

Sustainable Method for Recycling Nd from Old Electronic Parts with Element-specific Sorting that Makes Hydrometallurgy Easier

Delgado VA*

Department of Applied Physics, School of Sciences and Modelling Nature (MNat) Scientific Unit of Excellence, University of Granada, Spain

Abstract

Neodymium is a pricey strategic metal that is absolutely necessary for our society; the most important issue is to ensure its availability and ensure its sustainable use. We are far from this, however, as less than one percent of it is recycled at the moment. Despite their 12 percent share of the Nd market, recycling small Nd-containing ceramic-based components has received little attention despite some work being done to recycle electrical engine's large Nd-based magnet alloys. A method for economically viable Nd recycling from such electronic waste components is demonstrated in this paper. We handled the issue of high Nd weakening in complex e-squanders through the upstream arrangement of synchronous dismantling, machine vision, and multi-energy X-beam transmission arranging (first-of-its-sort), trailed by exact beneficiation in righteousness of the distinction in attraction, thickness, and break sturdiness. The concentrates that are produced are mostly dielectric ceramics (BaO-Nd₂O₃-TiO₂) with 38.3% by weight of Nd. Despite the fact that earthenware production are hard-headed by and large, Nd can be just filtered in concentrated HNO₃ from these dielectrics with high selectivity under upgraded conditions. Oxalate precipitation can then be used to recover Nd, and 60 percent of HNO₃ can be regenerated. Through theoretical calculations, the underlying kinetics, thermodynamics, and bonding nature of the leaching were investigated to expand on the mechanism. Nd₂O₃ of commercial grade (> 99.6%) is produced with an overall efficiency of 91.1%. All the more critically, the reusing course is assessed beneficial in no less than four years in light of moderate edges. A promising approach to recovering rarely recycled strategic metals in the direction of a closed-loop design is presented in this work.

Keywords: Clay; Mining for copper; Electro acoustics; Flocculation; Silica surfactants

Introduction

Modern technologies like communications, electric vehicles, renewable energy, cutting-edge materials, and other applications that affect our day-to-day lives are revolutionized by rare earth elements (REEs). Albeit not being scant in geographical terms, REEs definitely stand out as essential assets because of the lopsided local circulation of monetarily suitable minerals, and worries about shortage in supply chains [1]. The worldwide market for REEs is assessed at 221 thousand tons for the year 2020, which is fundamental for businesses worth trillions of dollars. Among the 17 REEs, Nd is recognized as one of the most basic as far as its significance to clean energy creation and its production network related dangers, and request is expected to ascend by 700% from 2010 to 2035. Because of fixed Chinese commodity amounts, numerous nations have accumulated pace in broadening their stock chains.

Given the specialized challenges in mining and partition, and the related unfavorable natural outcomes, a couple of nations, similar to the U.S, are supporting their REE creation from essential assets, while different nations that need REE assets, similar to Europe and Japan, are equipping REE reusing from optional assets. This is especially gainful whenever reused REEs are those that are hard to find. As a result, chemical methods have been the focus of some recent research on recycling Nd from obsolete permanent magnets and rechargeable batteries. Although significant chemical use and effluent treatment prevent a profitable industry, recycling has obvious potential. Different undertakings were dedicated to settling the high fluctuation of information materials or focusing on green REE creation and reusing, for example, bioleaching, peptide-based mineralization, particular crystallization, specific assimilation through nanoengineered cellulose, and particular ligand science in light of thermodynamic or active control. By and by, the ongoing reusing pace of Nd is still very low

(~1% in Europe) because of the test of monetarily coordinating the new cycles into existing cycles. If we are to move toward a circular economy made of Nd, we absolutely need environmentally friendly recycling routes right away.

