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ARTICLE

Rare Earth Recovery from End-of-Life Motors employing Green Chemistry Design Principles

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This manuscript describes the development of an efficient process for the recovery of rare earth elements from materials mixtures such as in motors with a recovery rate of >80%. While heat treatment is required for processing, all other steps can be performed at room temperature, thus resulting in a process designed for energy efficiency. Selective dissolution enables efficient separation of steel and copper by taking advantage of the different reduction potentials of the materials in the mixture, while selective precipitation of RE salts is the key for obtaining pure RE products. Overall, the established process applies green chemistry principles for designing a hydrometallurgical process.

Introduction

Strong price fluctuations for Rare Earth (RE) materials during 2011 caused by Chinese export restrictions[1] have initiated a review of supply strategies for these critical materials in the Western hemisphere.[2,3] Due to the concentration of mining, separation, and manufacturing of RE materials in China,[4] the future risk of supply chain disruptions remains significant. Strategies to RE independence in other countries have focused on mining, stockpiling, and substitution of REs; however, recycling as a complementary approach has also been widely considered and recently been shown to enable price stabilization of critical materials.[5]

Several rare-earth containing end-of-life products such as energy-efficient light bulbs[6] and HD drives[7] are already being recycled successfully. In contrast, end-of-life motors containing RE magnets – e.g. from electric or hybrid vehicles – are currently not recycled. One issue with these materials is that the magnets are typically contained in the innermost part of the motors and thus not easily accessible. Another challenge for recycling these materials is caused by the strong magnetism which makes them desirable for manufacturing motors: As common recycling equipment such as shredders contain substantial amounts of ferromagnetic material, treating magnets with standard technologies remains difficult. Previous efforts to recycle RE magnets have so far only focused on the magnet alloys[8] without considering the materials mixture that is present in end-of-life products such as motors. In order to address these issues and to develop a methodology to selectively isolate REs from end-of-life motors without requiring previous manual disassembly, we describe herein an efficient recycling process based on green chemistry principles: waste prevention, less hazardous processes, design for efficiency, use of renewable feedstocks, selectivity, and inherently safer chemistry for accident prevention. This process, as detailed below, has been established by using mechanical and thermal processing prior to chemical processing, through closely monitored, selective dissolution of the desired materials, the use of a renewable precipitation reagent (oxalic acid), and by executing the hydrometallurgical steps at room temperature, resulting in high recovery efficiencies up to 82% and excellent product purity (>99%).

Methodology

Process development was guided by two general design parameters: (i) with existing technologies and (ii) the principles of green chemistry. Incorporation of these parameters at the design stage is expected to result in a commercially viable technology which can be implemented without the need to develop specialized equipment. Thus, all hydrometallurgical steps are executed at room temperature, while selective leaching of the desirable materials (RE magnets), simple filtrations, and selective precipitation all aim to ensure product purity and simple execution. All potential by-products of the initially hypothesized process are expected to be valuable input materials for other processes such as scrap sortation (steel and copper scrap)[9] and pyrohydrolysis[10,11] for the regeneration of HCl from the leaching solution. Diluted acid instead of concentrated acids were preferably used for the selective dissolution step and the use of solid acids as precipitation reagent was explored; both of these approaches generate inherently safer chemical processes.

Based on all these requirements for a sustainable and commercially viable recycling process, we proposed the process flowsheet shown in Figure 1. The first process steps

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consist of mechanical pre-processing through heat treatment and shredding, as this treatment will (i) simplify liberation of magnets from motors as well as further processing and (ii) allow processing bonded as well as coated magnets in the dissolution step. We then postulated that NdFeB magnets can be selectively dissolved upon addition of an acid A in the presence of both steel and copper; this hypothesis was formulated based on the low reduction potential of Nd (E° = -2.323 V),[12] which can be expected to affect the reduction potential of the NdFeB alloy and result in instability to acids.

