



Physical separation, mechanical enrichment and recycling-oriented characterization of spent NiMH batteries

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Abstract

Nickel–metal hydride (NiMH) batteries contain high amount of industrial metals, especially iron, nickel, cobalt and rare earth elements. Although the battery waste is a considerable secondary source for metal and chemical industries, a recycling process requires a suitable pretreatment method before proceeding with recovery step to reclaim all valuable elements. In this study, AA- and AAA-type spent NiMH batteries were ground and then sieved for size measurement and classification. Chemical composition of the ground battery black mass and sorted six different size fractions were determined by an analytical technique. Crystal structures of the samples were analyzed by X-ray diffraction. Results show that after mechanical treatment, almost 87 wt% of the spent NiMH batteries are suitable for further recycling steps. Size classification by sieving enriched the iron content of the samples in the coarse fraction which is bigger than 0.25 mm. On the other hand, the amounts of nickel and rare earth elements increased by decreasing sample size, and concentrated in the finer fractions. Anode and cathode active materials that are hydrogen storage alloy and nickel hydroxide were mainly collected in finer size fraction of the battery black mass.

Keywords NiMH battery · Spent batteries · Physical separation · Mechanical enrichment · Waste characterization

Introduction

The importance of electrical energy storage systems have been increasing last two decades due to not only increasing amount of electrical and electronic equipment, hybrid electrical vehicles (HEV) and electrical vehicles (EV), but also growing demand for renewable energy sources [1–3]. Nickel–metal hydride (NiMH) battery is an established energy storage technology which has high volume commercial production for small portable battery applications as well as vehicle packaging for automotive and marine industries [3–6]. These batteries provide most of the critical properties for the general and specific applications, such

as high safety, power, wide range of operating temperature and flexible battery packaging with the choice of cylindrical or prismatic type cells [4, 5]. For example, cylindrical D-type or prismatic NiMH batteries were used for HEV and/or EV battery packs [7, 8]. Although Li-ion battery technology began to dominate the vehicle market, the forecast indicates that NiMH battery demand will slightly increase in the coming years [9].

NiMH battery cell composes of potassium hydroxide (KOH) electrolyte, nickel hydroxide (NiOH) in cathode, hydrogen storage nickel-based alloy in anode, steel cover, current correctors, organic separator and sealing. The common alloys in the electrode designs are AB_5 (LaCePrNd-NiCoMnAl), A_2B_7 (LaCePrNdNiCoMnAl plus Mg), and AB_2 (VTiZrNiCrCoMnAlSn), where the “ A_xB_y ” refers to the ratio of the A-type elements (LaCePrNd or TiZr) to that of the B-type elements (VNiCrCoMnAlSn) [4, 10]. A spent nickel–metal hydride cylindrical type battery composes of about 36–42 wt% nickel (Ni), 22–25 wt% iron (Fe) 3–4 wt% cobalt (Co) and 8–10 wt% mischmetal consisting of rare earth elements [lanthanum (La), cerium (Ce), praseodymium (Pr) and neodymium (Nd)], 1–2 wt% potassium (K), 3–4 wt% plastics and < 1 wt% graphite [10, 11].

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The recycling of spent NiMH batteries is attractive because of not only the high amount of valuable metallic content, but also considerable amount of Co and REE, which are accepted as critical raw material for European Union [12]. For example, a NiMH-type HEV battery pack contains 3.5 kg REE [13].

The current industrial approach to recycle the NiMH batteries is adding them to the stainless steel production to reclaim the Ni, Fe and Co. However, the rest of the elements, especially REEs, stay in the slag, and then difficult to recover them. Recently Umicore (Belgium) and Rhodia (France) developed a combined pyro- and hydro-metallurgical process to recover the REEs [10, 11]. Several studies have also been conducted on recycling of spent NiMH batteries by hydrometallurgical route. Different leaching reagents (sulfuric acid, nitric acid, hydrochloric acid, hydrogen peroxide, etc.) and various solid–liquid ratios were tested to develop an efficient method to recover the valuable metals [14–19]. After leaching, REEs and other metals can be separated by solvent extraction or precipitation methods [16–19]. Petrankova et al. [20] reported optimization of the pilot-scale process using mixer settlers equipment to separate the valuable metals from the leaching solution. Their results showed that hydrometallurgical method needs several stages to separate the metals.

On the other hand, mechanical pretreatment reduces the material complexity of the spent batteries by the separation of plastics and papers, as well as enriching metal content in the fine fractions for further recycling step. It is generally accepted that mechanical pretreatment of batteries is a necessity before the hydrometallurgical treatment [21–23]. Granata et al. [24] tested 3-step mechanical treatment process including two-rotor crushing, hammer crushing and then thermal treatment. The mechanical route allowed to recover around 40% of the valuable fractions of NiMH batteries, and the rest, 49%, is smaller than 1 mm that processed by hydrometallurgical methods to recover the valuable content. Bertuol et al. [25] studied sequential hammer mill and knife mill to grind the spent batteries and applied magnetic separation after the milling steps. The samples classified as non-magnetic, weak and strong magnetic materials. The strong magnetic fraction was a Fe–Ni alloy that suitable for secondary steel production as a raw material. The weak magnetic fraction was powder and needs additional recovery processing. Huang et al. [26] investigated mechanical treatment and size classification of the spent NiMH batteries. They showed that sample > 2 mm size is mainly Fe and Ni, and smaller fractions than < 2 mm contain lesser Fe but nearly 5 times higher amount Ni. The distribution of the Ni, Co, Zn and REEs were nearly same in the smaller fractions than < 2 mm size. Tenório et al. [27] presented that about 33 wt% of the spent battery was recovered as a valuable Ni and/or alloys by a several step process including milling,

