

Review

Preprocessing and Leaching Methods for Extraction of REE from Permanent Magnets: A Scoping Review

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Abstract: The demand for REEs is continuously increasing in the European Union due to the rapid development of high-tech applications that contain REEs, mainly those based on electrification. However, the REE supply in Europe is limited because of the exclusive production of these metals by third-world countries. The European supply/demand gap for REEs can be covered with the development of recycling technologies from secondary resources, such as REE permanent magnets. NdFeB and SmCo magnets are the two main categories of REE-containing permanent magnets. In the following work, studies focusing on the preprocessing and leaching methods in order to extract REEs were identified and discussed. Although preprocessing includes controversial steps, i.e., milling and demagnetizing, numerous studies have focused on the leaching of REEs from NdFeB magnets using either inorganic or organic solvents. Meanwhile, the literature based on Sm recovery methods from SmCo magnets has been limited.

Keywords: permanent magnets; REE; NdFeB; preprocessing; leaching; electrification



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1. Introduction

Rare Earth Elements (REEs) consist of the lanthanides (La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu) and the elements yttrium Y and scandium Sc, which are divided into two main subgroups: the Heavy REEs (HREEs) from Gd to Lu and the Light REEs (LREEs) from La to Eu, based on their atomic number. Within LREEs, neodymium (Nd) and samarium (Sm) are included [1–3].

The REEs exhibit exceptional physical properties, which have made them necessary in the development of high-tech applications, such as smartphones, digital cameras, computer hard disks, fluorescent and light-emitting-diode (LED) lights, flat-screen televisions, computer monitors, and electronic displays. For example, the permanent neodymium-iron-boron magnets are essential for elaborating the electrification of vehicles and wind energy. In addition, portable electronic devices, such as computers and mobile phones, consist of NiMH batteries containing REEs (La, Pr, and Ce) [4–7].

The European Commission has committed to announcing at least every three years a list of the materials and elements that are crucial for the technological development of Europe. This list is defined based on two pillars, the supply risk and the economic importance of the materials for the European area. REEs were included in the CRMs from the first published list in 2011, while the REEs were mentioned in the CRMs' list, as HREEs and LREEs, in 2014 [8,9].

The supply risk is dependent on the main producers and suppliers of the global primary production of rare earths, and the dependence of the European Union (EU) on external imports to cover its demands and geopolitical conditions that are creating concerns regarding defining reliable REE sources. On the other hand, the economic importance is

examined according to the end-user applications. REEs belong to Critical Raw Materials according to the most recently updated list of the European Commission of 2020 [10].

Especially for the REEs, China is the leading producer of global primary production covering 60%, while the primary production of CRMs is extremely limited within the European Union area. At the same time, China covers 98% of imports of REEs to the EU. However, China recently announced restrictions on REE exportations, citing the increase in their demands in the internal market and the environmental impact of the continuous mining operations [11–14].

For instance, the production of electric vehicles decreased from 2019 to 2021 by 1.7 million vehicles despite the increased demand for electric vehicles. That happened due to the ongoing shortage of electronic microchips containing REEs, which are needed for several operations of electric vehicles, such as engine management systems. Hence, the limitation of the REEs could result in a high supply risk and limited application development in the EU [15–17]. Therefore, the created gap between the supply and demand of REEs could be covered by recycling rich secondary sources of REEs.

Among the most important industrial applications of REE, in metallurgy, they are employed as raw materials for metal refining and metal alloying, while they are also used as catalysts in the automotive and petrochemical industries. They are also met in many products and goods in everyday life, such as cell phones, LED lights, lasers, rechargeable solid-state batteries (Ni-MH), fiber optics, and solar panels. Moreover, they are crucial parts of cutting-edge technologies, i.e., high-performance permanent magnets, hydrogen storage applications, superconductors, and fuel cells. All these materials are vital for numerous emerging technologies including hybrid cars, cell phone parts, and wind turbines [18–22] (Figure 1).



Figure 1. Main applications of Rare Earth Elements [18,19,23–25].

REEs are used in various applications in several technological and industrial sectors. According to recent data of 2020, the REEs are mainly used in the catalysts and magnets, where more than 50% of the total mined amount is used. However, the total consumption of the REE for magnets is forecast to increase until 2035 because of the demand boost for electric motors, which include several magnetic components of REEs [18,19,23–25].

Therefore, permanent magnets on electrification would be the main application of REEs with significantly increased demands in the future. For electrification applications, four main types of permanent magnets are used, namely Neodymium Iron Boron Magnets (NdFeB), Ferrite Magnets, Samarium-Cobalt Magnets (SmCo), and Alnico Magnets (AlNiCo). However, only NdFeB and SmCo permanent magnets incorporate REEs, especially neodymium (Nd), dysprosium (Dy), praseodymium (Pr), and samarium (Sm) [22].