Recycling Nd from ceramic-based electronic components, which account for 12% of all Nd end uses, has a significant potential that has been overlooked, but it can be used in more applications than just batteries and magnets. Pottery that contain Nd are a group of microwave dielectric materials usually utilized in cell phones, base stations, and other electronic and correspondence applications. Due to their excellent dielectric properties, the number of applications for these ceramics will skyrocket with the advent of 5G and the trend toward electronic miniaturization. These waste dielectrics are discarded with a flood of highly complex e-wastes in which Nd is so diluted that it is at trace concentrations, in contrast to recycling of large magnets or battery alloys, which have easier access to their resources. Pre-concentration of Nd from individual components is much more difficult to achieve due to the lack of element-specific sorting technology, despite existing industrial processes that dismantle e-wastes [2]. In fact, industrial applications of sensor-based sorting are element-independent (with

*Corresponding author: Delgado VA, Department of Applied Physics, School of Sciences and Modelling Nature (MNat) Scientific Unit of Excellence, University of Granada, Spain, E-mail: dalg.ado@avdel

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the exception of laser-induced breakdown spectroscopy, which can only perform near-surface analysis). Although the concomitant photon energies span the spectroscopic characteristics of the majority of elements, X-ray imaging may provide elemental identification, no actual application to e-waste processing has yet been established beyond single or dual energy transmission. As a result, we hypothesize that, despite the difficulties, an integrated technological innovation based on optical, X-ray spectroscopy, and other physicochemical techniques that is able to fully exploit selectivity ought to provide sufficient Nd pre-concentration to enable Nd recycling.

Methods and Materials

Methods

Vans Chemistry Pte donated e-waste for collection, Metalo International Pte and Ltd., both based in India Ltd., in Singapore. PCBs, computers, mobile phones, televisions, and other small household appliances were among the e-waste that was obtained. In order to retrieve their PCBs, these complete electrical and electronic devices were first disassembled manually. To melt the solder and release the surface-mounted ECs, all PCBs were shaken and heated for 10 minutes in a box furnace (Lindberg/Blue M, Thermo Scientific) at 250 °C. These ECs were then blended as the first e-squander stream for the arranging study. In addition, Vishay Intertechnology, Inc. provided single-layer ceramic capacitors (SLCCs, C0G/NP0 Dielectric) as an additional source of Nd-containing ECs for the research of the subsequent hydrometallurgy and beneficiation processes. Sigma-Aldrich supplied the oxalate standard (TraceCERT®), nitric acid (AR, 70%), hydrochloric acid (AR, 37%), sulphuric acid (AR, 95–98%), oxalic acid dihydrate (AR, 99%), sodium hydroxide (AR, 97%), and neodymium (III) nitrate hexahydrate (99.9%). PerkinElmer supplied the metal standard solutions, which included neodymium, barium, titanium, silver, copper, and tin. A water purification system (WaterPro®, Labconco Co.) with a resistivity of 18.2 M Ω at 25 °C produced extremely pure water. The chemicals were used exactly as they were given.

EC optical sorting: To explicitly target ceramic capacitors utilizing optical arranging, we restricted the size scope of ECs by choosing two classifications, 1–5 mm, and 5–10 mm, for the sieved tests. These ECs were optically sorted using a self-designed conveyor belt prototype with a vibratory bowl feeder that was 2.5 meters long and 10 centimeters wide [3]. An industrial camera with a 12 mm fixed focal lens, 25 images per second, BU505MCF Telicam, Toshiba, lit by a white LED ring (V2DR-i90A-W, Vital Vision Technology), the ECs were continuously conveyed on the belt at a speed of 0.9 m/s and passed through a filming zone. After that, machine vision techniques were used to process the images and identify the distinct characteristics of the various ECs, such as their colors, appearances, edges, and sizes. The database's traits were specifically categorized using a convolutional neural network (CNN). The CNN was written in Python (form 3.6.8) utilizing an open-source library (TensorFlow) and was based on a successive mode in light of three 2D convolution blocks with a maximum pool layer relating to every convolution layer. At last, electro-pneumatic spouts redirected the perceived ECs off the belt into arranging receptacles by directional wind current.