magnetic and size separation. According to Tanabe et al. [28], a mechanical pretreatment can efficiently separate metals, polymers and powder fractions of spent NiMH batteries. In addition to these, Czerny and Reichel [29] patented a combined mechanical and heat treatment method for metal recovery from NiMH batteries. After the pretreatment process and size separation, the coarse fraction was recycled by pyrometallurgical approach as ferronickel, and fine fraction was further processed alumina thermal method to reclaim the valuable materials in metallic form. Ruffino et al. [30] studied the mechanical treatment of different types of spent alkaline, Zn–C and Ni–Cd batteries, and showed that particle size classification and chemical distribution change by battery type.

Although there are some studies on mechanical treatment of the spent NiMH batteries, there are not enough data on elemental distribution in the milled samples according the size classification. This study investigated the mechanical

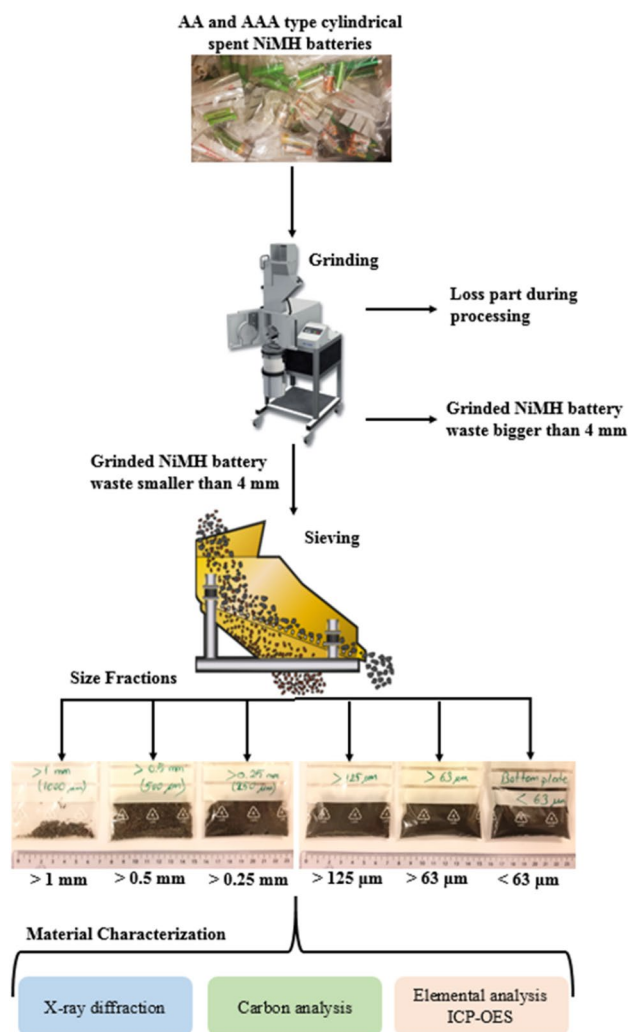


Fig. 1 Process flow and steps of experimental procedure

pretreatment of AA- and AAA-type cylindrical spent NiMH batteries, and enrichment possibility of the specific metallic contents according to size fractions, which is smaller than 4 mm. The enrichment of the metallic content in different size fractions decreases the complexity of the waste and increases the recycling method options.

Experimental

The spent NiMH batteries were industrially collected and separated from other types of batteries (alkaline and Li-ion) by Renova AB, Sweden. AA- and AAA-type cylindrical spent NiMH batteries, which belong to various brands and having different capacities, were used. The laboratory process flow with experimental steps is given in Fig. 1. The spent batteries were ground by Retsch SM 300 cutting mill at 2900 min^{-1} rotation speed with a continuous feeding inlet. A 4-mm sieve was located under the milling chamber and fine battery black mass filled the collection chamber during

the mechanical treatment. Figure 2 shows the equipment, illustration of milling step and spent battery samples. Experiments were performed in two sets using 988 and 1007 g of spent batteries for 5 and 10 min treatment time, respectively. After milling, battery black mass was sorted as undersize sample that are fine powders smaller than 4 mm, oversize sample that are coarser than 4 mm, and loss that are stuck on the equipment as given in Fig. 3.

The quartering method was used to take samples from the fine battery black mass samples for the size classification experiments. Particle size analyses were performed using 1, 0.5, 0.25, 0.125 and 0.063 mm size sieves. The images of the samples after sieving test are given in supplementary document. Then all samples were analyzed by material characterization techniques.

