presence of valuable metals and rare earth elements (REEs). The results (Table 1) indicate a high concentration of lanthanides, with lanthanum (14.6 %), neodymium (1.85 %), and cerium (1.32 %) as the most abundant. These elements, crucial for electrochemical performance, highlight the potential for REE recovery from spent batteries. Additionally, significant amounts of nickel (7.33 %) and manganese (3.07 %) were detected, confirming their role as key electrode components. The presence of cobalt (1.03 %) and copper (0.14 %) suggests recovery potential for secondary applications, while yttrium (0.20 %) and gadolinium (0.06 %) further emphasize the importance of recycling REEs.

Table 1 Elemental chemical composition (%) of the anode material

Element	Ce	Gd	La	Nd	Pr	Y	Co	Cu	Li	Mn	Ni
Content (%)	1.32	0.06	14.6	1.85	0.58	0.20	1.03	0.14	<0.01	3.07	7.33

A similar study was conducted by Bertuol *et al.* [6], who characterized five different NiMH batteries and concluded that the chemical composition of spent NiMH batteries can vary significantly depending on battery type, manufacturer, and intended application.

These findings reinforce the need for sustainable recycling strategies to minimize environmental impact and secure critical raw materials for future applications. Efficient recovery methods can support the circular economy by reducing dependence on primary sources and promoting the reuse of valuable metals.

Phase Composition Analysis

To determine the phase composition of the recovered materials, XRD analysis was conducted on both the anode and cathode samples. The resulting diffraction patterns provide insights into the phase composition of these materials, which are essential for evaluating their suitability for reuse and further processing. The diffractograms of anode and cathode materials are presented in Figures 2 and 3, respectively.

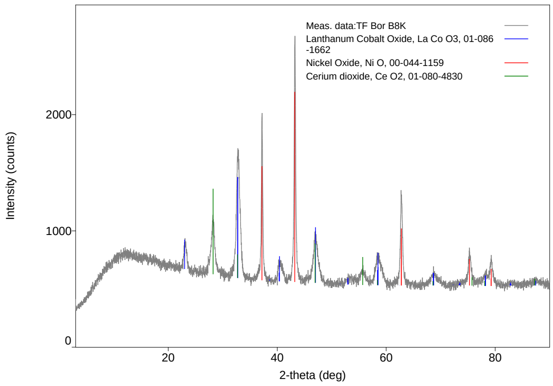

Figure 2 Diffractogram of the anode material

The diffractogram of the anode material (Figure 2) confirms the presence of three crystalline phases, identified as lanthanum cobalt oxide ($LaCoO_3$), nickel oxide (NiO), and cerium dioxide (CeO_2). These findings are in agreement with the results reported by Kanamori *et al.* [7], who also identified NiO, CeO_2, and $LaCoO_3$ as the main phases recovered from NiMH battery waste by chemical separation methods. Their study further demonstrated the applicability of these oxides as catalysts in methane dry reforming, emphasizing their potential for both metal recovery and functional reuse.

Other elements, such as Gd, Nd, Pr, Y, Cu, and Mn, may substitute for La, Co, Ce, and Ni within these crystalline structures. However, the exact distribution of these elements cannot be determined solely by XRD analysis, as phase identification relies on peak positions, which may overlap among different crystalline phases. Additionally, the XRD pattern exhibits well-defined peaks with no significant amorphous background, indicating a high degree of crystallinity in the analyzed sample.

The composition of the original NiMH anode typically consists of a metal alloy primarily composed of Ce, La, Pr, Nd, Ni, Co, Mn, and Al [8]. The detection of $LaCoO_3$ and NiO suggests that oxidation occurred during the annealing process, which was used to separate the anodic material from the current collector. Additionally, the formation of CeO_2 further confirms the oxidation of rare earth elements, emphasizing the impact of thermal treatment on the phase composition. These findings reinforce the need to optimize recycling processes to maximize the efficient recovery of critical raw materials.

The diffractogram of the cathode material (Figure 3) reveals two distinct crystalline phases, with the identified peaks corresponding to β-$Ni(OH)_2$, which is a more abundant phase, and metallic nickel (Ni), which is less abundant. The presence of β-$Ni(OH)_2$ indicates a nickel hydroxide phase, which is characteristic of secondary battery cathode materials. This observation is consistent with previous studies, including the work of Batsukh *et al.* [9], which identified β-$Ni(OH)_2$ as a major phase in the cathode materials of spent NiMH batteries. These findings provide essential insights into the structural properties of the recovered cathode material, which are crucial for determining its feasibility for further processing and reuse in battery recycling applications.