To be more precise, neodymium permanent magnets are mainly composed of neodymium, iron, and boron alloys in a tetragonal crystalline structure. Except for Nd, additional REEs such as Dy and Pr are included in the Nd magnet in order to provide supplementary properties in the materials according to their use. For instance, the Nd magnets are considered the magnets with highest strength among all reported magnets. As a consequence, they are used in a wide range of applications. However, by adding dysprosium and praseodymium in Nd magnets, the resistance to high temperatures and chemical resistance tolerance can be improved [26,27]. On the other hand, samarium permanent magnets mainly consist of samarium-cobalt alloy including Fe, Cu, and Ni traces [28]. The composition of the metals in the magnets alloy defines the physicochemical properties, while, depending on the application, the concentration of the metals varies accordingly [29–34] (Table 1).

Table 1. Typical chemical composition of Nd and Sm permanent magnets [29–34].

Type of Magnet	Element	Range
NdFeB magnet	Nd	22–31%
	Fe	53–67%
	B	0.74–1.04%
	Pr	0.07–13%
	Ni	0.02–6.4%
	Co	0.54–4.2%
	Dy	0.77–4.2%
SmCo magnet	Sm	22–40%
	Co	50–66%
	Fe	0.02–16.04%

2. Research Methodology

2.1. Research Questions

A scoping review was conducted in order to systematically map the research that has been performed in this scientific area, as well as to identify possible existing gaps in the methodology of preprocessing and leaching for the extraction of REEs from permanent magnets. The following research questions guided our review:

What is known from the literature about the methods for the extraction of REEs from permanent magnets?

What are the main recycling issues raised during permanent magnets treatment at the end-of-life of permanent magnets? How can they be identified? How can we find existing gaps in the knowledge of preprocessing and leaching methods for extracting REEs from permanent magnets?

2.2. Search String

Our search was based on the following string that was used for the identification of relevant studies and reports:

“neodymium magnets recycling” OR “samarium magnets recycling” OR “rare earth elements recovery”) AND (“preprocessing” OR “demagnetization” OR “rare earth leaching”).

2.3. Selection of Articles

As of April 2022, numerous publications were retrieved based on the “Neodymium permanent magnets recycling” and “Samarium permanent magnets recycling”. More than 6145 publications were detected related to the recycling of Nd from magnets and 2080 publications were dedicated to the recycling of Sm from magnets. However, in this manuscript, publications regarding the purification, separation, and extraction steps are

also included. The current study is also focused on the preprocessing of Nd and Sm permanent magnets and the leaching of the REEs out of them.

It is worth mentioning that the literature on preprocessing and leaching of Sm from SmCo permanent magnets is limited. Most of the already published research focuses on the liquid–liquid separation of Sm and Co from leachate solutions after the leaching process.

3. State of the Art of REE Recovery Methods

The recycling of permanent magnets is challenging due to their preprocessing, purification, and separation steps, which demand a high cost, resulting in the process being inaccessible to those with limited processing resources. Thus, recycling REEs from permanent magnets has not been developed and operated on a commercial scale. However, several researchers have focused on investigating REE extraction from NdFeB and SmCo magnets through either pyrometallurgical, hydrometallurgical, or even a combination of both methods [35] (Table 2).

Table 2. Operating conditions of different recycling processes.

Process	Source	Steps	Experimental Conditions	Reagents	Reference
Pyrometallurgical	Swarf/sludge	1. Oxidation 2. Melting	1300–1550 °C 1–16 h	O ₂ , C, Ar	[36]
	Solid scrap	Hydrogen decrepitation 1. Hydrogenation 2. Vacuum desorption	125–450 °C 270–1000 °C	H ₂	[37,38]
Hydrometallurgy	Solid scrap	Leaching	110 °C 6 h	HCl, oxalic acid	[39]
	Solid scrap	Leaching	27 °C 15 min	H ₂ SO ₄	[40]
Chlorination	Solid scrap	Chlorination 1. Heating 2. Leaching	300 °C 3 h	NH ₄ Cl	[41]

However, the pyrometallurgical method, although a common process for metal extraction from EoL materials, incorporates high temperatures and high-energy processing steps; thus, the cost and the environmental footprint increase. On the other hand, the hydrometallurgical process is based on the extraction of metal ions in a liquid medium solvent at a medium temperature (25–95 °C). Normally, hydrometallurgy could be considered a more environmentally friendly process, as low energy consumption and small-unit plant installation are required [42,43]. The main benefits and disadvantages of pyrometallurgy are compared to those of hydrometallurgy in Table 3.