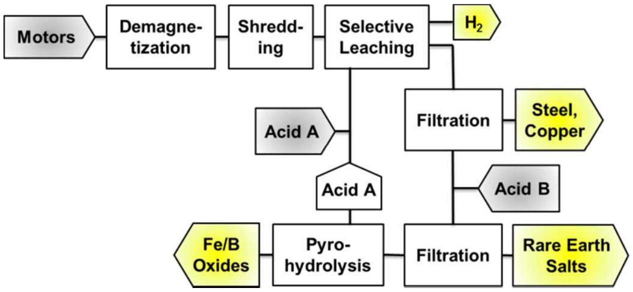

Figure 1. Proposed Closed-Loop Process for Recovery of REs from End-of-Life Motors. Grey arrows depict process input materials; yellow arrows depict process products.

After selective dissolution of the magnet alloy, we propose a filtration step to remove remaining metal scrap (steel, copper) from the mixture, resulting in a filtrate containing the REs. Selective precipitation of RE salts by addition of an acid B in the presence of other metals (e.g. Fe) would then be followed by isolating the precipitate by filtration. The filtrate from this last step is expected to be a good input stream for pyrohydrolysis used in steel pickling,[10] resulting in regeneration of acid A and production of mixed oxides, which in turn can be used for iron production in blast furnaces.[11]

Results and Discussion

Leaching of NdFeB Magnets.

Our initial investigations for realizing the proposed process depicted in Figure 1 focused on identifying a suitable acid to achieve complete oxidative dissolution of NdFeB magnets, which at the same time would not react substantially with steel and copper. For this study, we purchased non-bonded, non-coated NdFeB magnets and documented their dissolution in different acidic solutions. Figure 2A demonstrates that employing acetic acid, formic acid, oxalic acid, and lactic acid does not result in complete dissolution of the magnet material after 24 h; in contrast, mineral acids (HCl, H_2SO_4) completely dissolve the magnet after 24 h. In a next step (Figure 2B), different molarities of HCl and H_2SO_4 were used for this experiment; the dissolved mass after 24 h was then plotted against the acid concentrations. Interestingly, all HCl solutions that were 4 M or more concentrated completely dissolved the magnetic material after 24 h; H_2SO_4 solutions also dissolved all the material at an acid concentration of 2 M (= 4 M H^+ concentration), but some higher concentrations lead to incomplete dissolution. This is likely due to the formation of a precipitate on the surface of the magnet material, which might act as a passivating layer.

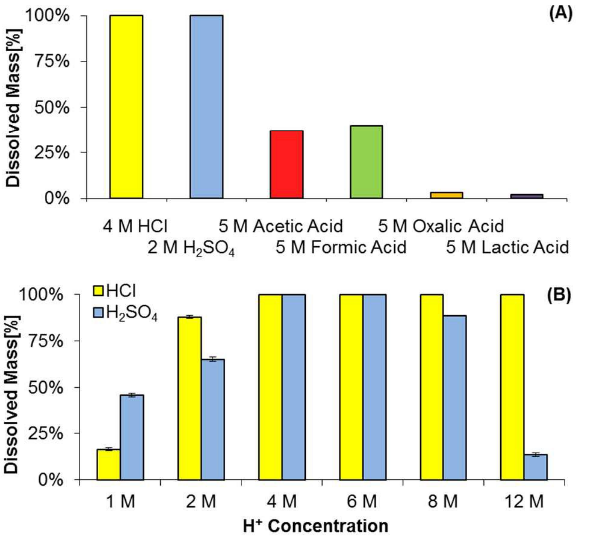

Figure 2. Dissolution of NdFeB Magnets. (A) Dissolution in different acids. (B) Dissolution in HCl (yellow) and H_2SO_4 (blue) at different acid concentrations. Conditions: 24 h, room temperature.

Leaching Selectivity.

Having established conditions for dissolving the NdFeB alloy, we then aimed to identify conditions which would allow the selective leaching of this alloy. Ideal leaching conditions would not only dissolve RE magnets, but also show no reactivity with other materials common in motors. This approach is important in order to minimize the amount of impurities leached from a heterogeneous mixture of after shredding in order to result in high purity and yield of RE precipitate in the following process step.