X-ray diffraction (XRD, Bruker D8 Advance) using Cu $K\alpha$ radiation was used to examine the phase content, crystalline structure and size of the battery waste. The conditions for data collection were continuous scanning of a detector covering a 2θ angular range from 10° to 90° with

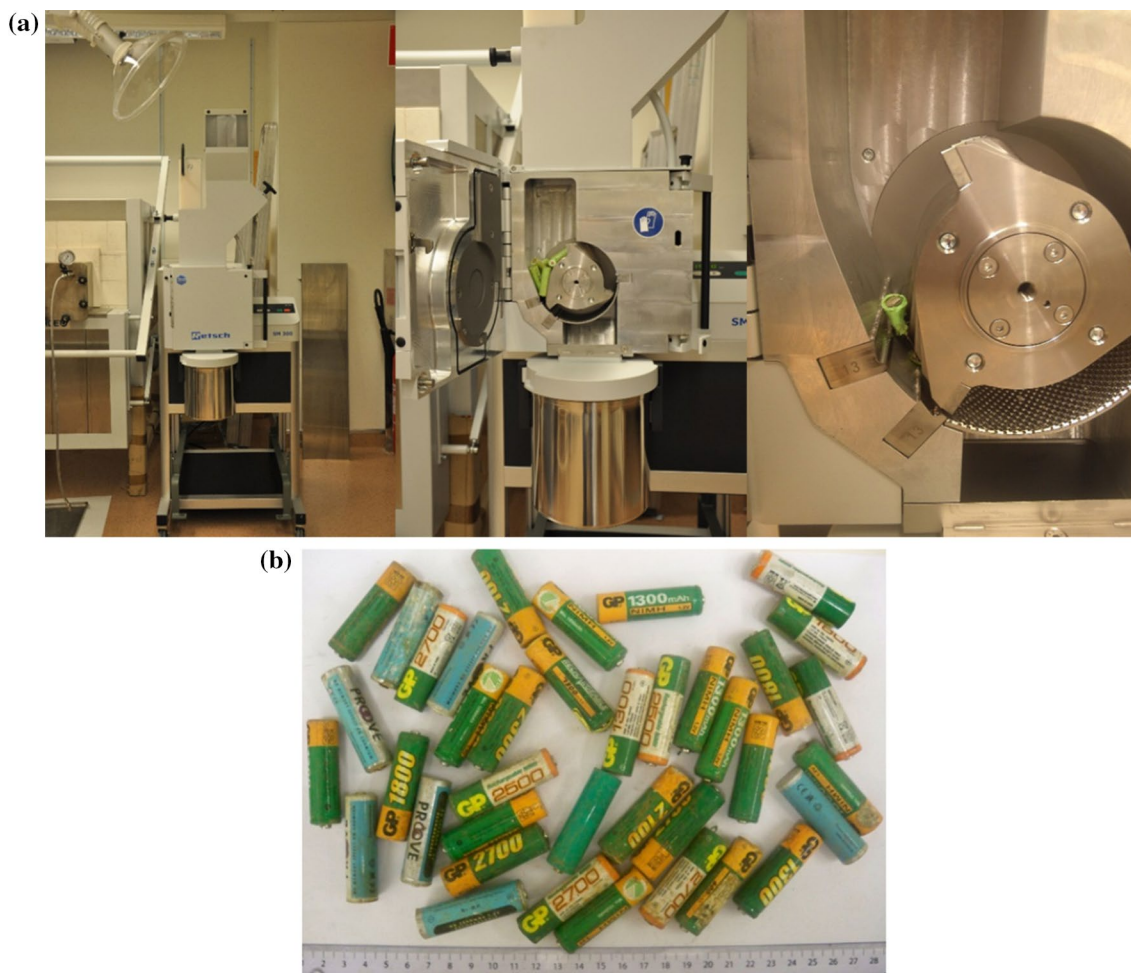
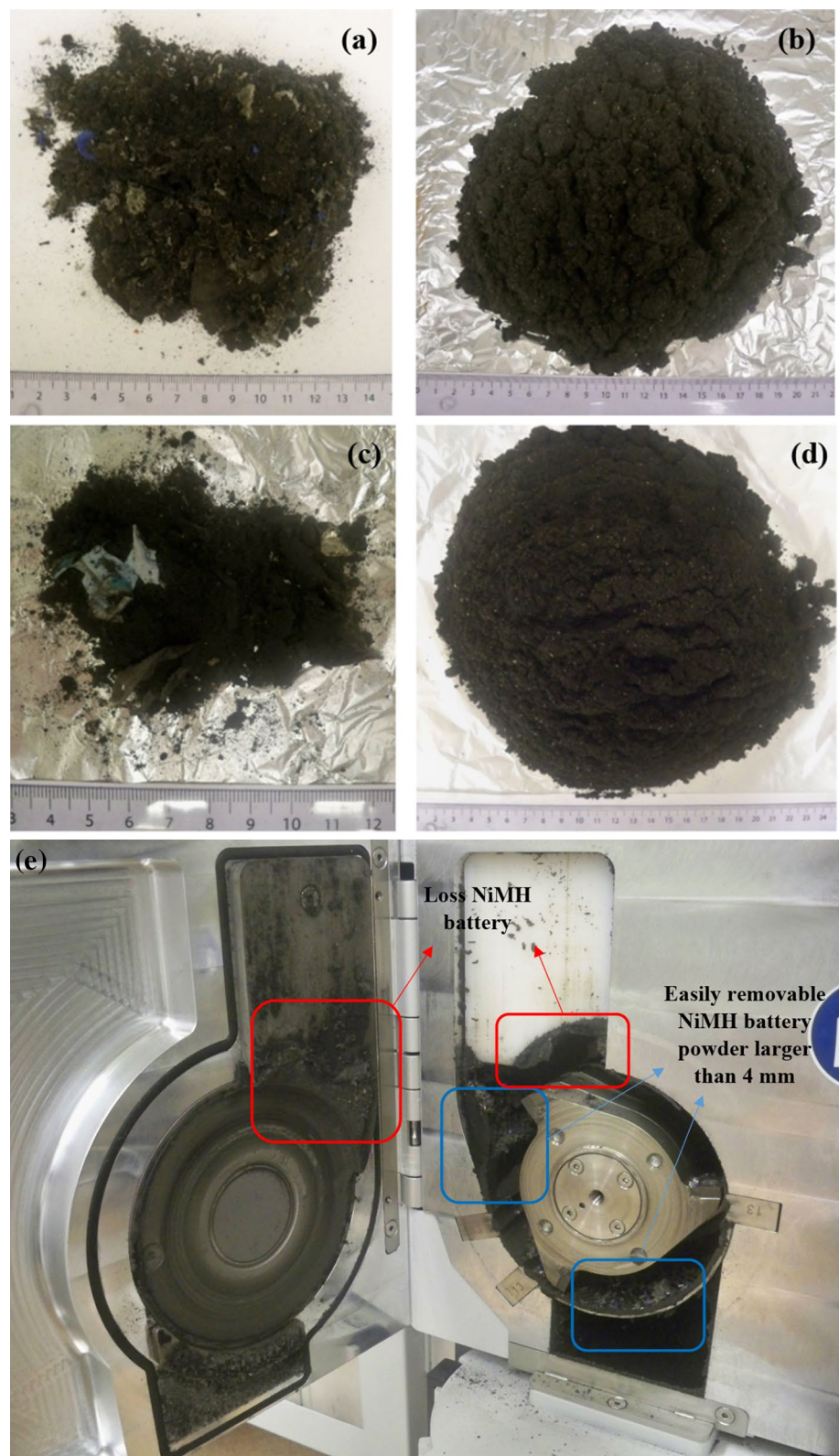