Sorting of SLCCs via ME-XRT: The component based ID of SLCCs was performed on a particular formed ME-XRT gadget. A Xylon source with a 1 mm aluminum filter, operating at 120 kV and 1.2 mA, produced X-rays [4]. The XRT range was recorded by a multi-energy X-beam locator (ME100, Recognition Innovation) with an obtaining season of 100 ms for each line, made out of 128 pixels, comparing to

the actual pixels of the identifier. The SLCC tests were put on cardboard that was joined to a mechanized stage going at 0.4 cm/s along a course opposite to both the identifier line and the X-beam pillar. The sample was 100 cm away from the source, which was 150 cm away from the detector. By integrating the total transmittance of each pixel across the entire energy range (20–150 keV), the XRT mapping for the entire scanning area was obtained. The XRT range of every individual SLCC was basically found the middle value of from every one of the pixels that create the SLCC body region to eliminate the photon signal commotion from the finder. The XRT spectrum can be used to interpret the K-edges of elements. Using a Python program, the K-edge distinctiveness of various elements and the conveyor belt's ejection accuracy were taken into account when simulating ME-XRT sorting. Our sorting conveyor is currently incorporating ME-XRT sorting, which is awaiting X-ray device operation licensing.

Beneficiation of Nd concentrates physically: The physical beneficiation process involved a number of processing units that were processed in succession. A cutting mill with a built-in 1 mm sieve was used to coarsely shred the sorted Nd-containing SLCCs in order to separate the inner materials from the cladding epoxy resins [5]. A permanent magnet was then used to separate ferromagnetic particles, most of which came from connecting wires. Non-magnetic materials were placed in a measuring cylinder that was one liter in volume and half-filled with tap water. The cylinder was periodically stirred until the particles stratified according to their varying settling velocities. The epoxy resin fraction was then extracted from the top layer using tap water. Brittle fracturing was used to fracture the remaining heavy portion that was transferred to a mortar. As pottery are fragile and metals are for the most part more malleable, we could promptly comminute the dielectric portion into better powder while the metal division was straightened. After passing the powder through a 0.25 mm mesh sieve, an Nd-containing dielectric fraction was obtained.

Acidic draining of Nd: Prior to preferential leaching, the remaining metal impurities were processed with a pulp density of 1500 g/L in 10 M HNO₃ for five minutes at 25 °C. The solid phase that resulted from filtration and drying was used as the actual dielectric material (DM) for Nd leaching [6]. To reduce acid loss from evaporation, the leaching was carried out in a 100 mL round bottom flask connected to a reflux condenser. A Teflon attractive stirrer and weighted DM were stacked into the flagon, which was lowered in an oil shower. The draining was performed while charging a known measure of corrosive. The temperature was directed by a hot plate (VWS-C7, VWR) and the blending speed was kept up with at 500 rpm. 10 M HNO₃, 10 M HCl, and 5 M H₂SO₄ were thought about utilizing similar filtering conditions (50 g/L and 70 °C) to choose the mineral corrosive that had the best draining selectivity. Moreover, for the draining with HNO₃, we applied a Case Behnken reaction surface plan to concentrate on the impact of corrosive fixation, temperature, and mash thickness on the filtering rate, Nd focus at balance, and filtering proficiency. A correlation between the concentration of HNO₃ and its boiling point that was generated by the EquilWin software was used to select the acid concentration range of 7.5–12.5 M and the temperature range of 70–110 °C. The pulp density was examined over a range of 50–400 g/L, which is suitable for use in industry. The leaching condition was optimized by considering the reasonability of each operating parameter and the maximization of the three responses. The Nd concentration was measured with an X-ray fluorescence (XRF) spectrometer, which was calibrated by an inductively coupled plasma–optical emission spectrometer (ICP-OES), until it reached a plateau in order to follow the leaching kinetics. In addition, in order to characterize the final leachate solution and the

solid residues that remained after filtration (0.22 m), we carried out the leaching with samples that were only taken at the plateau time.

Precipitation and calcination

Specification simulations performed with the Hydra-Medusa software were used to guide the oxalic acid dosage and Nd feed concentration optimizations in oxalic acid-based Nd precipitation experiments [7]. The precipitation tests were done in encased vials at 25 °C for 3 h with consistent blending. Before and after precipitation, the oxalate (ox) and Nd concentrations were measured. The final metal oxide products were produced by calcining the filtrate (0.22 m) for one hour in a box furnace at 900 °C.