To this end, the most effective leaching acids (HCl, H_2SO_4) were first investigated for their capability to dissolve a steel washer (made of case hardened black steel) at different initial acid concentrations (Figure 3A). Interestingly, significant mass loss by dissolution (>10%) was detected in all H_2SO_4 leaching solutions, while less than 10% mass loss was observed in 4 M or less concentrated HCl. Heating the leaching solutions to 50 or 80 °C did not improve the leaching selectivity (for details, see the SI). Based on these data in combination with the magnet dissolution study presented in the last section, 4 M HCl was chosen as the ideal concentration for selective dissolution of NdFeB magnets, as this acid solution was highly reactive towards the NdFeB alloy, but much less reactive towards the simple steel alloy used for the dissolution study. Figure 3B depicts the kinetics of dissolution for these conditions for a non-bonded NdFeB magnet, the widely used steel alloys 1018 and 304, and copper (another common component of motors) at room temperature; strikingly, only the magnet material dissolves under these conditions, suggesting that the desired selective leaching of magnets is possible.‡

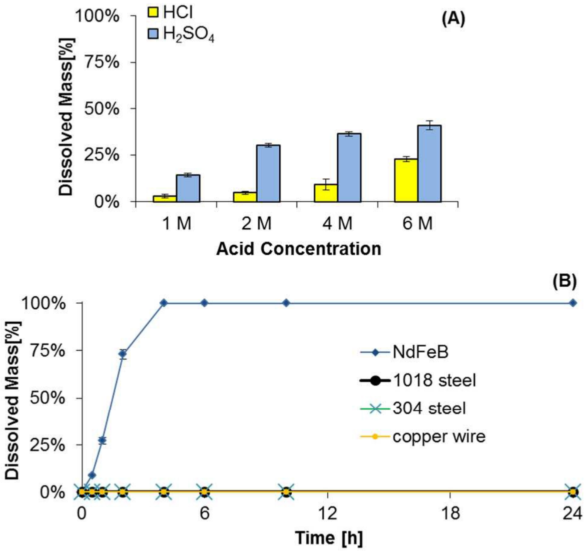

Figure 3. Dissolution of Steel, Copper, and NdFeB Magnets. (A) Dissolution of case hardened black steel in HCl and H_2SO_4 (1 to 6 M) after 24 h at RT. (B) 1018/304 Steel, Copper, and NdFeB Magnet Dissolution Kinetics in 4 M HCl.

Selective Precipitation of RE Salts.

With the resulting leaching solution in hand, we systematically explored the selective precipitation of REs through addition of a second acid as precipitation reagent. This approach was considered desirable, since it reforms *in situ* the acid (HCl) used for leaching; thus, this approach enables acid recovery from the process. We tested two reagents that have been documented to precipitate mixed RE salts in the literature: sulfuric acid to form RE sulfates of the general formula $(RE)_2(SO_4)_3$ (RE = Nd, Dy, Pr) and oxalic acid to synthesize RE oxalates $(RE)_2(C_2O_4)_3$.

As documented in the literature,[13] the precipitation of sulfates required adjustment of the pH of the solution from pH -0.3 to pH 0.6 with NaOH solution. This method was considered not ideal for several reasons: (i) The resulting acid-base reaction released a significant amount of heat in our experiments, resulting in boiling of the solution; thus, on larger scales, external cooling would be required which would in turn decrease the energy-efficiency of the process. (ii) The sulfate precipitation approach introduces an additional chemical (NaOH) into the system and results thus in a less sustainable process, also because quantitative recovery of HCl after addition of NaOH is not possible. (iii) An additional drawback was the very low yield of obtained precipitate: <10% of REs precipitated as $(RE)_2(SO_4)_3$.

