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“Ways forward to promote
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Recovery of Rare Earth Elements from NdFeB Waste Magnets

DURSKI, M ^{a,*}, NAIDOO, P ^b, MANILAL, N. ^a, MOODLEY, K ^a

a. University of KwaZulu-Natal, Durban

b. Stellenbosch University, Stellenbosch

**Corresponding author, durskim@ukzn.ac.za*

Abstract

The increasing volumes of waste electrical and electronic equipment (WEEE), reported in developed countries, will soon be a major concern facing local municipalities. This waste contains a critical raw material group of rare earth elements (REEs), among other components. With the surge in electrical and electronic equipment use in recent years, responsible management of these associated solid wastes is developing. However, it is yet to be addressed practically in South Africa.

Hydrometallurgical processing by acid dissolution of waste permanent magnet (WPM) powders is used to recover REEs and their compounds from neodymium-iron-boron (NdFeB) magnets. These magnets were removed from discarded hard disk drives. The chemical treatment processes are predominantly based on inorganic acids such as hydrochloric, nitric, and sulphuric acids, which offer a good transition of the metals into salts dissolvable in the aqueous solutions. The first stage of operation in the extraction of REEs from waste (NdFeB) magnets includes the physical processes of demagnetization, crushing, and grinding to obtain WPM powders. In contrast, the chemical process is initiated by leaching the WPM powder with an acid. Then leachate is contacted with a selective organic extractant to recover demanded ions from the aqueous solution. The last step of the processing is precipitation, where ions create insoluble metalorganic complexes or REEs, which can be used in further manufacturing processes.

Experiments were performed with nitric acid to recover neodymium and iron from crushed NdFeB magnets. The results indicate that the best leaching conditions were obtained for experiments conducted using 100-150µm powder digested with 12.3M HNO₃ at 60°C for 24 hours. HDEHP of 1M in n-dodecane showed the highest transfer of neodymium ions to the organic phase. The precipitation process utilizing the saturated aqueous oxalic acid solution showed a significant recovery of the Nd³⁺ ions from the organic phase. Recoveries above 95% were obtained for precipitant-to-extractant ratios 5:1 and 10:1.

Keywords: recycling, neodymium magnets, rare earth metals

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1. Introduction

Neodymium-iron-boron (NdFeB) magnets are mainly composed of iron, neodymium, and boron with additives in the form of other rare earth elements (REEs) such as praseodymium (Pr) and dysprosium (Dy) or other metals, i.e., cobalt (Co). The final composition of the permanent magnet is defined by the future application (Tunsu, 2018). To prevent corrosion of the permanent magnets, common additives are nickel, used for coating the manufactured product or adding 1-2% of cobalt to the alloy (Gruber et al., 2020; Lee et al., 2017). The Curie temperature is an important parameter of the permanent magnets which is a critical temperature of transition between ferromagnetic and paramagnetic properties of the material (Fabian et al., 2013). According to Sarfo (2019), market demand for neodymium, dysprosium, yttrium, europium, and terbium is nearing the quantities obtained from geological deposits. Thus, the collection and recycling of NdFeB magnets and other WEEE are critical to meet the sustainable growth goals of this industry sector (Chu, 2010).

Some key applications of neodymium magnets include wind turbines, electric cars, hard disk drives (HDDs) and military equipment (Xie et al., 2014). One of the promising sources of waste magnets that can be used in the recycling process is hard disk drives (HDDs). Each HDD contains between 1 and 30 g of neodymium magnets with an Nd and Dy content of approximately 31% and 4-7%, respectively (Erust et al., 2021).

The implementation of commercial processes for waste electrical and electronic equipment recycling, especially REEs recycling, is limited. (Binnemans et al., 2021). Commercially available processes mostly focus on recovering scrap magnets from the waste electrical and electronic equipment (WEEE) and repurposing them using the magnet-to-magnet (M2M) approach.

