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# PHYSICAL SEPARATION AND RECYCLING-ORIENTED CHARACTERIZATION OF METALLIC MATERIAL CONTAINED IN SPENT NiMH BATTERIES

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*With the increase in demand for electronic and electrotechnical equipment comes the need to produce an increasing number of different types of batteries to support their operation. Batteries can irreversibly affect the environment if they are not properly stored after the end of their use cycle, and the best way to avoid their negative impact on the environment and to use an alternative source of metals is to recycle them. In this paper, we focused on the physical-mechanical processes of processing used batteries - Ni-MH with the aim of obtaining intermediate products that can later be exploited and of interest to the field of metallurgy (ferrous and non-ferrous metal fractions) as well as the characterization (SEM-EDAX, XRF) of the material contained in these types of batteries (Ni and Co, two very important metals for the production of batteries and, as it resulted from the XRF analyses, of the rare metals La, Ce, Ta in high concentrations). Their recovery and recycling is of economic interest due to the critical level at which they are located and especially the continuously growing demand for them, also having benefits for the environment through the conservation of resources.*

**Keywords**: recycling; characterization; NiMH batteries; rare metals

## 1. Introduction

Mineral resources are increasingly rare and inaccessible. Rechargeable batteries have become indispensable to modern man; through the electrification of vehicles and the development of renewable energy storage capacities, the need for scarce metals will lead to the impossibility of meeting demand [1, 2, 3, 4, 5, 6]. The production prices in extractive metallurgy and the pollution resulting from the processing of ores represent economic barriers that are difficult to overcome. Used batteries end up representing valuable sources of raw materials. Today, the

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recovery and recycling of useful metals from used batteries is a civic obligation and an economic requirement [1, 2, 4, 6, 7, 8, 9, 10, 11, 12, 13].

If some metallic materials contained in these NiMH type batteries can be easily used in new products (Ferro-nickel or Ferro-nickel-molybdenum) with much lower costs and being able to successfully replace the products based on primary nickel and molybdenum [6, 7], not the same the same can be said about cobalt and rare metals [3, 4, 7]. They still represent a sensitive subject in terms of extraction and processing, not to mention their prices, which have been on an upward trend for some time. These things make the recovery and valorization of useful and especially economically valuable metals necessary and intensively researched [1, 3, 4, 14].

There are two main methods of processing and valorizing NiMH used batteries - pyrometallurgical [15] - SNAM-SAVAN, SAB-NIFE and INMETCO industrial processes and a combination of hydrometallurgical processes [11] – material dissolution using reagents and selective precipitation, solvent extraction crystallization and other chemical operation for recovery of dissolved metals.

These methods are very complex and very ineffective (a large amount of important metals are lost), and so far no optimal recycling strategy has been established, and this is because there are many types of batteries with different chemical compositions on the market [6, 11, 15, 16]. The recovery of the component metals is selective and highly dependent on the process used. We can increase the yield of extraction or reduce pollution by using environmentally friendly and affordable substances, so that these processes become economical and can be applied on a larger scale [1, 3, 7, 11, 14, 16, 17].

Even if new types of rechargeable batteries appear periodically for energy storage, NiMH type batteries maintain their position on the market [18, 19], which represents approx. 28% after Li-ion batteries – 37% [17] due to good operating characteristics for general or specific applications, mainly increased security, higher temperatures of use and even due to obtaining different shapes of cells [1, 7, 11, 18].

The major manufacturing advantage is the safety of NiMH batteries over Li-ion batteries, and so far no incidents have been reported in the media [18]. Furthermore, NiMH batteries are preferred in industrial and consumer applications due to their design flexibility, environmental acceptability, low maintenance, high power and energy densities, cost, and most important, safety (in charging and discharging modes, especially at high voltages) [11, 14, 18].

The major disadvantage of using NiMH batteries, according to the study of [19], is that there is a major environmental impact comparative with the Li-ion baterries, which makes it even more necessary to collect and not store these batteries, so that they do not end up in landfills.

Spent NiMH batteries represent more than 8% of all portable batteries collected worldwide, with a huge useful metal content [1, 6, 17], and their recovery and recycling is a beneficial opportunity for both the environment and the economy.

The metal valuable content in NiMH batteries is for nickel -36…42%, cobalt – 3…5% and REEs – 5…25%, making this category of waste become a reservoir of potential precious resources for the future [3, 4, 7, 11, 16, 17].