Table 3. Comparison between pyrometallurgical and hydrometallurgical recycling process [42,43].

Method	Advantages	Disadvantages
Pyrometallurgical	No mechanical pretreatment High recovery efficiency Processing a wide range of materials independently of their composition	Operating at high temperature Using additional materials as collector Hazardous emission impact on the environment
Hydrometallurgical	High recovery efficiency Mild conditions Conduction of low-grade materials Better control of co-products Simple stages	High water consumption High water wastes

4. Preprocessing and Hydrometallurgical Methods

The current work is focused on the literature review regarding the hydrometallurgical REE recovery from REE permanent magnets, especially in the preprocessing and leaching

steps. Thus, the proposed hydrometallurgical route is based on the following main steps: collection, sorting, preprocessing, and leaching (Figure 2).

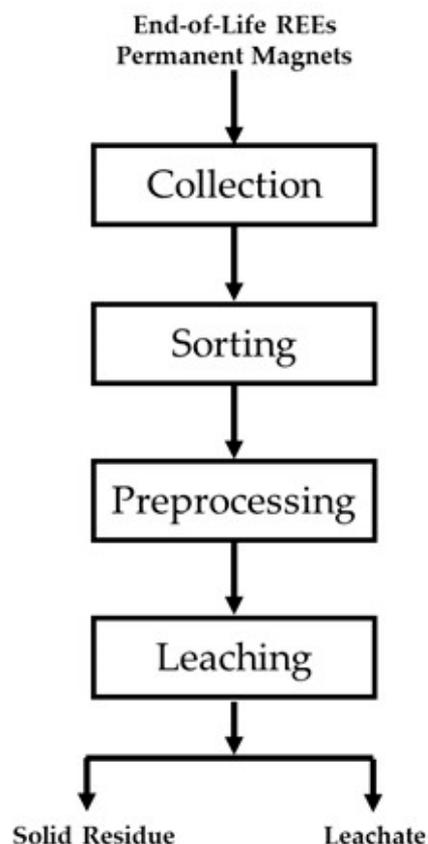


Figure 2. Flowsheet of the permanent magnet hydrometallurgical process.

4.1. Collection—Sorting

As already reported, permanent magnets are used in several applications, from handheld digital and electronic devices to large-scale applications such as wind turbines. In addition, the life cycle of permanent magnets depends on the application they have been installed on. For example, the electric vehicle permanent magnet lifetime is 10 times lower than those of the permanent magnets installed on wind turbines [44–46]. At the same time, handheld devices usually end up in landfills, the environment, or recycling routes for other contained materials, such as iron alloys. The sorting of permanent magnets from the included devices is complicated due to the presence of additional ferrous materials and their small size. However, in France, a collection, dismantling, and sorting unit of computers for the manual separation of hard disk drives (HDDs) is operated by SUEZ in order to develop an efficient dismantling process to recover permanent magnets from EoL electronic products [47,48].

4.2. Preprocessing

4.2.1. NdFeB Permanent Magnets

An integral part of the recycling process is the preprocessing step, which is vital for the development of an efficient recycling process. However, the preprocessing of permanent magnets is defined as a hazardous process because of the materials' flammability. In order to eliminate the chance of ignition, vacuum or inert atmosphere methods are suggested for the preprocessing (under the absence of oxygen) in order to avoid the material's ignition of the materials. During the step of preprocessing, the material is smashed, creating new surfaces on magnets that interact with the environmental conditions due to the removal of the protective coating. Thus, rare earths contained in the magnet come into contact with the

air and humidity and they are consequently rapidly oxidized [49,50]. This is based on the unavoidable thermodynamically favored phenomenon that spontaneously occurs due to their strongly negative electric potential (-2.2 to -2.5 V) [51]. For that purpose, samarium is often alloyed with cobalt to obtain high protection against corrosion [52], whereas neodymium magnets are more prone to corrosion [53]. In the case of Nd magnets, two types of corrosion could be caused, namely white and red corrosion. The red corrosion is the surficial corrosion of the magnet, which arises in roughly the same way as rust appears on ferrous materials. On the other hand, white corrosion penetrates the bulk magnet reaching each magnetic grain [54]. Therefore, the combination of high temperature and air humidity leads to the oxidation of the binding material, pure Nd, forming Nd hydroxide (NdOH). Thus, the magnet turns into powder, and a high probability of explosion arises [55–57].