Recycling NdFeB magnets is necessary to increase the available amounts of REEs, which can delay the depletion of the known deposits (Reisdörfer et al., 2019) and address unsustainable mining practices. The three possible approaches to REE recycling include magnet-to-magnet (M2M), pyrometallurgical and hydrometallurgical methods. The magnet-to-magnet approach refers to the production of new magnets from spent magnets. The waste magnets are refurbished and prepared for sintering by adding approximately 3% fresh permanent magnet alloy. Greater than 90% of REEs are recovered with less than 5% freshly mined rare earth metals (Jin et al., 2016). The most important disadvantage of this approach is the decrease in the final product quality, mostly due to the inconsistency in the composition of the final product. Several applications report using these magnets in electrical engines, wind turbine generators or magnetic separators (Nlebedim et al., 2018). The pyrometallurgical approach primarily processes freshly mined ores with high concentrations of REEs (Tunsu, 2018). The main disadvantages of this approach are high energy consumption used to reach the required temperatures, low selectivity of the process, and high levels of pollution in the form of emissions of gas and solid particles into the air (Tunsu, 2018).

Hydrometallurgy is one approach to recovering rare earth elements from WEEE (Gruber et al., 2020), with leaching being a well-established technique in converting metals into ions of soluble salts via chemical reactions (Behera et al., 2016; Lee et al., 2013). REEs react with sulfuric acid, nitric acid, and hydrochloric acid to form hydrogen gas and cations in salts containing sulfate, nitrate, and chloride anions, respectively. The low energy consumption, reduced gas emissions and adaptability to varied materials by adjustment of the reagents to achieve lower environmental impact (Reisdörfer et al., 2019). However, the amount of acidic waste generated in liquid form poses a significant drawback to this approach (Gruber et al., 2020). NdFeB magnets are

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demagnetized and comminuted to small diameter particles in hydrometallurgical processes, preferably in leaching processes. Then waste permanent magnet (WPM) powder is digested with acid to obtain mixtures containing ions of metals present in the alloy used to manufacture magnets. This approach is based on the use of inorganic acids such as hydrochloric, nitric, and sulfuric acid, which, according to the literature, allow for high leaching recoveries (Erust et al., 2021; Lee et al., 2013; Ni'am et al., 2019). Leaching is affected by WPM powder modification, type of acid, particle size, concentration of acid, solid-to-liquid ratio, stirring speed, the temperature of the solution, and contact time with leachate (Ni'am et al., 2019). Lee et al. (2013) conducted batch experiments to establish optimal leaching conditions for Nd recovery from permanent magnets. The authors concluded from the large list of parameters investigated that for WPM powder of particle size less than 297 μm , optimal leaching conditions were obtained using 3M H₂SO₄ and HCl over 15 minutes using a solid/liquid ratio of 0.02. Ni'am et al. (2019) reported that optimal WPM leaching conditions were obtained by utilizing 5 M HCl at a temperature of 368 K for 24 h using 0.02 solid-liquid ratio, 800 rpm stirring speed, and 0.250 mm particle size of the WPM powder.

Reisdörfer et al. (2019) characterized roasted and unroasted NdFeB magnet powders sourced from HDDs. Roasting is performed at high temperatures to convert the elements in the sample into oxides. This enhances the selectivity of Nd in the leaching procedure. Unroasted NdFeB powders contained 32.36% of Nd and 58.5% of Fe. Lee et al. (2013) reported similar values, with 31.27% of Nd, 59.62% of Fe, and 1.26% of B. It must be noted that the Nd percentages of 25.19% and 32.36%, with and without roasting, respectively, were considerably higher than the amounts typically found in mineral ores. Nd extraction from bastnasite is approximately 18.5 wt%, while percentages in the 15-20% range are extracted from monazite.

Leaching, extraction and precipitation experiments were conducted in this study using HNO₃, bis(2-ethylhexyl) phosphate (HDEHP), oxalic acid and WPM powder from HDDs. The effect of particle size, nitric acid concentration, temperature and contact time were reported for the leaching experiments. The concentration of HDEHP and the time of the extraction process are reported for the extraction experiments. Oxalic acid was utilized as a precipitant to selectively recover REEs from the organic phases collected during the extraction measurements. The sample analyses were performed using Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES) to determine concentrations of ions in samples.

2. Methodology

The experimental work consisted of four processes: pretreatment of the waste magnet powder leaching, extraction and precipitation. Table 1 presents a list of the chemicals used in this work. Waste magnet powders were obtained by dismantling hard disk drives (HDDs) supplied by the University of KwaZulu-Natal.

2.1 Pretreatment of waste magnet powders

HDDs were dismantled manually to remove the NdFeB magnets by removing the aluminum casings. NdFeB magnets of varying sizes and compositions were isolated and stored for subsequent waste processing. All other components were dispatched for waste treatment. To remove the magnetic properties of the magnets, these were heated above Curie temperature in a Scientific 909 model laboratory furnace at 400°C. Once the temperature of the furnace stabilized

Table 1. List of chemicals used during this study.