In the paper, we focused on the physical-mechanical methods of processing used Ni-MH batteries and on the preliminary characterization of the materials obtained with the aim of obtaining intermediate products that can later be exploited and of interest to the field of metallurgy

## 2. Materials, methods and experimental

The research was carried out in the Hydrometallurgy Laboratory within the Department of Engineering and Management of Obtaining Metallic Materials of the Faculty of Materials. The batteries used for the experimental research were of the Ni-MH type (rechargeable / secondary batteries) being processed as follows and illustrated in Fig. 1:

- manual disassembly by cutting;
- manual, magnetic and sieving separation of different fractions;
- extraction of the mixture of cathodic and anodic material;
- washing the fractions to recover the active mass and the electrolyte;
- neutral leaching to remove easily soluble components;
- filtering the resulting solutions;
- oven drying of the obtained material.

To select and establish an appropriate recovery process for the valuable materials (nickel, cobalt, and rare earth elements) contained in rechargeable NiMH batteries, the physical-mechanical processing of the used batteries and the separation by types of components was carried out to identify and analyze the categories of content materials. The spent batteries were manually disassembled by cutting, the component materials were separated into different fractions and the mixture of cathode and anode material was extracted.

The fractions obtained separately were washed with distilled water to remove the active mass, the electrolyte and other components adhering to these battery components, and especially for neutralization. The washing process lasted about 60 minutes to remove any alkali metals (e.g. Na and K) from the electrolyte, which may act as an initiator for the double precipitation of rare earths (REE) during solubilization and lead to loss of REEs.

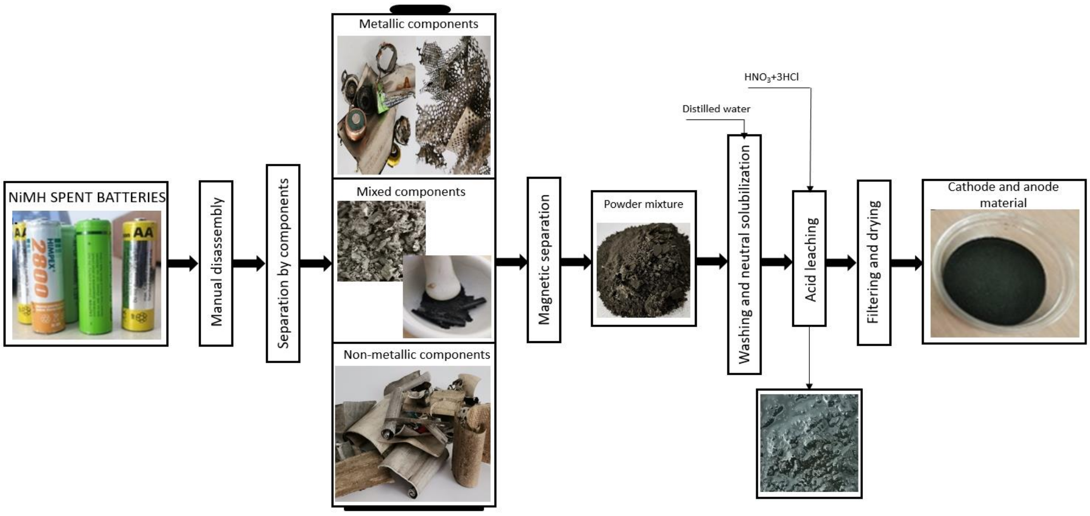

Fig.1. The main steps in the processing of used NiMH batteries

The sorting of materials into categories was done manually and by magnetic separation, due to the small amount of material used in the experiments. After dividing by categories were resulted the weights reported in Table 1:

*Table 1*

**Weight of component materials from disassembled batteries**

| Material | Composition | (g) | (%) |
|---|---|---|---|
| Non-ferrous | *Nickel / lithium hydride grids or nickel / lanthanum* | 3.5 | 4.13 |
| Ferrous | *Steel* | 15.2 | 17.95 |
| Blended | *Anodic powder* | 35.5 | 41.91 |
| | *Cathodic powder* | | |
| | *Coarse product* | 22.2 | 26.21 |
| Non-metallic | *Polymer, Paper, Plastic, Cotton* | 3.1 | 3.66 |
| Moisture | | 3.37 | 3.98 |
| Losses (on disassembly and on the filter paper) | | 1.83 | 2.16 |
| **Total** | | **84.7** | **100** |

The material obtained was dried in an oven at a temperature of $75^0$C for two hours to remove moisture and before chemical analysis to determine the constituent elements by X-ray fluorescence spectrometer (WDXRF S8 Tiger Bruker). The microstructural and microcompositional investigation of the samples was carried out by scanning electron microscopy (SEM) on the scanning electron microscope Quanta Inspect F50, with a Field Emission Gun (FEG) with resolution of 1.2 nm, and an EDX (Energy Dispersive X-ray) analyzer having a resolution of 133 eV at Mn.