In contrast, the alternative oxalate route showed none of these drawbacks: An oxalate precipitate was obtained from the leaching solution by simple addition of oxalic acid, resulting in >99% yield of $(RE)_2(C_2O_4)_3$ after optimization (purity >99% by ICP-OES of redissolved precipitate; see SI for details). An additional benefit of this process is that oxalic acid can be used as a solid reagent, thus enhancing the safety of the process. Furthermore, no heat development was observed with this approach, suggesting that this precipitation process is inherently safer and more efficient than the alternative sulfate route.

Kinetics of Dissolution for Bonded and Non-bonded NdFeB Magnets.

After developing the basic hydrometallurgical steps of the proposed process, we focused on enhancing the scope of the employable magnets. As NdFeB magnets are brittle and can easily be attacked by chemicals, magnet manufacturers have found several ways to improve their stability. One of the common strategies is to mix the NdFeB materials with a resin to produce so-called bonded magnets. Following the kinetics of dissolution of bonded magnets (Figure 4) shows that the presence of the resin indeed causes a delay in leaching compared to non-bonded magnets; however, all magnetic material was consumed after stirring in 4 M HCl for 24 h, thus suggesting that a longer leaching time would enable complete recovery of REs even from these less corrosion-resistant materials under selective leaching conditions.

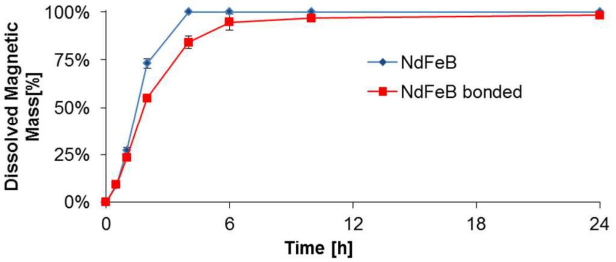

Figure 4. Dissolution Kinetics of Non-bonded and Bonded NdFeB Magnets in 4 M HCl.

Demagnetization and Shredding of NdFeB Magnets.

Another common strategy to improve the chemical resistance of NdFeB magnets relies on coating the surface of the magnet with Ni. Since Ni has a much higher reduction potential (Eº = -0.236 V) than Nd (Eº = -2.323 V),[12] we do not expect that the above developed conditions are able to dissolve coated magnets readily. However, we hypothesized that leaching can be expected to proceed once the inside of coated magnets is exposed. Thus, we decided to investigate how to shred magnets using a hammermill, as fragmentation of magnets can be expected during this step before leaching takes place. Initially, we employed non-bonded magnets (18.6 g) as model materials. Interestingly, only 26% of material input was recovered from the hammermill, even after manual removal of all visible magnet material. This suggests that demagnetization is a required step before shredding can occur, since the attractive forces between the ferrous materials constituting the hammermill shredder and the magnets would remove materials from the process stream. In contrast, when repeating this experiment with magnets (18.6 g) that had been previously demagnetized by heating to 450 °C for 1 h, [§14] 88% of the original material was recovered. However, another issue occurred during the test: the magnet materials caught fire, as can be expected based on the pyrophoric properties of fine NdFeB powders.[15]. In order to avoid specialized equipment such as shredding under protective gas atmospheres, we investigated if a larger shredder screen would address the issue. This hypothesis was formulated based on the fact that the reaction rate of magnet materials should be proportional to the exposed surface of the alloy, which will rely on the quantity of shredding events. A larger screen thus would reduce the shredding events and reduce the fire hazard. To our delight, adjusting the shredder configuration and using a shredder screen with larger openings (diameter 19 mm) prevented the production of smoldering magnets. This is likely due to the formation of larger scrap and thus less surface area of the resulting particles. In summary, the studies described in this section suggest that both demagnetization and shredding of RE motors are beneficial to establishing a generally applicable, high-yielding process for the recovery of REs from motors.

Recovery of REs from Small Motors.