Chemical compound	CAS Number	Molecular Mass, g/mol	Supplier	Purity
Bis(2-ethylhexyl)	298-07-7	322.4	DLD	0.999
n-Dodecane	11-40-3	170.33	Sigma Aldrich	0.995
Nitric acid	7697-37-2	63.01	Honeywell	0.650
Sodium hydroxide	1310-73-2	39.99	Sigma Aldrich	0.98
Phenolphthalein	77-09-8	318.32	Sigma Aldrich	0.9995

at the set point, the magnets placed in a ceramic crucible were carefully inserted into the furnace and heated for 30 minutes, left to cool for 30 minutes. The magnets were then comminuted using a roller mill. Several repetitions were performed until magnet powder fines were obtained. The magnet fines were screened using a series of laboratory test sieves of varying apertures (100 μm , 150 μm , 200 μm , 300 μm , 355 μm , 425 μm , 500 μm , and 600 μm). This allowed the particle size distribution after comminution to be determined with the agitation of the sieve trays set for 30 minutes by an electrical shaker. The final masses of each tray were then weighed using a mass balance, and the contents of each tray were stored in labelled airtight bags for further waste processing.

2.2 Leaching

All leaching experiments were performed on a batch scale, with a solid-to-liquid ratio (S:L) of 1:50 being utilized, as Erust et al. (2019) recommended. Magnetic stirrer plates with integrated heating elements were employed. Borosilicate glass beakers were filled with nitric acid solution (6.7M and 12.3M). Each beaker was placed on a heated magnetic stirrer plate, with a stirrer rate set at 333 rpm. After the temperature had stabilized at the desired setpoint (45°C or 60°C), magnet powder (100-150 μm and $\geq 600 \mu\text{m}$ grain size) was weighed and added to each beaker. After the leaching period had elapsed, the run was terminated, and samples were collected immediately. A 45 μm micro-filter plunger syringe was used to draw out a 1ml sample, diluted with a factor of 100 to prepare for ICP-OES analysis. Sampling was performed in triplicate, and each experiment was conducted in triplicate. The remaining solution was filtered and stored in an airtight container for further waste processing.

2.3 Liquid-liquid equilibria

Two sets of experiments were performed to measure the distribution of the metals between aqueous and organic phases. In the first setup, 10 ml glass cells were used. In the second setup, a 100 ml extraction cell was utilized. This approach was used to determine the scalability of the process. The water bath temperature was kept at 25°C throughout the experiments. The leached solutions from the previous experiments were utilized as the aqueous phase. The organic phase (1M and 0.5M HDEHP in n-dodecane) was prepared by combining predetermined volumes of HDEHP and n-dodecane. The glass cells were each loaded with the aqueous and organic phases in a 1:1 volume ratio. The contents of the cells were stirred for 12h. After agitation, the mixtures within the glass cells were left to separate for 12h in isothermal conditions to prevent any equilibrium disturbance. Approximately 5 ml of the aqueous phase was withdrawn from each cell without disturbing the equilibrium. The withdrawn samples of the aqueous phase were diluted with a factor of 100 for the ICP-OES analysis. The organic phase concentrations of REE were

determined following the ICP-OES analysis and were calculated based on the mass conservation law. The REE concentrations of the aqueous feed prior to extraction and the equilibrium concentration of the REE of the aqueous phase in the cell after extraction and settling were used to perform mass balance calculations.

2.4 Precipitation

Various acid concentrations and organic: acid (O:A) ratios were used to precipitate REEs. A 100mL was used during this process. Calculations were performed using mass balance. The organic phase was drawn to the vial. The desired volume of a saturated mixture of oxalic acid was added and contents of the vial were stirred vigorously for 10 minutes. The vial was left at ambient temperature to settle for 48 hours to allow precipitation. The contents of the vials were filtered, washed, and dried. Solids were then dissolved in concentrated sulphuric acid and diluted with a factor of 100 for ICP-OES analysis.

2.5 Safety

Each experiment was conducted using personal safety equipment. The remaining acidic solutions were collected and stored in properly marked containers under the fume cupboard for the chemical recycling company to neutralize. The remaining HDEHP was also collected and stored for future experiments regarding multistage extraction and recycling of the extractant.