Examples of descriptions

After acidic digestion at 240 °C for 1.5 hours with a microwave digestion system (ETHOS UP, MILESTONE), the metal content of raw SLCC samples and calcined products was measured with an ICP-OES (OP Optima 8000, PerkinElmer) [8]. After the necessary dilution, either an ICP-OES or a handheld XRF spectrometer (Vanta C series, Olympus) calibrated by the ICP-OES was used to measure the concentration of metals in the solution. The pH of the arrangement was estimated by a logical pH meter (SevenCompact, METTLER TOLEDO) after weakening multiple times thinking about that the separation of profoundly weakened HNO₃ is practically finished. Ion chromatography (930 Compact IC Flex, Metrohm) was used to examine the concentrations of oxalate ions in solution. A powder X-ray diffractometer (XRD-6000, Shimadzu) with Cu K (= 1.5406) radiation and a step size of 0.01° over a range of 10 to 80 was used to examine the samples' phase compositions. The elemental composition was used to calculate the phase quantities, and MAUD software (version 2.98) used Rietveld refinement to analyze them. Surface morphology and basic planning of the strong stage were described by filtering electron microscopy furnished with energy-dispersive X-beam spectroscopy (SEM-EDS, JSM 7600F, JEOL). Using a digital microscope (50, RS Pro), microscopic images were taken. The water displacement method was used to measure the density of the particles. Bulk SLCCs were ground with sandpaper to expose a polished surface of the DM in order to test the DM's fracture toughness. The bulk SLCCs were hot mounted in an epoxy material in the shape of a cylinder [9]. The test was performed by a miniature Vickers hardness analyzer (Wilson VH1150, Buehler) at a power of 49 N with the pinnacle load kept up with for 10 s and the break durability was determined following the condition.

Economic evaluation and simulation of a flowchart

SuperPro Designer was used to perform the flowsheet simulation and economic analysis for the entire commercial-scale Nd recycling process. We simulated the two sub-processes separately because the downstream hydrometallurgy section was designed to operate in a batch mode while the upstream sorting and beneficiation section can operate continuously. The same operating parameters that were optimized from laboratory-scale experiments were used to calculate the mass and energy balance. The fixed capital costs, operating costs, and revenues from major products were used to estimate the economic indices. The information from commercial websites or the built-in database of SuperPro Designer were used to determine the price of each input and output material. For a general mix of electronic components obtained from PCB disassembly, we utilized Nd-based ceramic capacitors in a fraction of 1.0 weight percent. This is based on our mass balance of 100 kg of PCBs that were disassembled and contained less than 10% ceramic-based capacitors, with a probability of 0.1 that these

are Nd-based ceramics. We have defined a very conservative scenario by focusing solely on the Nd and not taking into account the recovery of other metals that are known to be economically viable. In addition, the optical-X-ray sorting process's operating costs could be as low as 0.2–0.3 dollars per metric ton of processed material, while our utility costs are 2.8 dollars per metric ton and labor costs are 72.4 dollars per metric ton.

Hydrometallurgy selectively

After that, the DM fraction underwent a brief initial 5 minute course of leaching with 10 M HNO₃. As 93.4 percent of the Cu and 98.2 percent of the Ag were selectively removed into the leachate, while 99.9 percent of the Nd remained in the solid phase. Sn could also form insoluble metastannic acid (H₂SnO₃), which is why it mostly remained in the solid phase [10]. Then, we compared how well three mineral acids leached. For Nd and Ba, the leaching efficiency of HCl and HNO₃ was comparable to that of H₂SO₄, which was higher. However, there was significantly less Ti transfer into the leachate in HNO₃ than in HCl, which resulted in superior selectivity. As a result, HNO₃ was chosen as the most effective mineral acid for DM leaching. Based on a Box-Behnken response surface experimental design, a further investigation of the effects of operating parameters on Nd leaching performance was carried out. According to the findings, raising the temperature had little effect on the Nd leaching efficiency but significantly increased the reaction rate. Nd focus at harmony expanded directly with mash thickness in the concentrated on range. The concentration of HNO₃ had no direct effect on the three responses; However, it may have an effect on HNO₃'s boiling point, which limits the maximum temperature that can be heated at ambient pressure. Subsequently, the ideal filtering not entirely set in stone to be at 10 M HNO₃, 110 °C, and 400 g/L. These ideal circumstances gave the benefit of a superior draining selectivity for Nd over Ba. Additionally, we investigated the DM Nd leaching kinetics. According to shrinking core models, the kinetic data fit better with indicating that product layer diffusion control dominated the leaching kinetics rather than surface chemical control. The leaching equilibrium was reached within 25 hours under these ideal conditions, with final neodymium concentrations close to 1 M and an Nd leaching efficiency of up to 99.3 percent.