Having investigated and established each individual step of the proposed process independently, our next goal was to combine these steps in order to establish a complete process using motors as feedstock. To this end, we first focused on recovering the RE content of small motors, which contain Nd magnets, since we hypothesized that potential issues in a system with many materials being present could be more easily detected and addressed on a small scale. We obtained several identical small motors (Figure 5A) from online vendors;[§§] Figure 5B shows one of these motors after dismantling, which allowed us to determine an accurate materials inventory (composition of RE magnets, weight of steel and copper; see SI) that would later serve to calculate the recovery rates.

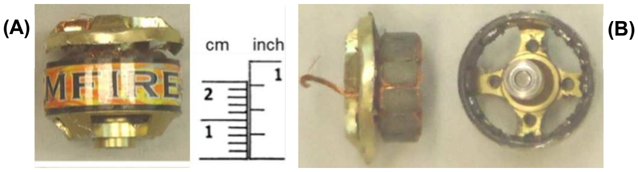

Figure 5. (A) Small Motors containing RE magnets before dismantling; (B) Motor core containing RE magnets and single RE magnets after manual dismantling.

Demagnetization and shredding were performed as detailed above (450 °C, 1 h; Schutte Buffalo Hammer Mill, Laboratory Scale, Model-6-H, screen with holes of 6 or 19 mm diameter), resulting in a mixture of materials which was treated with 4 M hydrochloric acid. As described above, the smaller screen size did not provide satisfactory recovery of magnetic material and resulted in some smoldering of the material in air, while use of the 19 mm screen provided minimal crushing, no smoldering, and disassembly of the small motor to suitable scrap sizes (Figure 6). When analyzing the obtained solution after filtering non-dissolved materials off, Fe, Nd, Dy, Pr and B concentrations were high as expected based on the selective dissolution of RE magnets, while the concentrations of other elements (Cu, Zn, Co, Ni, Mn) were all less than 1%. However, when attempting to precipitate the REs by oxalic acid addition to the leaching solution, only very little precipitate was obtained. Comparing the pH of the leaching solution (pH 1.39) before addition of oxalic acid with the pH of the leachate from only magnets (pH 0.82), which was used to optimize oxalate precipitation, suggested that a higher pH might hinder the precipitation process. Thus, the pH was adjusted to 0.60 with concentrated HCl, which was then followed by addition of oxalic acid. Remarkably, this treatment resulted in precipitation of the mixed RE oxalate salt; ICP-OES analysis of redissolved RE oxalate (see SI for details) showed a purity of >99%. Overall, this is equivalent with a recovery of 82% of RE elements in one motor.

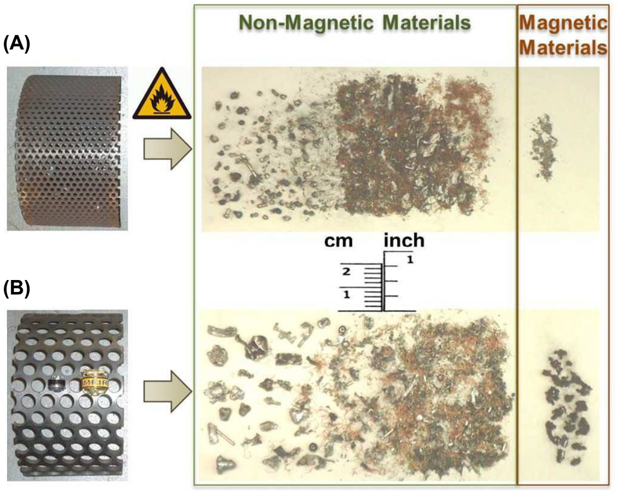

Figure 6. Shredded Magnetic and Non-magnetic Scrap Obtained with Different Size Screens. (A) 6 mm screen. (B) 19 mm screen. For size comparison, picture (B) has been taken with motors in front of it.

Recovery of REs from 2014 Chevrolet EV Spark FWD Drive Unit Rotor Assembly.