Hence, selecting the proper preprocessing is of high importance to avoid extreme conditions and the case of ignition [58]. Within the available literature, the extreme corrosion of magnets and the possibility of ignition are not extensively mentioned. The main steps of the preprocessing are focused on the demagnetization of magnets and the milling to obtain a nonmagnetic powder. In some research studies, additional preprocessing steps were attempted to improve and simplify the leaching step to follow (Figure 3).

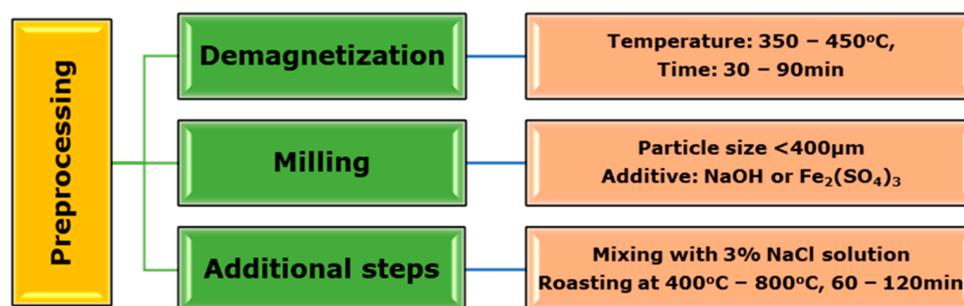


Figure 3. Proposed main steps of the NdFeB magnets preprocessing.

The demagnetization step is usually applied to remove the magnetic properties of permanent magnets to avoid any magnetization effect between the metallic parts of the used equipment and the magnetic parts of preprocessing equipment. Thus, this process is performed by heating the magnets at an elevated temperature above the temperature where the material loses its magnetic properties. This temperature is called the Curie temperature and varies for different types of magnets. The Curie temperature of NdFeB and SmCo magnets is 300 °C and 850 °C, respectively.

Additionally, the milling step is vital before the conduction of the leaching process. The smashing and reduction in the material's particle size (p.s) affect the efficiency of the following process due to the increase in the reaction surface. However, the excessive decrease in the granulometry could cause filtration delays regarding the separation of liquid from the solid phase after leaching.

Erust et al. [40] and Ciro et al. [32] performed the demagnetization of NdFeB magnets from a HDD at similar temperatures and time durations, 350 °C and 400 °C for 30 min and 45 min, respectively. Then, the demagnetized magnets were milled in fine powder with granulometry under 500 µm. On the other hand, Kitagawa et al. [59] and Kataoka et al. [60] proposed the forced corrosion of the demagnetized and milled powder using NaCl solution in 3% *w/v*. The powdered sample was mixed with the NaCl solution for one week, in order to accelerate the oxidation on the surface of the magnets. Bonin et al. [29] tested their leaching method in magnet pieces and powder after demagnetization to verify the necessity of the preprocessing. The demagnetization was conducted at 350 °C for 60 min, and the sample was milled in particle size under 250 µm. In that case, the absence of the demagnetization step did not affect the efficiency of the process, accomplishing the 100% recovery of REEs in both cases. The examined roasting step did not further increase the REE

extraction efficiency. However, the leaching of REEs was tested in nonpretreated samples in order to avoid the high cost of preprocessing, resulting in the low efficiency of the process.

Therefore, the preprocessing, which includes the demagnetization and milling of the NdFeB, is critical and necessary to facilitate and enhance the dissolution of REEs in leaching solvent [27] (Table 4).

Table 4. Proposed preprocessing methods of NdFeB permanent magnets.

Source	Demagnetization Conditions	Milling Process	Additional Preprocessing Steps	Recovery of REE	Reference
Hydrogen decrepitated NdFeB powder	400 °C, 90 min	<355 µm	-	95% REE (Nd, Pr, Dy)	[31]
NdFeB magnet from HDD	400 °C, 45 min	<420 µm	-	98% Nd	[32]
NdFeB magnet	Demagnetized	Milled material	Mixing with 3% NaCl solution for 1 week	97% Nd	[59,60]
Magnets from Spent Hard Disc Drives (HDDs)	350 °C, 30 min	<500 µm	-	100% Nd, 80% Dy	[40]
NdFeB magnet scraps	-	Adding NaOH solution, 30 min	Roasting at 400 °C, 120 min	94% Nd, 93% Dy	[61]
Nickel-coated magnet	nonmagnetic material	Adding Anhydrous iron (III) sulfate	-	100% REE (Nd, Dy)	[62]
NdFeB magnet from HDDs	350 °C, 30 min	-	-	98% REE (Nd, Pr, Dy, Tb)	[27]
Scrap NdFeB permanent magnet	-	106–150 µm	-	100% Nd	[63]
NdFeB magnet	350 °C	<250 µm	-	100% REE (Nd, Dy)	[29]
NdFeB magnet	Demagnetized	<400 µm	Roasting at 800 °C, 60 min	80% Nd, 20% Dy	[30]