Fig. 2 Images of **a** grinding equipment, and **b** spent NiMH batteries

Fig. 3 Images of ground samples **a** > 4 mm for 5 min, **b** < 4 mm for 5 min, **c** > 4 mm for 10 min, **d** < 4 mm for 10 min, and **e** the stuck samples on the equipment accepted as loss



a step size of 0.04 and a wavelength of 1.541874 Å. The chemical compositions of the samples, which are smaller than 4 mm and all subsize fractions, were analyzed by

inductive coupled plasma optical emission spectrometry (ICP-OES, ICAP 6500, Thermo Fischer). The battery black mass, 0.2 g powder, were dissolved in 30 mL aqua

regia ($\text{HNO}_3+3 \text{HCl}$) and diluted in the desired amounts for the ICP-OES analyses. Carbon analyzer (Eltra Carbon/Sulfur Analyzer CS-800) was used to determine the amount of carbon in the battery black mass.

Results and discussion

Total mass distributions of the three fractions, which are smaller than 4 mm, coarser than 4 mm, and loss, were analyzed after grinding process to evaluate the recyclable amount. Figure 4 shows the total mass distributions of the samples depending on grinding time. The weight of the fine fraction (< 4 mm) increased from 76.8 to 86.9 wt% of total weight of the mechanically treated spent NiMH battery by elongating grinding duration. At the same time, the coarser fraction amount (> 4 mm) sharply decreased from 9.7 to 1.0 wt% of the total weight. The generated heat during the mechanical treatment evaporates humidity from the battery waste. The solid loss part stuck on the

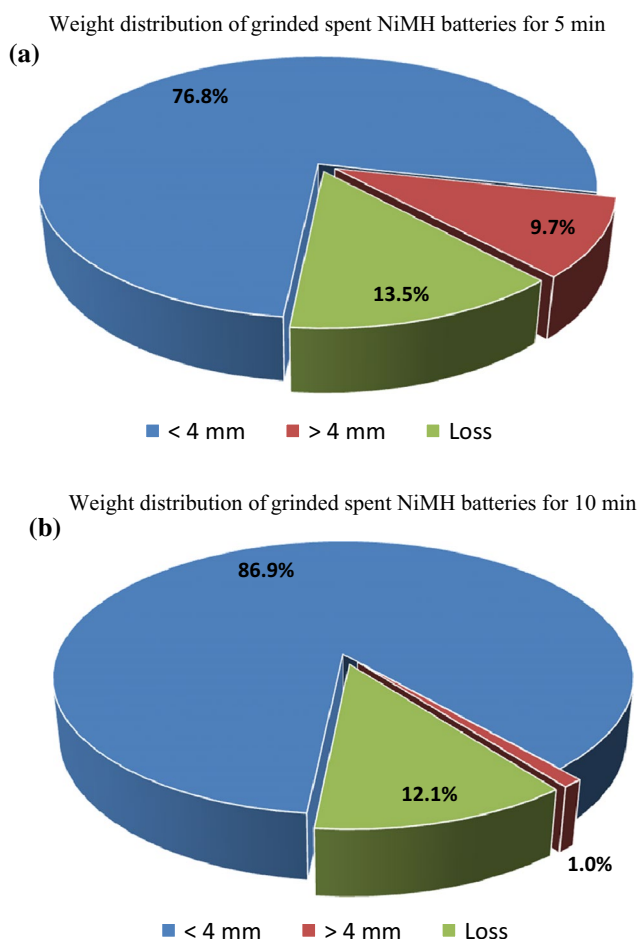


Fig. 4 Weight distribution of ground spent NiMH batteries for **a** 5 min and **b** 10 min