## 3. Results and discussion

The materials with high content of valuable metals were analyzed by X-ray Fluorescence Spectroscopy (XRF), Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Analysis (EDAX). The results are presented in Tables 2 to 4 and in Figs. 3.

*Table 2a*

**Chemical composition of the blended material (anode and cathode)**

| Z | Element | Concentration | Unit | Abs. Error | Unit |
|---|---|---|---|---|---|
| 11 | Na | 7.14 | % | 0.23 | % |
| 12 | Mg | 0.337 | % | 0.0063 | % |
| 13 | Al | 0.4685 | % | 0.003 | % |
| 14 | Si | 0.1319 | % | 0.0007 | % |
| 15 | P | 356.3 | µg/g | 2.1 | µg/g |
| 16 | S | 0.04392 | % | 0.00015 | % |
| 17 | Cl | 80.9 | µg/g | 0.4 | µg/g |
| 19 | K | 0.04242 | % | 0.00071 | % |
| 20 | Ca | 0.02647 | % | 0.0003 | % |
| 22 | Ti | 0.0182 | % | 0.0017 | % |
| 23 | V | 387 | µg/g | 22 | µg/g |
| 24 | Cr | 520.2 | µg/g | 8.6 | µg/g |
| 25 | Mn | 3313 | µg/g | 9 | µg/g |
| 26 | Fe | 0.09873 | % | 0.00048 | % |
| 27 | Co | 4.68 | % | 0.099 | % |
| 28 | Ni | 79.79 | % | 0.05 | % |
| 29 | Cu | 8925 | µg/g | 36 | µg/g |
| 30 | Zn | 6304 | µg/g | 14 | µg/g |
| 31 | Ga | < 0.5 | µg/g | 0 | µg/g |
| 32 | Ge | < 0.5 | µg/g | 0 | µg/g |
| 33 | As | < 0.5 | µg/g | 0 | µg/g |
| 34 | Se | < 0.5 | µg/g | 0 | µg/g |
| 35 | Br | 7.7 | µg/g | 0.6 | µg/g |
| 37 | Rb | < 0.5 | µg/g | 0 | µg/g |
| 38 | Sr | 4.3 | µg/g | 0.4 | µg/g |
| 39 | Y | 308.1 | µg/g | 2.4 | µg/g |

*Table 2b*

**Chemical composition of the blended material (anode and cathode)**

| Z | Element | Concentration | Unit | Abs. Error | Unit |
|---|---|---|---|---|---|
| 40 | Zr | 179.5 | µg/g | 2.7 | µg/g |
| 41 | Nb | < 1.0 | µg/g | 0 | µg/g |
| 42 | Mo | 11.4 | µg/g | 1.1 | µg/g |
| 47 | Ag | < 2.0 | µg/g | 0 | µg/g |
| 48 | Cd | 4.2 | µg/g | 0.6 | µg/g |
| 50 | Sn | 67.8 | µg/g | 1.5 | µg/g |
| 51 | Sb | 4.7 | µg/g | 0.6 | µg/g |
| 52 | Te | 4 | µg/g | 0.3 | µg/g |
| 53 | I | < 3.0 | µg/g | 0 | µg/g |
| 55 | Cs | < 4.0 | µg/g | 0 | µg/g |
| 56 | Ba | < 2.0 | µg/g | 0 | µg/g |
| 57 | La | 3.467 | % | 0.007 | % |
| 58 | Ce | 1.096 | % | 0.005 | % |
| 68 | Er | < 5.1 | µg/g | 0 | µg/g |
| 70 | Yb | < 2.0 | µg/g | 0 | µg/g |
| 72 | Hf | < 1.0 | µg/g | 0 | µg/g |
| 73 | Ta | 0.613 | % | 0.013 | % |
| 74 | W | < 1.0 | µg/g | 0 | µg/g |
| 80 | Hg | < 1.0 | µg/g | 0 | µg/g |
| 81 | Tl | < 1.0 | µg/g | 0 | µg/g |
| 82 | Pb | 19 | µg/g | 1.2 | µg/g |
| 83 | Bi | < 1.0 | µg/g | 0 | µg/g |
| 90 | Th | < 6.3 | µg/g | -4.9 | µg/g |
| 92 | U | < 4.8 | µg/g | 0 | µg/g |
|  |  | **100** | % |  |  |