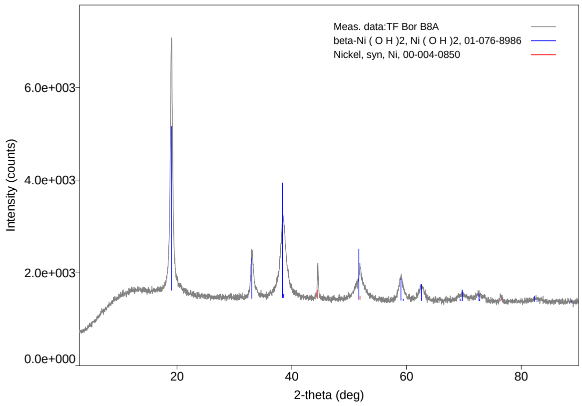

Figure 3 Diffractogram of cathode material

CONCLUSION

The characterization of electrode materials from spent NiMH batteries revealed significant amounts of valuable metals such as nickel, cobalt, and rare earth elements. Chemical and phase analyses identified stable oxide phases, including NiO, CeO_2, and $LaCoO_3$, indicating a high degree of stability and well-defined metal speciation within the electrode structure. These insights are essential for optimizing recycling processes, as they enable selective planning of extraction and separation methods. While some oxides, such as NiO, can be readily reduced and used as raw materials, rare earth oxides require more intensive approaches, including reduction, complexation, or thermal treatment.

In addition to metal recovery, the obtained materials also show potential for direct functional applications, including catalysis and photocatalysis, further increasing the value of these secondary raw materials. This approach contributes not only to the economic viability but also to the sustainability of recycling processes. Understanding the mineralogical and chemical forms of metals in battery waste is key to developing efficient and environmentally friendly technologies that enable the implementation of circular economy principles.

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REFERENCES

1. Ahn, N.-K., Shim, H.-W., Kim, D.-W., Swain, B. (2020) Valorization of waste NiMH battery through recovery of critical rare earth metal: A simple recycling process for the circular economy. Waste Management, 104, 254–261.
2. Sarkar, M., Hossain, R., Sahajwalla, V. (2024) Sustainable recovery and resynthesis of electroactive materials from spent Li-ion batteries to ensure material sustainability. Resources, Conservation & Recycling, 200, 107292.
3. Ahn, N.-K., Swain, B., Shim, H.-W., Kim, D.-W. (2019) Recovery of rare earth oxide from waste NiMH batteries by simple wet chemical valorization process. Metals, 9, 1151.
4. Nogueira, C.A., Margarido, F. (2007) Chemical and physical characterization of electrode materials of spent sealed Ni–Cd batteries. Waste Management, 27, 1570–1579.
5. Lie, J., Lin, Y.-C., Liu, J.-C. (2021) Process intensification for valuable metals leaching from spent NiMH batteries. Chemical Engineering & Processing: Process Intensification, 167, 108507.
6. Bertuol, D.A., Bernardes, A.M., Tenório, J.A.S. (2006) Spent NiMH batteries: Characterization and metal recovery through mechanical processing. Journal of Power Sources, 160, 1465–1470.
7. Kanamori, T., Matsuda, M., Miyake, M. (2009) Recovery of rare metal compounds from nickel–metal hydride battery waste and their application to CH_4 dry reforming catalyst. Journal of Hazardous Materials, 169, 240–245.
8. Marins, A.A.L., Boasquevisque, L.M., Muri, E.J.B., Freitas, M.B.J.G. (2022) Environmentally friendly recycling of spent Ni–MH battery anodes and electrochemical characterization of nickel and rare earth oxides obtained by sol–gel synthesis. Materials Chemistry and Physics, 280, 125821.
9. Batsukh, I., Adiya, M., Lkhagvajav, S., Galsan, S., Gansukh, M., Batmunkh, M. (2024) Recovering nickel-based materials from spent NiMH batteries for electrochemical applications. ChemElectroChem, 11, e202400135.