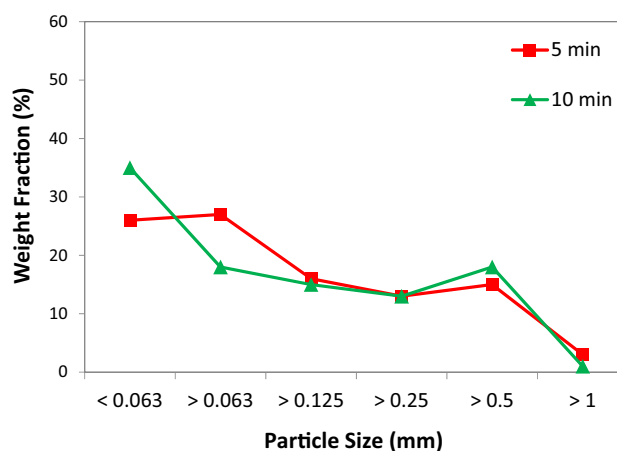


Fig. 5 Particle size distribution for the fine fractions (< 4 mm) that ground for **a** 5 min and **b** 10 min

equipment as it mentioned before. According to results, nearly 87 wt% of the NiMH batteries is suitable for further recycling after the suggested milling method. On the other hand, the coarser fraction is a complex mixture of the organic and inorganic battery parts, and especially battery powder coated onto the battery label (paper) and polymeric parts. The soft and flexible organic parts can plug the sieves. Considering the small fraction amount and also its possible side effect to the sieving step, finer sorting/enrichment was applied only to fine fractions.

Particle size distribution for the fine fractions (< 4 mm) of ground samples were measured by sieving analyses and results are given in Fig. 5. Weight fraction of the both samples are nearly same for size ranges between 0.125 and 4 mm. Although the finest particle fraction (< 0.063 mm) amount for the 10-min ground sample is higher than the shorter grinding time, the total weight fractions at < 0.125 mm size range for the both sample is equal, which is 53 wt%.

Chemical composition of the fine fraction (< 4 mm) of ground NiMH batteries for 5 and 10 min and the samples after size classification were detected as shown in Tables 1 and 2. The fine fraction of the battery black mass ground for 5 min contains nearly 44.3 wt% Ni, 14.0 wt% Fe, 3.6 wt% Co, 6.1 wt% La, 1.7 wt% Nd, 1.6 wt% Ce, 1.4 wt% Pr and other minor elements. When we analyzed the chemical composition after the size classification, it was detected that Ni and Co contents increased in finer fractions smaller than 0.25 mm. Fe showed an opposite behavior and enriched in the samples bigger than 0.25 mm. REEs (La, Nd, Ce and Pr) content concentrated at very fine particle size range as also observed in lab-scale gravity separation from ores [31, 32]. REEs total amount reached to 15.3 wt% for NiMH battery black mass smaller than 0.063 mm.

The fine fraction of the battery black mass ground for 10 min contains nearly 44 wt% Ni, 11.7 wt% Fe, 4.9 wt% Co, 6.4 wt% La, 0.95 wt% Nd, 2.14 wt% Ce, 0.89 wt% Pr and other minor elements. Larsson et al. [33] presented the metal distribution in HEV NiMH battery—one cell having almost similar composition comparing to AA- and AAA-type NiMH batteries except Fe amount. The analyzed HEV NiMH battery contained 30.8 wt% Fe, which is much higher than household-type NiMH batteries. After the size classification, the elemental distribution of the sample is nearly same as shorter time ground battery black mass. However, Ni, Co and REEs contents exhibit a

positive linear relation with decreasing particle size. On the contrary, Fe mainly concentrated at coarser fraction and its amount sharply reduced by decreasing particle size. REEs total amount reached to 14.6 wt% for NiMH battery black mass smaller than 0.063 mm, which is richer than most of the REE deposits [31, 32].

Table 3 shows the carbon composition of the NiMH battery black mass depending on particle size. It is clear that organic components, which passed through the 4-mm sieve during the mechanical treatment, such as separator and battery label, can be easily separated from finer fraction by sieving. The carbon analysis results for > 1 mm sample fraction

Table 1 Elemental analyses of ground NiMH batteries for 5 min, and after sieving as a function of particle size

