Identification and recovery of rare earth elements from electronic waste: Material characterization and recovery strategies

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Identification and recovery of rare earth elements from electronic waste: Material characterization and recovery strategies

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ABSTRACT

The sustained growth of the electronic and electrical industries necessitates not only efficient energy utilization throughout all manufacturing stages but also the recycling of end-of-life electric and electronic components. However, rapid advancements, miniaturization, and added value have led to a significant accumulation of e-waste, posing environmental concerns. Rare Earth Elements (REE) are considered critical raw materials that face a risk of global supply shortage due to their highly desirable performance-enhancing properties such as corrosion resistance. This study focuses on identification and exploring the recycling possibilities of different types of electronic waste for Rare Earth Materials. The electronic waste is first disassembled and categorized, after which material characterization techniques are employed to identify Rare Earth Elements (REEs). X-Ray Diffraction, X-ray fluorescence spectroscopy, Scanning Electron Microscopy, and Energy-dispersive X-ray spectroscopy are utilized for this identification. Subsequently, an in-depth review of existing literature is conducted to ascertain the most appropriate method for recovering these REEs. Neodymium and Dysprosium are among the REEs identified in the electronic waste samples.

1. Introduction

Electronic waste, also known as e-waste, is generated when electronic products are discarded after the end of their useful life. The fast-paced growth of technology and the consumption-driven society contribute to the substantial increase in e-waste production every year. E-waste encompasses all discarded parts and items of electronic and electrical equipment (EEE) that were not intended for reuse [1]. In 2019, the United Kingdom generated a record-breaking 53.6 million tonnes of e-waste worldwide, which is a 21 % increase in the past five years. This amount of e-waste surpasses the total tonnage of all commercial aircraft ever produced, providing a sense of scale to the magnitude of the issue. The UK is among the top ten nations in e-waste production per capita, along with the United States, France, and several countries in Scandinavia [2]. Annually, the global consumption of Electrical and Electronic Equipment (EEE) increases by 2.5 million metric tons (Mt). The worldwide production of e-waste reached a remarkable 53.6 million metric tons in 2019, equating to an average of 7.3 kg per capita. Experts project that this number will grow to 74.7 Mt by the year 2030 [3]. In Mendeleev's periodic table, a group of 17 elements, are known as rare earth elements. These elements have become essential strategic resources in

national defence, energy, glass ceramics, and agriculture industries. As science and technology continue to advance globally, the potential of rare earth elements in various fields is continuously being explored [30]. Numerous studies have been conducted for the recovery of materials from e waste. In one study, shredded e-waste materials obtained from various sources, such as user scrap, LCD scrap, laptop-computer shred, motherboard, and hard drive shred, were subjected to analysis for their metallic content and characterization. The results revealed that user scrap exhibited the highest copper content of 34.5 wt%, while a reduction in metallic content was observed with particle size reduction in the majority of the analyzed e-waste materials [32]. In another study, the study successfully recovered copper from e-waste leaching solution using LIX984 N extractant with kerosene as the diluent. Optimized conditions resulted in over 94 % copper transfer and less than 10 % iron content during the loading phase, with 92 % stripping efficiency [31]. Electronic waste contains valuable Rare Earth Materials, making the identification and extraction of these materials critical for the electronics industry [4]. In recent years, concerns about the availability and sustainable recovery of rare earth elements (REE) have grown, leading to their classification as critical raw materials due to the supply risk. The earth's natural reserves are unable to meet the global demand for these

strategic resources, further highlighting the importance of finding a more sustainable method for REE recovery from electronic waste. Consequently, there has been a more proactive global response to address this growing shortage issue [5]. Production of rare earth elements (REE) is mainly concentrated in China. These elements play a crucial role in the manufacturing of various technologies, including fluorescent lamps, fluid catalysts, and permanent magnets used in computer hard drives (HDD), among other electrical and electronic appliances, due to their performance-enhancing features [6]. Neodymium (REE) magnets are a prime example of the importance of rare earth elements in technology. Even though they represent only a fraction of the total weight of hard disk drives, the voice coils and spindle motors would not be able to function without them [7]. When these products reach the end of their life cycle, they can pose a significant threat to the environment, particularly if they contain radioactive elements such as thorium and uranium [8]. The expanding electronics and electrical industry have led to a significant increase in the demand for rare earth elements (REE), creating concerns about the sustainability of these materials [9].