Finally, we applied our process to the recovery of REs from a drive unit used in 2014 Chevrolet EV Spark FWD. As mentioned above, drive units and traction motors are relatively easy to dismantle out of electric and hybrid vehicles after their end-of-life and are thus prime target materials for the herein developed process. The initial analysis of the drive unit revealed that all RE magnets are located in the rotor, the outside part of the assembly. Thus, the motor core was removed manually in order to simplify the demagnetization and shredding steps. As no industrial scale hammermill was at hand, we cut the obtained rotor into pieces (Figure 7A; see SI for detailed information) which fit into the entry passage of the previously used hammermill (Schutte Buffalo Hammermill, Model W-6-H, 19 mm screen openings). Every other step was completed as detailed above, resulting in the recovery of 80% of the RE elements present in the original motor piece in >99% purity (see Figure 7C) together with steel chips separated from the leaching solution by simple filtration (Figure 7B).

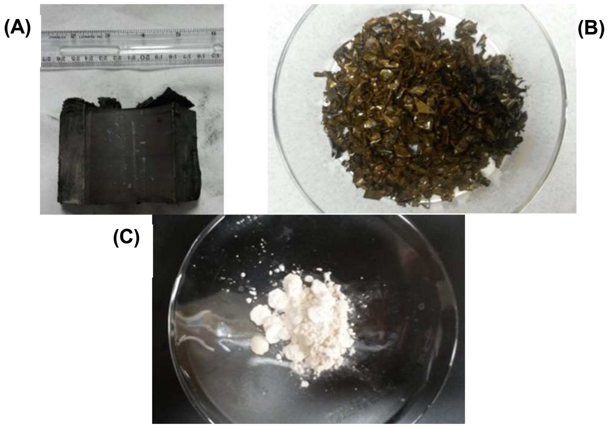

Figure 7. (A) Piece of 2014 Chevrolet Drive Unit Subjected to Recovery Process. (B) Steel Chips Isolated by Filtration after Leaching with 4 M HCl. (C) Isolated RE Oxalate Precipitate.

In combination with the recovery of REs from small motors demonstrated above, this successful separation of REs shows that our process consistently provides high recovery efficiencies. Most importantly, we have shown that the processing principles can be used independently of the size of the motors as long as a suitable shredder can be employed.

Conclusions

In summary, we have established an efficient process for the recovery of rare earth elements from materials mixtures such as in shredded motors with a recovery rate of 80% or higher. While heat treatment (1 h, 450 ºC) is necessary for the further processing of RE motors, all other steps (hydrometallurgical and shredding) can be performed at room temperature, thus providing a process which is designed for energy efficiency. Selective leaching enables efficient separation of steel and copper by taking advantage of the different reduction potentials of steel, copper, and RE magnets, while the selective precipitation of RE oxalates is the key for separating the REs from other compounds present in the leaching solution. Thus, we have demonstrated that Green Chemistry principles are efficient guidelines for the design of hydrometallurgical processes; we expect that these principles will be widely applied to this end in the future.

Experimental Part

General Procedure for Leaching Optimization.

The weight of each magnet was determined before the each setup. A magnet was added to a 20 mL scintillation vial equipped with a magnetic, Teflon-coated stirbar. Water and acid were added sequentially and the vial was sealed with a pressure relief cap (capable to vent for releasing overpressure formed by H_2 evolution). The resulting mixture was stirred at room temperature (1500 rpm) for 24 h. After the reaction time was complete, the remaining magnetic material was removed from the vial, washed with DI water (3 x 10 mL), and dried in air, after which the remaining weight was determined.

Leaching Kinetics of Rare Earth Magnets, 1018 Steel, 304 Steel, and Copper

In analogy to the general procedure, 4 M HCl (20 mL) and one piece of material (typically 0.5 to 1 g; bonded/non-bonded magnets, steel washers made of 1018 or 304 steel, copper wire) were stirred in a vial equipped with a Teflon-coated stirbar and a pressure-relief cap at RT. After the respective reaction times, recovery and weight determination of the undissolved material (see Table 1) was performed as described in the general procedure.