3. Results and discussion

3.1 Demagnetization

The high magnetism of NdFeB magnets causes strong coercive forces, making them nearly impossible to separate. It is advised to have the proper padding or material on hand for optimal storage. Demagnetization made handling the NdFeB magnets and their subsequent comminution stage more convenient. Lee et al. (2013) reported that total demagnetization occurs at 350°C after 15 minutes. In contrast, Reisdörfer et al. (2019) used a temperature and duration of 350°C and 30 minutes, respectively. As a result, 400°C and 30 minutes were used to ensure complete demagnetization.

3.2 Comminution

The size distribution of crushed magnets is presented in Table 2. The mass fraction of crushed magnets consistently decreased with decreasing sieve sizes. This indicates that the comminution process should be improved in future work. In practical operations, further crushing would demand increased equipment to screen samples, thereby increasing the capital costs of the process and the workforce required. The desired particle sizes were 100 – 150 µm and 600 µm. A particle size range of 600 µm was used to determine if additional crushing or grinding is required. In practical operations, further crushing would demand increased equipment to screen down samples, thereby increasing the capital costs of the process and the workforce required. Additionally, an efficient crushing procedure is necessary to fast-track the leaching process. Behera et al. (2016) and Sahin et al. (2017) reported that optimal particle sizes were 46-75 µm and 63-90 µm, respectively. The mass distribution of the particles of different sizes is shown in Table 2. The standard uncertainties for mass measurements were 0.03 g.

Table 2. Sieve analysis of the waste permanent magnet powder after comminution.

Sieve Size (μm)	Mass retained (g)	Mass fraction	Cumulative Mass Passes (g)	Total Percent Passed (%)
600	339.3	0.554	273.0	44.59
500	40.88	0.067	232.2	37.91
425	37.24	0.061	195.0	31.83
355	32.69	0.053	162.2	26.49
300	30.39	0.050	131.8	21.53
200	57.50	0.094	74.34	12.14
150	28.79	0.047	45.55	7.439
100	33.86	0.055	11.69	1.910
Pan	11.69	0.019	0	0
Total	612.3	1	612.3	100

3.3 Leaching

The parameters investigated in the experiments were the leaching period, the concentration of acid, particle size of magnetic fines and temperature. The contact time between acid and WPM powder is crucial in leaching. Four different leaching periods were tested to investigate the effect of leaching time on leaching efficiency, namely 30, 60, 240 and 1440 min. The results are presented in Table 3, which shows that the leaching period significantly affected leaching performance based on the variation of metal ion concentrations. The concentration of both Fe^{3+} and Nd^{3+} ions was observed to have increased logarithmically as the leaching period was increased. It is noted that the reaction rate was visually observed to have been higher upon introducing the magnet fines to the acid solution. This results in the leaching rate being significantly higher for the shorter leaching periods and reduced for the increased leaching periods. Based on the results, the 24-hour leaching period consistently produced the best and optimal leaching performance.

The size of the particle being subjected to leaching also affects the leaching performance. The concentration of both Nd and Fe ions was higher for the 100–150 micron particle size than the ≥ 600 -micron particle size. This was observed across all leaching periods. This result is due to the larger contact surface area facilitating the leaching reaction for smaller particles. Due to the abovementioned, the 100–150-micron particle size was deemed optimal for the process. The concentration of acid used for leaching directly impacted the leaching performance. Two different concentrations of nitric acid were utilized to investigate the effect of concentration on leaching performance, namely 6.7M and 12.3M. The concentration of both Fe and Nd ions was higher when utilizing 12.3M nitric acid as a leaching agent, compared to 6.7M nitric acid. This was observed for all leaching conditions. Due to this result, 12.3M nitric acid was deemed optimal. The temperature of the leaching reaction directly impacts the process performance. Two temperatures were utilized in the leaching experiments to investigate the effect of temperature, namely 45°C and 60°C. The concentration of both Nd and Fe ions was higher for the 60°C leaching experiments than the 45°C leaching experiments. This was evident for all leaching conditions. It was therefore concluded that 60°C is the optimal temperature for leaching REEs. This result was due to the higher temperature providing access activation energy to facilitate the chemical reaction.

Table 3. Concentrations (ppm) of Nd³⁺ and Fe³⁺ ions obtained after leaching at a given nitric acid concentration (M), temperature (°C), time (h) and particle size (µm).