Therefore, demagnetization and milling as preprocessing steps are mainly proposed for enhancing the recovery of REEs (Nd, Dy, and Pr) from neodymium permanent magnets. The investigated demagnetization temperature ranged from 350 °C to 400 °C, above the Curie temperature, for 30–90 min, achieving high REE recovery. However, the recovery of Dy increased from 80% to 95% after temperature thermal treatment at 350 °C for 30 min and 400 °C for 90 min. In addition, the reduction in the magnet's granulometry from 500 µm to 250 µm did not affect the recovery of Nd instead of Dy, which was increased from 80% to 100%, respectively. At the same time, the additional preprocessing steps, such as mixing the demagnetized, milled powder with NaCl solution or further roasting the material at 800 °C, did not significantly improve the recovery of REEs. To summarize, the demagnetization step is necessary to facilitate the following steps at 350 °C for 30 min, while the milling in granulometry under 250 µm is proposed to improve the efficiency of the leaching.

4.2.2. SmCo Permanent Magnets

On the other hand, the preprocessing of SmCo permanent magnets is less demanding with respect to NdFeB as SmCo powder does not violently react with O₂, avoiding the ignition problem. Thus, the research on the preprocessing of SmCo magnets is focused on the conditions of crushing and milling steps [64] (Table 5).

However, according to Sinha et al. [28], demagnetization as preprocessing is mandatory to avoid sticking magnetic material in the metallic parts of the reactor during the leaching process. In addition, the roasting at a high temperature will reform the Sm metal into oxide. The demagnetization is suggested after the milling step at 850 °C for 6 h under an air atmosphere, where Sm oxide is formed as a result of the thermogravimetric analysis (TGA) and X-ray diffraction analysis (XRD), which confirmed the presence of SmCoO₃ and the absence of SmCo₅ alloy [28,65]. The milling step affects the efficiency of the leaching process, as was proved in the research of Tao et al. The recovery of Sm was increased from

75% to 95% when the particle size was reduced from 125 μm to 74 μm . However, a further decrease in granulometry seems not to further affect the leachability of Sm [33].

Table 5. Proposed preprocessing of the SmCo permanent magnets.

Source	Demagnetization Conditions	Milling Process	Additional Preprocessing Steps	Recovery of REE	Reference
SmCo magnet ring	850 °C	<150 μm	-	99.9% Sm	[64]
Cobalt-based magnetic scrap	-	74 μm	-	95% Sm	[33]
Machined scrap SmCo5 magnet	-	106–150 μm	-	95% Sm	[66]

Although the existing literature on SmCo permanent magnet preprocessing is very limited, demagnetization at 850 °C and milling in granulometry under 150 μm are proposed for higher recovery rates.

4.3. Hydrometallurgical Process for the Recovery of REE from Permanent Magnets

4.3.1. Recovery of REE from NdFeB Magnets

The crucial step for extracting REEs from permanent magnets is the leaching step, in which the REE is dissolved in a solution creating complexes depending on the leaching medium. In the hydrometallurgical process, the medium needs to be selective, prohibiting impurities such as Fe and B from NdFeB magnets and Co from SmCo magnets from being leached. Thus, several studies were recorded for the leaching of REE, mainly Nd and Dy in the case of NdFeB permanent magnets, using inorganic (HCl, HNO₃, and H₂SO₄) or organic (acetic, citric, and oxalic acid) solutions [67]. However, the recovery of REEs from permanent magnets is equally affected by preprocessing and leaching steps. Thus, plenty of research groups have already studied the combination of the appropriate preprocessing steps, the medium, and the conditions of the REE extraction (Table 6).

Table 6. Recovery yields of REE from NdFeB permanent magnets for different leaching mediums and conditions.