The results show that the concentration of Ni and Co in the resulting powder is 79.79% and 4.68% respectively, in addition we also find high concentrations of rare earths (La - 3.467%, Ce - 1.096%, Ta - 0.613%) all of which have an economic importance particularly high.

The coarse product obtained after sieving (Fig. 2a), which consists of a mixture of pieces of metal grids and anodic and cathodic powder, but also some small amounts of non-metallic fraction (paper, plastic, and cotton) was chemically treated with aqua regia ($HNO_3 + 3HCl$) to dissolve the components (Fig. 2b), then washed with distilled water and dried in an oven and subjected to an XRF analysis (Table 3a and b).

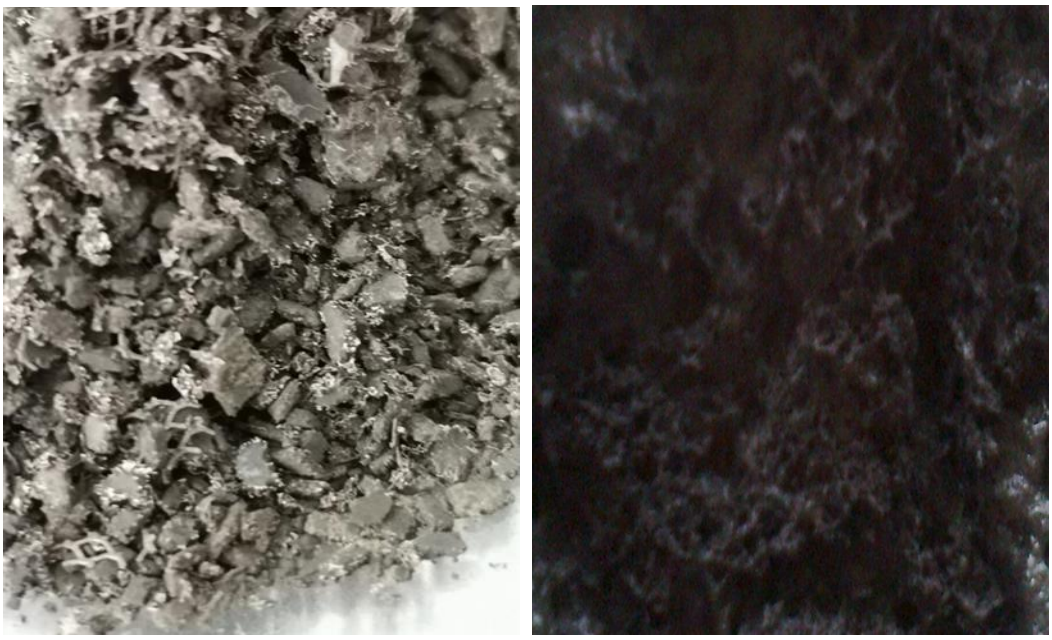

Fig.2. The macroscopic appearance of the coarse product: a) before dissolution and b) after dissolution

*Table 3a*

**Chemical composition of the coarse fraction**

| Z | Element | Concentration | Unit | Abs. Error | Unit |
|---|---|---|---|---|---|
| 11 | Na | 2.201 | % | 0.07 | % |
| 12 | Mg | 0.217 | % | 0.025 | % |
| 13 | Al | 2.837 | % | 0.041 | % |
| 14 | Si | 7.558 | % | 0.013 | % |
| 15 | P | 0.3143 | % | 0.0016 | % |
| 16 | S | 0.6116 | % | 0.0016 | % |
| 17 | Cl | 13.11 | % | 0.01 | % |
| 19 | K | 0.1743 | % | 0.0034 | % |
| 20 | Ca | 2.99 | % | 0.008 | % |
| 22 | Ti | 0.1231 | % | 0.0031 | % |
| 23 | V | < 18 | µg/g | -16 | µg/g |
| 24 | Cr | 839.7 | µg/g | 8.2 | µg/g |
| 25 | Mn | 1015 | µg/g | 8 | µg/g |
| 26 | Fe | 9.849 | % | 0.008 | % |
| 27 | Co | < 3.0 | µg/g | 0 | µg/g |
| 28 | Ni | 0.3605 | % | 0.0025 | % |
| 29 | Cu | 33.29 | % | 0.03 | % |
| 30 | Zn | 1266 | µg/g | 5 | µg/g |
| 31 | Ga | < 0.5 | µg/g | 0 | µg/g |