Elements	Ground sample for 5 min, % (w/w)						
	< 4 mm ^a	> 1 mm	> 0.5 mm	> 0.25 mm	> 0.125 mm	> 0.063 mm	< 0.063 mm
Al	0.85 ± 0.011	0.87 ± 0.021	0.14 ± 0.002	0.34 ± 0.010	1.45 ± 0.036	1.03 ± 0.029	0.98 ± 0.018
Ce	1.55 ± 0.005	1.22 ± 0.022	0.04 ± 0.008	0.46 ± 0.003	1.34 ± 0.034	1.98 ± 0.051	2.38 ± 0.054
Co	3.58 ± 0.122	3.36 ± 0.076	0.45 ± 0.148	1.59 ± 0.035	5.62 ± 0.133	4.70 ± 0.047	4.68 ± 0.097
Fe	14.03 ± 0.513	10.53 ± 0.291	78.33 ± 1.519	33.12 ± 0.288	11.84 ± 0.166	1.82 ± 0.020	1.05 ± 0.014
K	0.66 ± 0.007	0.84 ± 0.022	0.09 ± 0.001	0.34 ± 0.005	0.75 ± 0.022	0.73 ± 0.022	0.67 ± 0.014
La	6.13 ± 0.058	5.37 ± 0.098	0.57 ± 0.007	1.96 ± 0.015	5.24 ± 0.152	7.69 ± 0.214	9.47 ± 0.170
Mg	0.11 ± 0.002	0.07 ± 0.002	–	–	0.09 ± 0.003	0.17 ± 0.006	0.17 ± 0.003
Mn	0.95 ± 0.009	0.91 ± 0.024	0.19 ± 0.002	0.34 ± 0.005	0.88 ± 0.022	1.18 ± 0.031	1.15 ± 0.021
Na	0.35 ± 0.005	0.42 ± 0.010	0.05 ± 0.002	0.21 ± 0.003	0.48 ± 0.013	0.47 ± 0.024	0.36 ± 0.005
Nd	1.67 ± 0.019	1.15 ± 0.031	0.07 ± 0.006	0.41 ± 0.005	1.44 ± 0.041	2.29 ± 0.057	2.30 ± 0.060
Ni	44.34 ± 1.695	41.31 ± 1.069	9.03 ± 0.774	38.26 ± 0.463	53.69 ± 1.054	53.44 ± 0.511	49.82 ± 1.004
Pr	1.40 ± 0.004	1.00 ± 0.016	0.03 ± 0.005	0.39 ± 0.004	1.13 ± 0.033	1.75 ± 0.050	2.19 ± 0.048
Zn	1.28 ± 0.017	1.18 ± 0.029	0.14 ± 0.003	0.74 ± 0.005	1.57 ± 0.032	1.51 ± 0.040	1.54 ± 0.016

^aAll of the samples finer than 4 mm after grinding

Table 2 Elemental analyses of ground NiMH batteries for 10 min, and after sieving as a function of particle size

Elements	Ground sample for 10 min, % (w/w)						
	< 4 mm ^a	> 1 mm	> 0.5 mm	> 0.25 mm	> 0.125 mm	> 0.063 mm	< 0.063 mm
Al	0.92 ± 0.068	0.10 ± 0.002	0.25 ± 0.003	0.46 ± 0.001	0.67 ± 0.004	0.82 ± 0.019	1.17 ± 0.030
Ce	2.14 ± 0.148	0.18 ± 0.004	0.49 ± 0.009	0.96 ± 0.013	1.64 ± 0.017	2.26 ± 0.052	2.98 ± 0.121
Co	4.90 ± 0.384	0.54 ± 0.006	1.35 ± 0.025	2.73 ± 0.015	4.42 ± 0.018	5.52 ± 0.116	6.99 ± 0.197
Fe	11.70 ± 0.806	66.21 ± 0.498	57.79 ± 0.765	27.83 ± 0.156	6.93 ± 0.013	4.30 ± 0.094	1.93 ± 0.049
K	1.15 ± 0.088	0.21 ± 0.002	0.42 ± 0.006	0.72 ± 0.002	1.29 ± 0.435	1.14 ± 0.027	1.37 ± 0.023
La	6.41 ± 0.455	0.72 ± 0.005	1.51 ± 0.020	3.16 ± 0.029	5.31 ± 0.013	7.31 ± 0.155	9.05 ± 0.338
Mg	0.003 ± 0.001	–	–	–	–	0.012 ± 0.002	0.002 ± 0.0003
Mn	1.85 ± 0.132	0.30 ± 0.005	0.60 ± 0.010	0.81 ± 0.003	1.25 ± 0.009	1.70 ± 0.038	2.50 ± 0.088
Na	0.09 ± 0.010	–	–	0.03 ± 0.001	0.07 ± 0.001	0.07 ± 0.003	0.10 ± 0.002
Nd	0.95 ± 0.064	0.03 ± 0.007	0.15 ± 0.008	0.35 ± 0.004	0.62 ± 0.006	0.88 ± 0.022	1.33 ± 0.055
Ni	43.95 ± 3.207	13.69 ± 0.088	22.02 ± 0.342	39.89 ± 0.217	45.78 ± 0.164	42.73 ± 0.874	48.57 ± 1.338
Pr	0.89 ± 0.066	0.03 ± 0.003	0.14 ± 0.001	0.38 ± 0.006	0.71 ± 0.004	1.00 ± 0.021	1.24 ± 0.046
Zn	1.11 ± 0.077	0.18 ± 0.002	0.36 ± 0.003	0.71 ± 0.004	1.05 ± 0.002	1.13 ± 0.025	1.31 ± 0.023