Pretreatment	Leaching Medium	Leaching Conditions	Recovery
Demagnetization: (optional) Milling: p.s <400 μm	Organic Acids (Acetic Acid)	Acid Conc: 0.1–1 M, S/L: 1–5% w/v, 80–90 °C, 3–24 h	93–99% REEs
Demagnetization: (optional) Milling: p.s <400 μm	Inorganic Acid (HCl)	Acid Conc: 0.2–2 M, room temperature, S/L: 5–10% w/v, 2–3 h	>97% REEs
Demagnetization: 350 °C, 30 min Milling: p.s <500 μm	Inorganic Acid (H ₂ SO ₄)	Acid Conc: 2 M, room temperature S/L: 1–5% w/v, 15–60 min	>95% REEs
Demagnetization: 400 °C, 45 min Milling: p.s <420 μm	Inorganic salt (Ammonium persulfate)	Salt Conc: 1.3 M, S/L: 2% w/v, 75 °C, 15 min	97% Nd
Milling with Anhydrous iron (III) sulfate	Water	S/L: 3.33% w/v, 200 rpm, 25 °C, 24 h	100% REEs
Chlorination NH ₄ Cl at 500 °C for 3 h, N ₂ gas flow (50 mL/min)	Water	Soaking	90% REEs (Nd, Dy)

First, Klemettinen et al. [27] conducted a leaching experiment in nonmagnetic NdFeB magnets in crude form and pieces of demagnetized and broken magnets using the H₂SO₄, HCl, and HNO₃ acids. According to the results, the leachability of REEs was higher (2–4 times higher) for demagnetized magnets in pieces rather than the magnets in their initial form. Therefore, the stages of crushing and demagnetization, at 350 °C for 30 min, are necessary to increase the efficiency of the REE recovery. In the leaching process, the REE

recovery was achieved at 95.45% and 97.72%, with high selectivity for the nickel coating of the magnets in the optimal conditions: 1 M H₂SO₄ and 2 M HCl at 40 °C, respectively. In contrast, the HNO₃ acid was rejected because of the dissolving of the Ni-coating.

A similar study was performed by Hoogerstraete et al. [30], applying a complete flowsheet for the extraction of REEs (Nd, Pr, and Dy) from NdFeB magnets by inorganic acids (HCl and HNO₃). The most efficient conditions of roasting for the oxidation of metals were selected according to thermogravimetric analysis results. The selected temperature and time were at 800 °C for 10 min, respectively. The recovery yield of Nd was almost 80% after 10 min of treating time using 12 M HCl with an S/L ratio of 10%. This particular process revealed a high selectivity for Nd in contrast to Dy, Co, and Fe, which were not dissolved sufficiently under 20%.

Kataoka et al. [60] and Kitagawa et al. [59] performed a relevant study about the recovery of Nd from NdFeB magnets by applying corrosion with NaCl solution and then leaching with HCl solution. The demagnetized and milled magnets were mixed in a 3% NaCl solution for 1 week, and Nd leachability was achieved at 97% and 100%, respectively. Kitagawa et al. detected 30% of B in the leachate using 2 M HCl at room temperature.

An alternative hydrometallurgical process with mechanochemical pretreatment has also been utilized by Van Loy et al. [62] in nonmagnetic nickel-coated NdFeB magnets. The pretreatment included further milling the powdered magnets using an anhydrous iron (III) sulfate. The optimal mineral-acid-to-powdered-magnet ratio was 4:1 to form water-soluble REE sulfate compounds. Following that, the leaching step with water at room temperature was performed, achieving over 95% recovery of REE. However, this particular process exhibits low selectivity due to dissolving 100% of Co and a significant amount of Fe.

Additionally, Erust et al. [40] studied the recovery of REEs, especially Nd and Dy, from spent hard disc drives with a hydrometallurgical process based on several inorganic acids (HCl, H₂SO₄, and HNO₃) and organic acids (acetic and oxalic). The magnets in pieces were demagnetized at 350 °C for 30 min and then milled under a 500 µm particle size. Inorganic acids are more efficient in the recovery for both Nd and Dy than organic acids, in which the Nd and Dy recovery did not overcome the 50% recovery. On the other hand, the leachability of Nd and Dy was achieved at 100% using the H₂SO₄ solution. The optimal recovery yields of Nd (100%) resulted from 2 M concentration acid at an S/L ratio of 20 g/L at room temperature for 15 min.

The leachability of REEs was further investigated based on organic acids. The REE (Nd, Pr, and Dy) leaching from EoL NdFeB magnets using the organic acids, citric acid and acetic acid, is limited. In particular works, the leachability of REEs using citric and acetic acids was studied in pretreated and unpretreated NdFeB powdered samples after hydrogen decrepitation. The thermal pretreatment was performed at 400 °C for 1.5 h and then the particle size of the powder was below under 355 µm. Gergoric et al. [31] studied the concentration of organic acids (0.1–1 M), the solid-to-liquid ratio (1/30 g/mL to 1/80 g/mL), and the kinetics of the leaching process. The main difference between the thermally pretreated and unpretreated powdered samples was the elemental distribution, confirmed by Energy-Dispersive X-ray analysis (EDX). The elemental distribution in the unpretreated sample was even throughout the analyzed area in contrast with the uneven thermally treated samples. In some regions, higher REE concentrations were observed. The leachability of REEs achieved >95% for both 1 M citric acid and 1 M acetic acid after 24 h. However, the selectivity of the process was very low as Fe and Co were co-dissolved. The reduction in the solid-to-liquid ratio did not significantly affect the leachability of the REE in both cases.