*Table 3b*

**Chemical composition of the coarse fraction**

| Z | Element | Concentration | Unit | Abs. Error | Unit |
|---|---|---|---|---|---|
| 32 | Ge | < 0.5 | μg/g | 0 | μg/g |
| 33 | As | < 0.5 | μg/g | 0 | μg/g |
| 34 | Se | < 0.5 | μg/g | 0 | μg/g |
| 35 | Br | 3.299 | % | 0.003 | % |
| 37 | Rb | < 0.5 | μg/g | 0 | μg/g |
| 38 | Sr | 155 | μg/g | 1.1 | μg/g |
| 39 | Y | 2.4 | μg/g | 0.9 | μg/g |
| 40 | Zr | 101.6 | μg/g | 3.1 | μg/g |
| 41 | Nb | < 1.0 | μg/g | 0 | μg/g |
| 42 | Mo | 110.7 | μg/g | 2.7 | μg/g |
| 47 | Ag | 0.799 | % | 0.013 | % |
| 48 | Cd | < 2.0 | μg/g | 0 | μg/g |
| 50 | Sn | 1.284 | % | 0.002 | % |
| 51 | Sb | 273.7 | μg/g | 4 | μg/g |
| 52 | Te | 24,3 | μg/g | 1.4 | μg/g |
| 53 | I | < 3.0 | μg/g | 0 | μg/g |
| 55 | Cs | < 4.0 | μg/g | 0 | μg/g |
| 56 | Ba | 0.2526 | % | 0.0022 | % |
| 57 | La | 141 | μg/g | 11 | μg/g |
| 58 | Ce | < 2.0 | μg/g | 0 | μg/g |
| 72 | Hf | < 1.0 | μg/g | 0 | μg/g |
| 73 | Ta | < 1.0 | μg/g | 0 | μg/g |
| 74 | W | 784 | μg/g | 16 | μg/g |
| 80 | Hg | < 1.0 | μg/g | 0 | μg/g |
| 81 | Tl | 126.1 | μg/g | 6.5 | μg/g |
| 82 | Pb | 0.1733 | % | 0.0007 | % |
| 83 | Bi | < 1.0 | μg/g | 0 | μg/g |
| 90 | Th | 181.8 | μg/g | 3.5 | μg/g |
| 92 | U | 22.1 | μg/g | 3.2 | μg/g |
|  |  | 100 | % |  |  |

Compared to the previous XRF analysis, it can be observed that quite significant amounts of the following elements appeared in the coarse material: Mg, Al, Si, Ca, Ti, Fe, Cu, Br, Ag, Sn, Ba and Pb. Considering the economic values of some of these elements (Ag, Si, Br, Ba) this material recovered from the processing of used NiMH type batteries is of particular interest to the industry.

An SEM-EDAX analysis of the obtained mixed material was also performed; the morphological aspect is shown in Fig. 3 and the elemental micro-composition of the blended material (anode and cathode) is reported in Table 4.

Aggregations of particles with different sizes are observed, resulting from the electrochemical processes inside the battery during the charge-discharge cycles.