^aAll of the samples finer than 4 mm after grinding

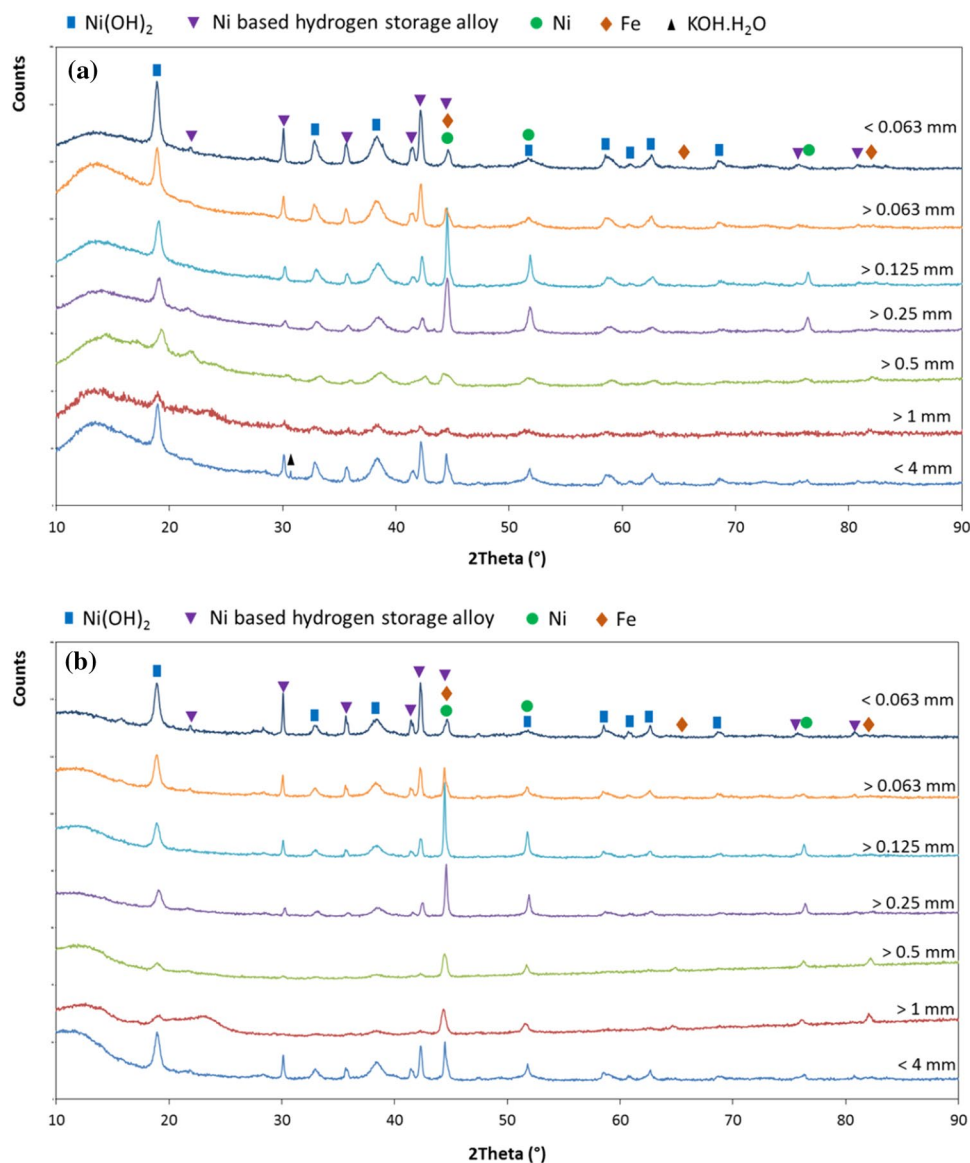
Table 3 Carbon analyses of ground NiMH batteries for 5 and 10 min as a function of particle size

Particle size	Carbon amount w%	
	Ground sample for 5 min	Ground sample for 10 min
< 4 mm ^a	3.58 ± 0.8301	3.05 ± 0.532
> 1 mm	7.03 ± 3.61	7.29 ± 4.13
> 0.5 mm	5.44 ± 1.230	1.93 ± 0.901
> 0.025 mm	2.45 ± 0.920	4.74 ± 1.41
> 0.0125 mm	2.34 ± 0.634	3.37 ± 0.796
> 0.0063 mm	2.00 ± 0.142	2.99 ± 0.315
< 0.0063 mm	2.10 ± 0.132	2.94 ± 0.18

^aAll of the samples finer than 4 mm after grinding

presents high standard deviation due to heterogeneous composition of organic parts, iron and nickel-based components. The amount of the organic content has a decreasing trend by reducing particle size. Reducing the carbon amount can be beneficial for the further recycling step for Ni and REEs which is leaching and solvent extraction. Ni and REEs can be adsorbed by various carbonaceous materials [34–39]. Thus high carbon amount can reduce the leaching efficiency of the valuable metals due to adsorption features of carbon. Moreover, carbon and organic compounds cause a problem in the filtration step of the leachate after dissolution of the metallic content [40].

XRD pattern of the samples is given in Fig. 6. The total battery black masses (< 4 mm) for both grinding durations have a complex phase structure, and mainly contains nickel-based hydrogen storage alloy, Ni(OH)₂, iron and nickel. The

Fig. 6 XRD pattern of the ground samples for **a** 5 min and **b** 10 min

characteristic diffraction peaks of Ni(OH)₂ (PDF Card no.: 04-013-4402) can be clearly observed at 19.1°, 33.0° and 38.5° 2θ degrees. The overt peaks at 30.1°, 35.8°, 41.7°, 42.3° and 44.6° 2θ degrees are indexed with AB₅-type hydrogen storage alloys [La_{0.5}Ce_{0.3}Pr_{0.1}Nd_{0.1}(Ni,Co,Al,Mn)₅, PDF Card no.: 04-008-6028 and 04-019-4647]. The highest peak (110) of body centered cubic Fe and Ni locate nearly same position at around 44.5° 2θ degree. Although it is difficult to distinguish them, (200) and (220) diffractions of Fe were detected at 65.8° and 82.5° 2θ degrees, as well as (200) and (220) diffractions of Ni were identified at 51.9° and 76.6° 2θ degrees. The majority of the peaks for the smaller fraction of the samples, > 0.063 mm and < 0.063 mm, represent the existence of Ni(OH)₂ and nickel-based hydrogen storage alloy in the structure. XRD patterns of the ground sample for 10 min obviously show that Fe peaks became dominant for the coarser samples, > 1 and > 0.5 mm.