Based on the study of Kitagawa et al. [59], NdFeB magnet scraps were milled by adding NaOH solution, with a stoichiometric molar ratio of 15 for 30 min to form Nd(OH)₃. Then, the mixture was washed with water and calcined at 400 °C for 2 h under atmospheric conditions. Nd and Dy recovery yields were reached at 94.5% and 93.1%, respectively, under specific conditions: 1 M acetic acid and S/L ratio of 1% at 90 °C for 180 min. The

selectivity of the process was relatively high because the main impurity (Fe) dissolution was only 1%.

The Nd leached from NdFeB scrap magnets using acetic acid in a specific fraction particle size of 106–150 μm [59]. The current leaching system followed the dissolution with the shrinking-sphere model according to the kinetics of the studied leaching process. The Nd recovery was 100% with an S/L ratio of 1% and 0.4 M acid concentration at 80 °C for 240 min. However, 100% of F was also dissolved, which is the system's main impurity.

A different approach to dissolving Nd and Dy from sintered NdFeB magnets scrap was used by Masahiso et al. [41]. The chlorination was performed in fine magnets powder, adding an excess of NH_4Cl at 500 °C for 3 h. The formed chloride REE compounds were leached by water, achieving a recovery rate of 90%.

As the magnet scraps were preprocessed, demagnetization in the crude form at 400 °C for 45 min and then milling in particle sizes under 420 μm were proposed. The salt concentration, temperature, and S/L ratio were evaluated in the leaching process. The Nd recovery yield was achieved at 98% at the optimum conditions, which were 1.3 M $(\text{NH}_4)_2\text{S}_2\text{O}_8$ concentration and an S/L ratio of 50 mL/g at 75 °C for 15 min. The high selectivity of the Nd recovery in the current leaching system was confirmed by the Nd precipitate containing traces of impurities, almost 0.1% [40].

Summarizing, the leaching of REEs can be achieved efficiently using either organic or inorganic acids. However, the conduction of the leaching experiment with inorganic acids is preferable because of its implementation at room temperature instead of the organic acids used at high temperatures, 80–90 °C. Additionally, organic acid, especially acetic acid, needs more time (3–24 h) for the extraction of REEs to be accomplished by demagnetized milled Nd permanent magnets, while organic acids, such as H_2SO_4 and HCl, need only a few minutes or hours, 60 min and 2 h, respectively. The solid-to-liquid ratio was maintained at relatively low values indicating low solid concentration. A process worth mentioning with high efficiency involves the leaching of REEs from a mixture of the milled, demagnetized magnet with anhydrous iron (III) sulfate, which was conducted in water at room temperature. Despite the fact that it is a more environmentally friendly process on a lab scale, its implementation is not proposed, especially on an industrial scale, because of its high leaching time, 24 h. On the other hand, the chlorination process is not proposed, due to the leachability of REEs being high enough compared to other methods.

4.3.2. Recovery of Sm from SmCo Magnets

The recycling of EoL SmCo permanent magnets is of great importance as they contain a high amount of Sm. Moreover, this type of magnet also includes cobalt, which is a critical metal that is essential for the novel technology of electrification in the automotive industry [68]. However, the literature about the hydrometallurgical recycling of spent SmCo permanent magnets is relatively limited compared to the recovery of the NdFeB magnets (Table 7) [65].

Table 7. Summary of the studied leaching conditions for the recovery of Sm from SmCo permanent magnets [33,34,64,65,69].