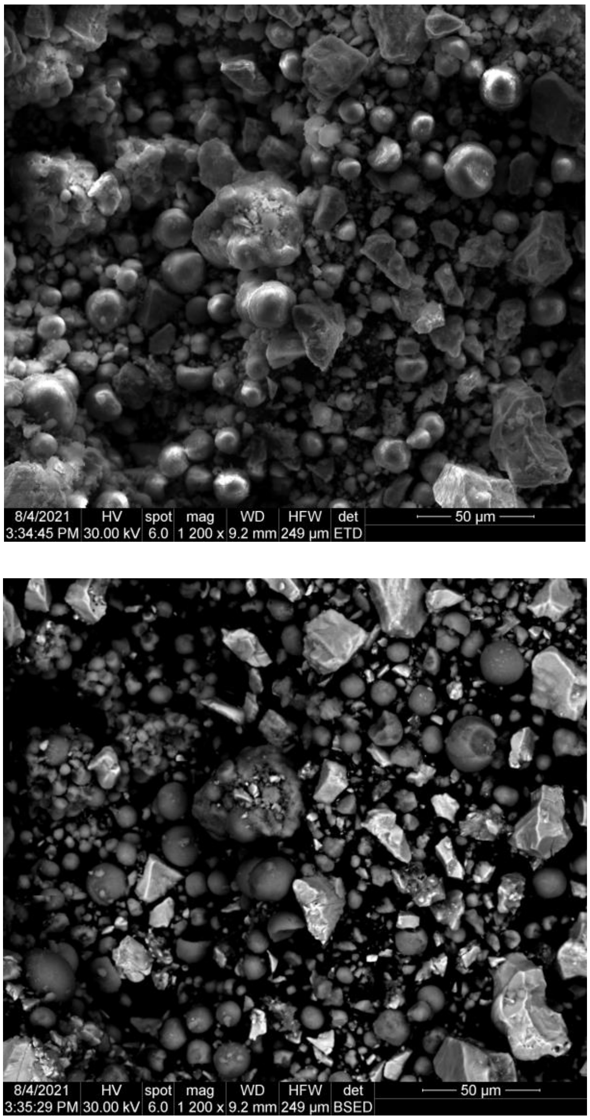

Fig. 2. SEM image of blended powder separated from Ni-MH batteries

Energy-dispersive X-ray microanalysis (EDAX) highlights the presence in the sample of the major elements Nickel, Cobalt, Lanthanum, in oxide forms as suggested by the presence of a large amount of oxygen and the minor elements Praseodymium, Neodymium, Cerium, and Manganese.

*Table 4*

**EDAX analysis of the blended powder separated from Ni-MH batteries**

| Analysis | Elem | Wt. % | At % | K-Ratio | Z | A | F |
|---|---|---|---|---|---|---|---|
| EDAX ZAF Quantification (Standardless) Element Normalized SEC Table: Default | OK | 19.69 | 50.26 | 0.0708 | 1.1128 | 0.3225 | 1.0018 |
| | LaL | 6.95 | 2.04 | 0.0681 | 0.8390 | 1.1009 | 1.0613 |
| | Cel | 2.56 | 0.75 | 0.0258 | 0.8477 | 1.1095 | 1.0728 |
| | PrL | 2.64 | 0.77 | 0.0275 | 0.8584 | 1.1161 | 1.0864 |
| | NdL | 3.07 | 0.87 | 0.0323 | 0.8535 | 1.1210 | 1.0993 |
| | MnK | 1.36 | 1.01 | 0.0137 | 0.9517 | 0.9365 | 1.1318 |
| | CoK | 5.35 | 3.71 | 0.0478 | 0.9546 | 0.9357 | 1.0000 |
| | NiK | 58.38 | 40.61 | 0.5505 | 0.9920 | 0.9505 | 1.0000 |
| | Total | 100.00 | 100.00 | | | | |

Following the EDAX analysis, one can observe the appearance of other important elements Pr and Nd, also from the lanthanide series, which escaped detection by XRF.

The material obtained, shown in Fig. 1 and analyzed from a microstructural and chemical composition point of view, will be used in experiments of solubilization and electrolytic extraction of metals, which can be reused for the production of Ni-MH type batteries or in other fields.

## 4. Conclusions

The metallic material recovered from the processing of used NiMH type batteries is of particular interest to the industry and the economic aspect is not to be neglected, as it was found the concentration for the main valuable metals in the resulting mixed powder was 79.79% for Ni and 4.68% for Co.

In addition, the most encouraging result is the presence in high quantities of some rare earths La - 3.467%, Ce - 1.096%, Ta - 0.613% all of which have an economic importance particularly high. The results are confirmed by SEM-EDAX analysis, which also highlights the presence of two other elements - Pr and Nd.

The separated powder with metal content will enter the hydrometallurgical circuit, where the metals will be selectively dissolved followed by a purification and cementation process, and the obtained solutions will be subjected to electrolysis processes for the selective recovery of metals.

Later, following the obtained results, an optimal strategy will be established for the extraction of elements of high economic importance:

- from a technical point of view - by establishing the appropriate electrolytic processing and extraction parameters.

- economical - by using minimal energy consumption and low-priced consumables.
- and environmental - by using substances with minimal impact on the environment.

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