Table 1. Percent Dissolution of Rare Earth Magnets, Steel and Copper in 4 M HCl versus Reaction Time in the Acid Solution.

Time [h] Material	0	0.5	1	2	4	6	10	24
NdFeB	0%	9%	27%	73%	100 %	100 %	100 %	100 %
NdFeB bonded	0%	9%	23%	55%	84%	95%	97%	98%
1018 steel	0%	0%	0%	0%	0%	0%	0%	0%
304 steel	0%	0%	0%	0%	0%	0%	0%	0%
copper wire	0%	0%	0%	0%	0%	0%	0%	0%

Rare Earth Oxalate Precipitation from Leaching Solution

An acidic mixture of $NdCl_3$, $FeCl_3$, and boronic acid was produced by dissolving a NdFeB magnet (0.77 g; corresponding to 1.23 mmol REs (Nd, Pr, Dy), 9.11 mmol Fe, and 0.71 mmol B) in 4 M HCl and the mixture was stirred for 24 h at room temperature. After this time, oxalic acid (as solid, 0.2 M solution or 0.6 M solution in DI water; 1.5 to 3.0 equiv.; 1.84 to 3.69 mmol) was added to the solution. The formed precipitate was collected by filtration on a glass filter frit and washed with 10 mL of DI water. After drying in air, the weight of the precipitate was measured (see Table 2 and Figure 8).

Table 2. Percent Rare Earth Recovery from Leaching Solution by Precipitation with Oxalic Acid.

equiv. $C_2O_4H_2$ added $C_2O_4H_2$ concentration	1.5 (1.84 mmol)	2.2 equiv. (2.70 mmol)	3.0 equiv. (3.69 mmol)
0.2 M	82 ± 3%	88 ± 2%	99.2 ± 0.2%
0.6 M	86 ± 3%	92 ± 2%	99.0 ± 0.2%
solid $C_2O_4H_2$	90 ± 2%	96 ± 3%	99.2 ± 0.0%

In order to analyze the purity of the obtained precipitate, 1.50 g precipitate was dissolved in 20 mL 4 M HCl, which was then diluted to 200 ml with Millipore water and analyzed by ICP-AES.

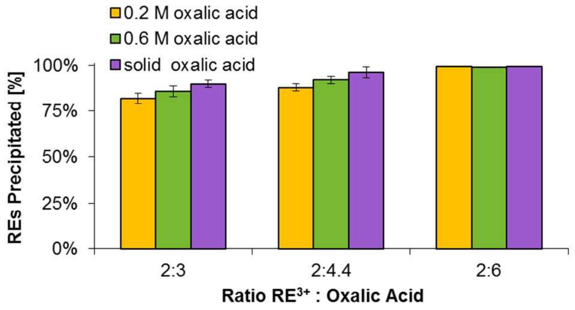

Figure 8. Optimization of Rare Earth Precipitation from Leaching Solution.

Process for Rare Earth Recovery from Motors

A motor (76.6 g overall, determined independently by weighing), consisting of NdFeB magnets (14 × 0.57 g magnets corresponding to 8.0 g; 17 mmol of a 15:1:4 mixture of Nd, Dy, and Pr, as determined by ICP-AES), copper coils (7 g), and steel casing (56 g) was demagnetized in a furnace (temperature program: heat to 450 C for 105 min, keep at 450 ºC for 60 min, cool to room temperature for 105 min; weigth after heating: 70.9 g). After cooling, the motor was shredded with a hammer mill (Schutte Buffalo Hammermill, Model W-6-H, 19 mm screen openings). The resulting scrap (67.3 g) was treated with 4 M hydrochloric acid (200 mL, 800 mmol, 47 equiv.) for 6 to 24 h; hydrogen evolution was observed during the leaching process and ceased after 6 h. The resulting leach solution showed the presence of 88% of rare earths (15 mmol; Nd:Dy:P = 15:1:4) from the original magnet materials as determined by ICP-AES. The solution was filtered through a paper filter (pore size 11 μm) to separate the remaining steel and copper solids. The pH of the filtrate was adjusted to pH 0.60 with 15 mL of concentrated hydrochloric acid. Oxalic acid (4.0 g, 45 mmol, 3 equiv. compared to previously determined 15 mmol of rare earths) was added as a solid to the solution. After stirring the resulting suspension for 120 min at room temperature, the precipitate (mixed rare earth oxalates) was isolated by filtration and washed with 20 mL of water. TXRF analysis of the remaining filtrate showed the presence of Zn, Fe, Co, Ni, Mn, Nd, Dy, and Pr in the filtrate. For purity analysis by ICP-AES, the precipitate was redissolved by addition of 20 mL of 4 M hydrochloric acid; the analysis showed the presence of 14 mmol of rare earths (Nd:Dy:Pr= 15:1:3) in a purity of 99.8% with an impurity of 0.02% Fe. The overall recovery can thus be calculated to be 82%, based on the amount of rare earths present in the original motor material.