Pretreatment	Leaching Medium	Leaching Conditions	Recovery
Demagnetization: 850 °C (optional) Milling: <150 μm	Inorganic Acid (HCl)	Acid Conc: 3–5 M, S/L: 2–10% w/v, 80–95 °C, 1–2 h	70–99% Sm
Milling: <150 μm	Inorganic Acid (H_2SO_4)	Acid Conc: 2–4 M, S/L: 2–20% w/v, 60–80 °C, 1–2 h	25–95% Sm

An acid hydrometallurgical leaching process was studied after demagnetization and milling of the SmCo magnets. The recovery of Sm was almost achieved at 100% in the optimum conditions: 4 M HCl, 500 rpm, S/L 10% at 95 °C for 120 min. However, this process did not show any selectivity to Sm extraction considering the high extraction of Co,

90% [64]. Zhou et al. [34] also studied the recovery of Sm from Sm-Co alloy using H_2SO_4 as a leaching agent. Most of the leaching parameters were quite similar to the previous ones with a concentration of acid of 4 M, and a solid-to-liquid ratio of 17% at a lower temperature of 60 °C, for less time, 75 min. Nevertheless, the recovery rate of Sm was relatively low at 24.1% instead of Co at 99.9%. Furthermore, Tao et al. [33] tested the H_2SO_4 as a leaching reagent to extract the Sm and Co from a cobalt-based magnetic scrap. The increase in temperature at 80 °C for 1 h and the decrease in S/L ratio to 10% resulted in the high recovery rate of Co and Sm up to 88.5% and 95.4%, respectively. However, this process is not selective in the Sm extraction, because of the dissolution of impurities such as Fe. The hydrochloric acid (15% vol) and sulfuric acid (10% vol) were tested in another study to extract Sm from SmCo magnets after the milling step directly. H_2SO_4 was more effective in Sm extraction than HCl, achieving a high recovery rate of 95% instead of 70%, respectively. The high recovery efficiency was reached with an S/L ratio of 50 g/L at 80 °C for 1 h [65]. An alternative hydrometallurgical route is an acid-free dissolution route using a copper (II) salt, especially CuSO_4 , to leach the REE from magnetic materials. The copper ion has the ability to oxidize and dissolve the metallic REE in the solution and receive metallic copper or copper oxide with high purity as a by-product. Inman et al. proposed this process, especially for recovering Sm from Sm-Co sinter magnets [69].

To summarize, the published work about the extraction of Sm from SmCo permanent magnets is very limited and focuses on using inorganic acids, HCl and H_2SO_4 , as the leaching medium. However, the proposed processes are not selective in the Sm leachability, as impurities, such as Co and Fe, are also dissolved.

5. Conclusions

In conclusion, Rare Earth Elements were defined as Critical Raw Materials according to the European Union because of their rapidly increasing cutting-edge technological sectors and, at the same time, the lack of deposits in the European Union region. REEs have significant physicochemical properties, which have made them capable of inclusion in several high-tech applications, such as electronic devices and electrification. One of their primary applications is the permanent magnets, which contain a high concentration in a wide range of values of REEs, especially Nd and Sm. However, in NdFeB permanent magnets, additional REEs are added to improve their properties. At the same time, SmCo permanent magnets contain a high percentage of Co (50–66%), which protect the magnet from rapid corrosion and, consequently, the possibility of ignition. Thus, REE permanent magnets can be used in plenty of devices based on their properties. Therefore, when these devices reach their End-of-Life cycle, they could be used as a concentrated secondary source. According to the relevant literature, there is no state-of-the-art process for the recycling of REE permanent magnets. Nevertheless, several researchers have focus on the development of recycling methods for End-of-Life permanent magnets through pyro- or hydrometallurgical processes.

This review was performed for the evaluation of NdFeB and SmCo permanent magnets' preprocessing and hydrometallurgical methods for REE extraction with respect to their recovery rate and impurities. Thus, according to the literature, preprocessing is an essential and crucial stage in preparing the material as several factors might inhibit the pretreatment and leaching steps such as ignition of magnets or magnetic interaction with equipment. The most common preprocessing steps were the demagnetization at temperatures above the Curie temperature of the magnets and milling to ensure the material is in a suitable form. The milling of the permanent magnets is challenging due to the rapid corrosion of magnets, especially of NdFeB magnets. The leachability of REEs from NdFeB permanent magnets was studied using either inorganic (HCl, H_2SO_4 , and HNO_3) or organic (acetic and oxalic) acids, achieving high recovery rates. Additionally, other leaching factors, such as time, acidity, and S/L ratio, were evaluated. Based on the published works, the inorganic acids, especially HCl and H_2SO_4 , were more promising because the leaching experiments were conducted under mild conditions at room temperature for a short time, lower than 3 h.

On the other hand, the preprocessing of SmCo magnets and extraction of Sm have not been studied thoroughly. The leachability of Sm has been investigated only with inorganic acids (HCl and H₂SO₄), achieving high Sm recovery at high temperatures (60–95 °C). However, the developed leaching processes of Sm were not selective, as impurities, such as Co, are dissolved in high percentages. Therefore, high recovery yields have been reported for both Nd and Sm, but impurities and traces should also be considered in subsequent studies.

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