The mass balance for the mechanical treatment of the spent NiMH batteries was calculated for 1000 g starting material amount using experimentally obtained data for the 10-min ground sample. This sample was chosen for the mass balance because of the high material collection amount for fine fraction (< 4 mm), which is rich in valuable metal content. Figure 7 presents the mass balance of the studied mechanical treatment method for the 10-min ground spent NiMH batteries. The difference between the masses of the samples and cumulative masses of the analyzed elements is due to the existence of other elements, such as oxygen, hydrogen and non-metallic components. The majority of the ground battery waste is collected in the fine particle size

fraction which is smaller than 4 mm. After sorting the fine fraction by size, it is clear that main Fe amount by weight resides in size fraction bigger than 0.25 mm with considerable amount of Ni. REEs and Ni by weight were enriched in the size fraction smaller than 0.25 mm.

Conclusion

Valuable contents of the NiMH battery waste were successfully enriched by a simple mechanical pretreatment. After mechanical pretreatment nearly 87 wt% of the NiMH battery waste is suitable for further recycling steps due to its high valuable metal content. The ground samples were sieved and then chemical and material characterization methods were applied to all of the samples, smaller than 4 mm, and the six different size fractions. The results clearly showed that Fe content was enriched in the coarser fraction of under-size of 4, > 0.25 mm, while Ni and REEs amounts were concentrated in the finer fractions (< 0.25 mm). Thus after grinding, size classification by sieving is a simple but an efficient step to enrich the metal contents in a specific size fraction. Organic content of the battery black mass reduced by decreasing particle size, which can ease the further leaching and filtration steps. The phase composition of the finer fractions is mainly AB₅-type nickel-based hydrogen storage alloy and nickel hydroxide. The mechanical pretreatment is a simple and efficient enrichment method which can help to reduce chemical consumption in a hydrometallurgical NiMH battery recycling process.

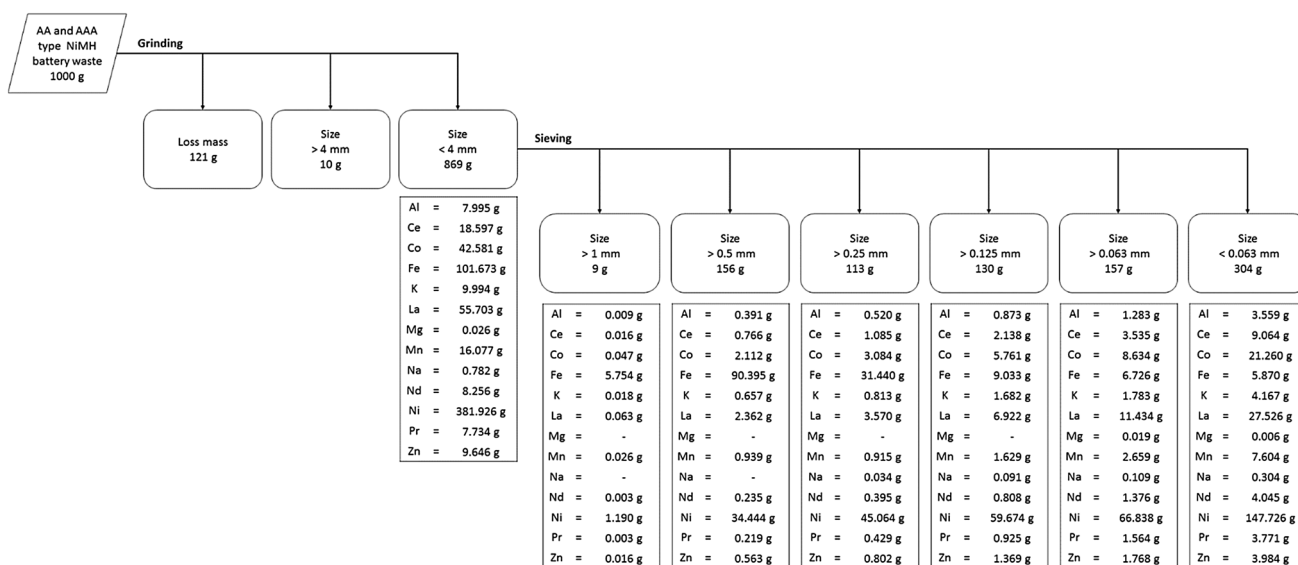


Fig. 7 Mass balance for mechanical treatment of spent NiMH batteries for the 10-min ground sample (the difference between the masses of the samples and cumulative masses of the analyzed elements is due

to the existence of other elements, such as oxygen, hydrogen, etc., and non-metallic components)

Various types of household battery waste such as spent alkaline, NiMH and lithium-ion batteries are generally collected together, and then a sorting depending on battery chemistry is required for the recycling. Thus further research should focus on mechanical treatment and enrichment of the other battery types especially lithium-ion batteries and also mixed battery waste to develop alternative processes and sustainable waste management strategies for batteries.

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