Acknowledgements

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Notes and references

‡ Even though we cannot conclude that all types of steels will be inert from being leached into solution under these conditions, this result with the very commonly used steel alloys 1018 and 304 suggests that other steel alloys might behave similarly. For example, in later experiments with the steel/magnet materials mixture obtained from shredding motors, steel leaching became only significant upon prolonged exposure to acid (>48 h).

§ The Curie point (temperature where permanent magnetism changes to induced magnetism) for NdFeB magnets lies typically between 310 °C and 370 °C, depending on the exact composition of the magnets.

§§ The motors were obtained through amazon.com from the seller "Great Planes". The model obtained was labeled as Great Planes ElectriFly RimFire .10 35-30-1250 Outrunner Brushless Motor. For more details, see SI.

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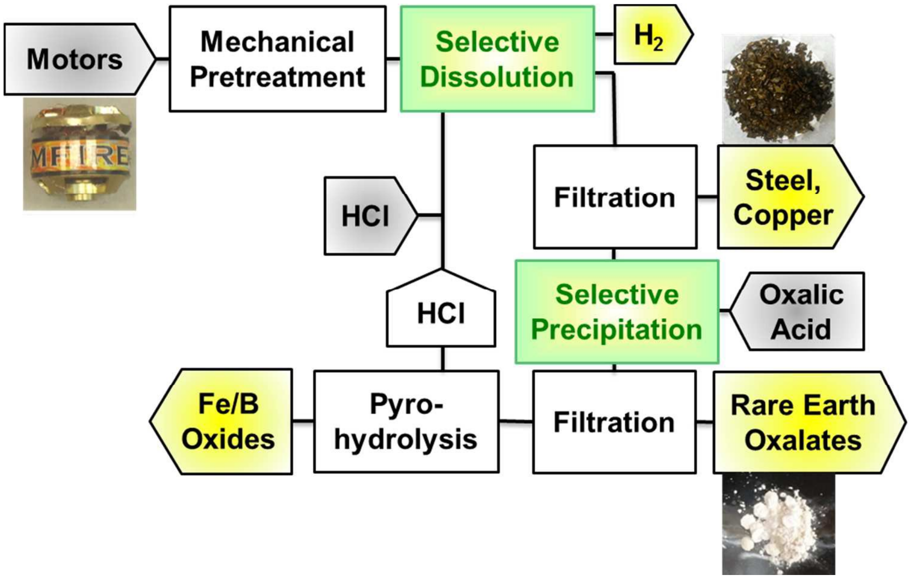
Graphical & Textual Abstract



This manuscript describes the development of an efficient process for the recovery of rare earth elements from materials mixtures such as in motors with a recovery rate of >80%. While heat treatment is required for processing, all other steps can be performed at room temperature, thus resulting in a process designed for energy efficiency. Selective dissolution enables efficient separation of steel and copper by taking advantage of the different reduction potentials of the materials in the mixture, while selective precipitation of RE salts is the key for obtaining pure RE products. Overall, the established process applies green chemistry principles for designing a hydrometallurgical process.