[HNO ₃]/M	T/°C	Time/h	Particle size			
			100-150 µm		≥600 µm	
			[Nd ³⁺]/ppm	[Fe ³⁺]/ppm	[Nd ³⁺]/ppm	[Fe ³⁺]/ppm
6.7	45	0.5	13400	17815	6950	14533
		1	14100	19483	7852	15573
		6	15200	21850	8765	16578
		24	15800	23117	11408	22106
	60	0.5	14277	19225	8165	16147
		1	14792	21817	9025	19883
		6	16268	24183	10023	20800
		24	17172	26450	14257	23217
12.3	45	0.5	15012	21397	9888	16990
		1	16192	24117	10983	20817
		6	17210	30729	12183	22017
		24	18003	37367	16767	24783
	60	0.5	15758	26950	12763	21133
		1	16738	29417	13002	23933
		6	18044	34333	14462	24450
		24	18763	39233	19133	26450

3.4 Liquid-liquid equilibria

The extraction measurements involved an organic phase of HDEHP in an n-dodecane solution, and an aqueous phase of REEs leached in a nitric acid solution. The concentrations of the aqueous and organic phases were varied to investigate their effect on extraction performance. Two aqueous phase concentrations were deployed, 0.5M and 1M of HDEHP in n-dodecane. The REE concentrations in the organic phase varied based on the leaching conditions that they were subjected to. The extraction measurements were performed on a laboratory vial scale (10 mL) and a pilot-plant scale (100 mL). This was performed to investigate the scalability of the REE extraction method proposed.

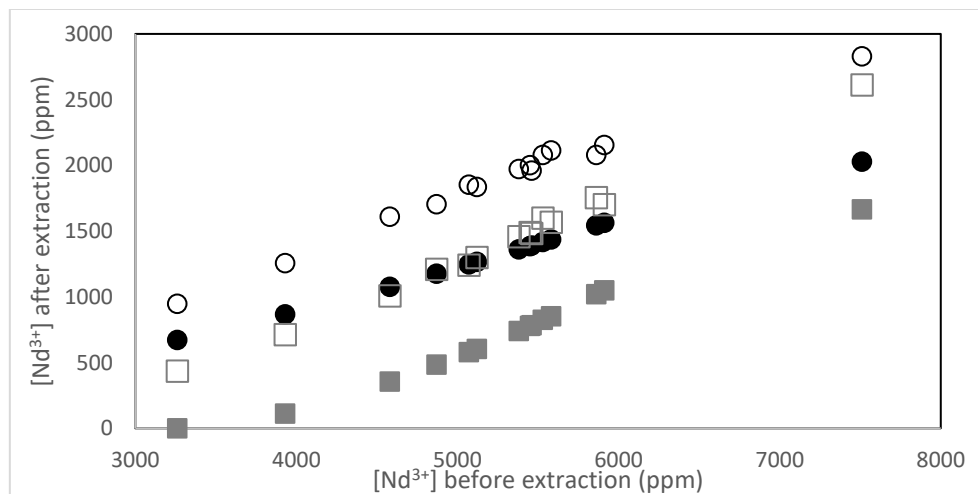


Figure 1: Nd³⁺ ions concentration before extraction vs Nd³⁺ ions concentration after extraction experiments; ● – 0.5M HDEHP in n-dodecane in 10 ml cell, ■ – 1M HDEHP in n-dodecane in 10 ml cell, ○ – 0.5M HDEHP in n-dodecane in 100 ml cell, □ – 1M HDEHP in n-dodecane in 100 ml cell