Hundreds of corrosion fronts and newly formed agglomerations of crystals, ranging in size from nano-scale to sub-micro-scale based on the duration of the leaching, replaced the DM particles' smooth morphology following the leaching [11]. The XRD results also confirmed that TiO₂ was formed instead of the DM's initial crystalline and amorphous phases, which gradually disappeared. The solid residue contained some unidentified peaks, but the amounts compared to TiO₂ appeared to be insignificant. At a leaching efficiency of 99%, the remaining Nd-containing phases were barely present in the solid residue, confirming the excellent leaching selectivity.

We then, at that point, accelerated Nd³⁺ with oxalic corrosive to recuperate Nd from the leachate according to the response. According to the Hydra-Medusa simulation, in order to complete Nd precipitation in a concentrated HNO₃ medium, an excess amount of oxalate (ox) is always required in addition to the stoichiometric amount [12]. The negligible part of non-accelerated Nd at the stoichiometric measurements, and the proportion of [ox]/[Nd] for careful precipitation both reduction with an expansion in beginning Nd focus. As a result, the leachate from Run 5 with the highest Nd concentration was the ideal feed solution for Nd precipitation. The mimicked measurement of oxalic corrosive for careful precipitation was checked tentatively to be more alluring than the stoichiometric dose, with regards to the

precipitation productivity of both Nd and oxalate particles. Since HNO₃ is more expensive than more conventional industrial acids, its ability to be regenerated through the precipitation reaction is crucial. We measured a recovery rate of 59.9%, with total [HNO₃] decreasing from 6.8 M to 6.3 M due to HNO₃ thermal decomposition and other side reactions. This is in contrast to the theoretical expectation that 100% HNO₃ would be recoverable from the leaching solution with no change in total [HNO₃] following the precipitation.

In contrast, Nd recovery was impracticable for D&CM when subjected to the same hydrometallurgical process due to the presence of some impure metals like Cu, confirming the prerequisite role of beneficiation and pre-leaching to the successful implementation of hydrometallurgy in the recycling route [13-18]. During the hydrometallurgical stage, the rate of Nd recovery reached up to 98.2%, and the calcined product at 900 °C showed a purity of Nd₂O₃.

Conclusions

In outline, that's what our review demonstrates, interestingly, one can productively reuse Nd from the ceramic portion contained in profoundly complex e-wasters following an in fact, monetarily, and naturally practical course. The issue of high Nd dilution in e-wastes can be effectively addressed through simultaneous disassembly, optical, and MEXRT sorting, and this element-specific sorting can stream other valuable ECs into different categories. Utilizing a variety of physical properties, the use of physical beneficiation can further simplify the elemental composition. In order to achieve high selectivity in Nd recycling, the obtained Nd concentrates only require straightforward leaching and precipitation procedures. In addition, H₂C₂O₄, which is more environmentally friendly, can replace nearly 60% of HNO₃ in each processing loop. This incorporated Nd reusing course prompts a high recuperation proficiency (91.1%) and a high immaculateness of neodymium oxide item (> 99.6%). To additionally expand the reusing potential, future examinations will explore reusing Nd-doped dielectrics, which contain less Nd, more scaled down MLCCs, and other REEs. In a broader sense, our research demonstrates a promising opportunity for the economically and environmentally advantageous urban mining of other unrecycled "specialty metals" from electronic waste.

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Conflict of Interest

None

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