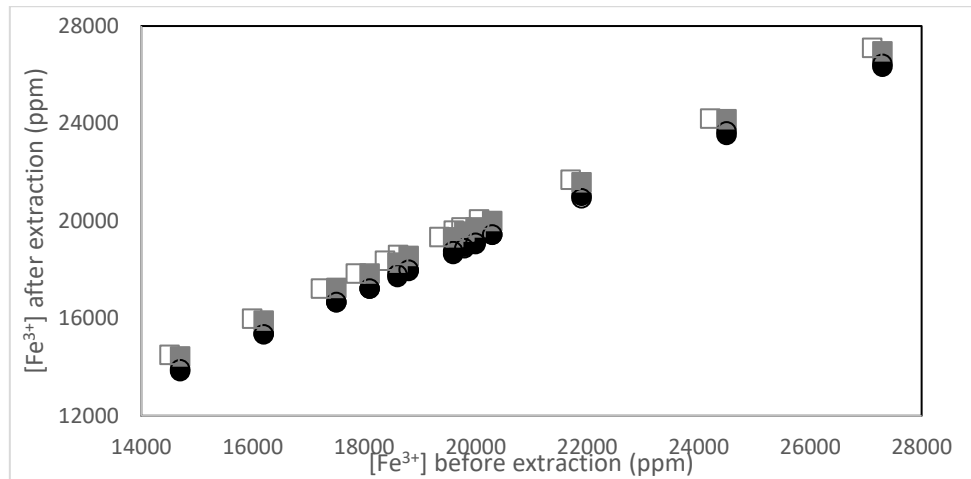


Figure 2: Fe^{3+} ions concentration before extraction vs Fe^{3+} ions concentration after extraction experiments; ● – 0.5M HDEHP in n-dodecane in 10 ml cell, ■ – 1M HDEHP in n-dodecane in 10 ml cell, ○ – 0.5M HDEHP in n-dodecane in 100 ml cell, □ – 1M HDEHP in n-dodecane in 100 ml cell

Figs. 1 and 2 show that the concentration of Nd^{3+} in the aqueous phase had significantly decreased, signifying that the Nd^{3+} ions were transferred into the organic phase. A 1M HDEHP solution was significantly more efficient during experiments in both scales. However, the results of small-scale experiments show a higher efficiency of the extraction in comparison with the larger-scale results. This might be caused by the inefficient stirring in the larger equilibrium cell. For Fe^{3+} ions, however, the decrease in concentration was barely noticeable throughout the experiments. This is due to the nature of the organic phase deployed, being selective towards REE ions.

3.5 Precipitation

Precipitation measurements were performed using conditions reported by (Binnemans et al., 2013). Table 4 presents the precipitant compositions and overall neodymium recovery. The volume ratio of saturated oxalic acid to the organic phase was crucial for the recovery of the neodymium in the precipitation process. The 1:1 volume ratio shows the lowest recovery of the Nd^{3+} ions, with approximately 71% recovery. Ratios of 5:1 and 10:1 show the best recovery rates exceeding 95% and 97% of recovery, respectively.

Table 4. Concentrations (ppm) of Nd^{3+} and Fe^{3+} ions obtained before and after precipitation processes at a given HDEHP concentration (M) and various oxalic acid (OA) to organic phase (OP) volume ratios.

[HDEHP] /M	AO:OP ratio	[Nd^{3+}] in the extract	[Nd^{3+}] in solids	[Fe^{3+}] in the extract	[Fe^{3+}] in solids	Recovery of Nd^{3+} /%
0.5	1:1	3499	2492	917.2	566.3	71.18 ± 0.10
0.5	2:1	3264	2965	839.8	681.4	90.86 ± 0.23
0.5	5:1	3114	2975	830.0	707.3	95.54 ± 0.12
0.5	10:1	3703	3569	815.0	696.4	96.37 ± 0.13
1	1:1	3971	2796	211.2	140.7	70.43 ± 0.11
1	2:1	3801	3443	241.8	203.2	90.59 ± 0.34
1	5:1	3625	3457	205.5	178.9	95.39 ± 0.22
1	10:1	4107	3993	223.3	203.5	97.22 ± 0.09

3.6 Limitations of this study

Experiments performed on a small scale (10ml) and larger scale (100ml) showed that the process can be upscaled. A pilot-scale operation is recommended for further tests to assess the scaleup potential. Regarding the process feasibility, additional studies must be undertaken to choose the optimal precipitant-to-extractant ratio. Waste management strategies must be implemented for a sustainable approach.

4. Conclusions and recommendations

A hydrometallurgical process involving leaching using extraction and precipitation to recover neodymium from waste permanent magnet powders successfully yielded recoveries greater than 95% neodymium. The set of optimal conditions for each subprocess was established. Optimal leaching conditions were obtained using 100-150 μ m powder digested with 12.3M HNO₃ at 60°C for 24 hours. HDEHP was proven a good extractant due to its selectivity towards neodymium ions.

The precipitation process with saturated oxalic acid solution showed a significant recovery of the Nd³⁺ ions from the organic phase, with recoveries of above 95% for the precipitant-to-extractant ratios above 5:1. Oxalic acid salts can be relatively easily thermally decomposed to the REE oxides, which can then be used in the manufacturing process of new magnets.

The trends shown by the data for the 10 and 100 ml cells are similar, indicating that upscaling of the process is possible. The insufficient mixing in the large cell can explain the discrepancy between both data sets.

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