Various methods have been attempted by researchers to extract rare earth elements (REE) sustainably from electronic waste (e-waste). These methods include chemical precipitation, ion exchange, solvent extraction, membrane separation, and adsorption. Ongoing research aims to optimize and identify the most efficient method for recovering REE from e-waste. [10]. Pyrometallurgy emerges as the prevalent method for metal separation and recovery, with smelting being the primary choice for over 70 % of waste PCBs. Various techniques, including incineration, plasma or blast furnace smelting, drossing, sintering, melting, and high-temperature gas reactions, have been commonly employed for extracting non-ferrous and precious metals from electronic waste. The process involves introducing crushed PCBs into smelters to extract copper and precious metals, followed by upgrading in a converter and anode furnace, and ultimately subjecting them to electrolysis or electro-winning for electro-refinement [33]. While pyrometallurgical manufacturing offers advantages, it is not without its challenges and drawbacks. The process generates toxic materials and dioxins from flame retardants during e-waste smelting, necessitating advanced pollution controls in state-of-the-art smelters to prevent their release. Moreover, the metal separation achieved during e-waste smelting is only partial, leading to limited value upgrading of the metals [33]. Hydrometallurgical techniques for dissolving metals from e-waste encompass acid or caustic leaching, along with separation and purification methods like cementation, solvent extraction, activated carbon, and ion exchange. These approaches are employed to separate and extract metals from the leached solution, with acid leaching being a commonly preferred method. While cyanide leaching has been frequently used in hydrometallurgy, its high toxicity has prompted researchers to explore alternative chemistries, such as thiosulfate, for the extraction of precious metals [34]. Bioleaching presents a captivating and promising leaching approach. Following the leaching process, various methods are employed for metal recovery [35]. Despite encountering these challenges, the researchers explored alternative chemistries involving the utilization of ammonia and Bronsted acidic ionic liquids. In the ammonia-based leaching chemistry, ammonium carbonate, NH3 / NH4Cl, and an oxidant, such as CuCl2, were used. Copper forms amine complexes, and the stability of ammonia groups relies on factors such as pH, redox potential, and ammonia concentration [36].

The objective of this study is to investigate and evaluate the most effective method for extracting rare earth elements (REE), specifically neodymium and dysprosium, from e-waste. Dysprosium is commonly utilized to enhance the temperature stability of magnets against demagnetization. The analysis will focus on e-waste components such as magnets and solder joints on the PCB board from a Hard Disk Drive (HDD). The study will commence with a material characterization process to identify the extraction methods. The paper is structured in a manner where it first discusses the materials and methods utilized,

Table 1
Parameters used for the XRD analysis.

Scan type	Coupled 'TwoTheta'
Time/Step (s)	0.15
Steps	3427
Total Time (s)	535.65
2Theta Start (°)	20
2Theta Increment (°)	0.020433383
2Theta Stop (°)	90.0048
X-Ray Generator Voltage (kV)	40
X-Ray Generator Current (mA)	40
K-Alpha1 [Å]	1.5406
K-Alpha2 [Å]	1.54443
K-Beta [Å]	1.39225

followed by the presentation of results and discussions. Finally, the study concludes with its findings and implications.

2. Materials and methods

2.1. Identification of electronic waste

The magnet and PCB used for the analysis were obtained from an outdated electronic device and are depicted in Fig. 5. Special tools were utilized to remove them from the electronic device, and the solder joints were meticulously extracted for analysis. Due to the small size of the components, this necessitated the use of specialized tools and care during the dismantling process. In addition, inductively coupled plasma-optical emission spectroscopy (ICP-OES) was performed to identify the rare earth element from the e-waste by following ASTM UOP389–15 standard [11]. It is found that the OES data confirmed the presence of Fe - 58 %, Nd - 32 %, Pr – 4.2 %, and B – 0.85 % in the magnets and Fe – 71 % Nd – 23.8 % and Dy – 1.6 % in the circuit board which is in good agreement with the literature [13–16].

2.2. Sample preparation for XRD

X-ray diffraction analysis is a widely used technique for determining the crystallographic structure of a material by analyzing the diffraction pattern produced by the interaction of X-rays with the material's atoms [11]. when X-ray wavelengths come into contact with a crystalline substance, the substance acts as a 3D diffraction grating. This is due to the interaction of the incident monochromatic X-rays, generated by a cathode ray tube, with the electrons of the crystal atoms. This interaction is known as electron scattering [12]. To prepare the samples for XRD analysis, the HDD was first dismantled to obtain the magnets and solder joints from the PCB. The components were weighed to ensure accurate measurements, and then the solder joints and magnets were cleaned using a sterile microfibre cloth and propanol solution. The samples were then powdered for analysis. The XRD analysis was performed using the parameters listed in Table 1.

2.3. Sample preparation for EDS

Composition analysis is carried out using Energy Dispersive Spectroscopy (EDS) and Scanning Electron Microscopy (SEM) technology. With a magnification of up to 500,000 times and a controlled electron beam (up to 30 kV and 20 nA), a specific area can be imaged and analyzed for its composition. Sample preparation for EDS requires demagnetizing the specimen by heating it in an oven to the Curie point (310 °C). The magnet is then polished using a Buehler grinder polisher and vibrator, and the solder joints are prepared accordingly. To ensure that the solder joints and magnet are sterile, a microfiber cloth and propanol solution are used. Finally, the samples are mounted for analysis [13].
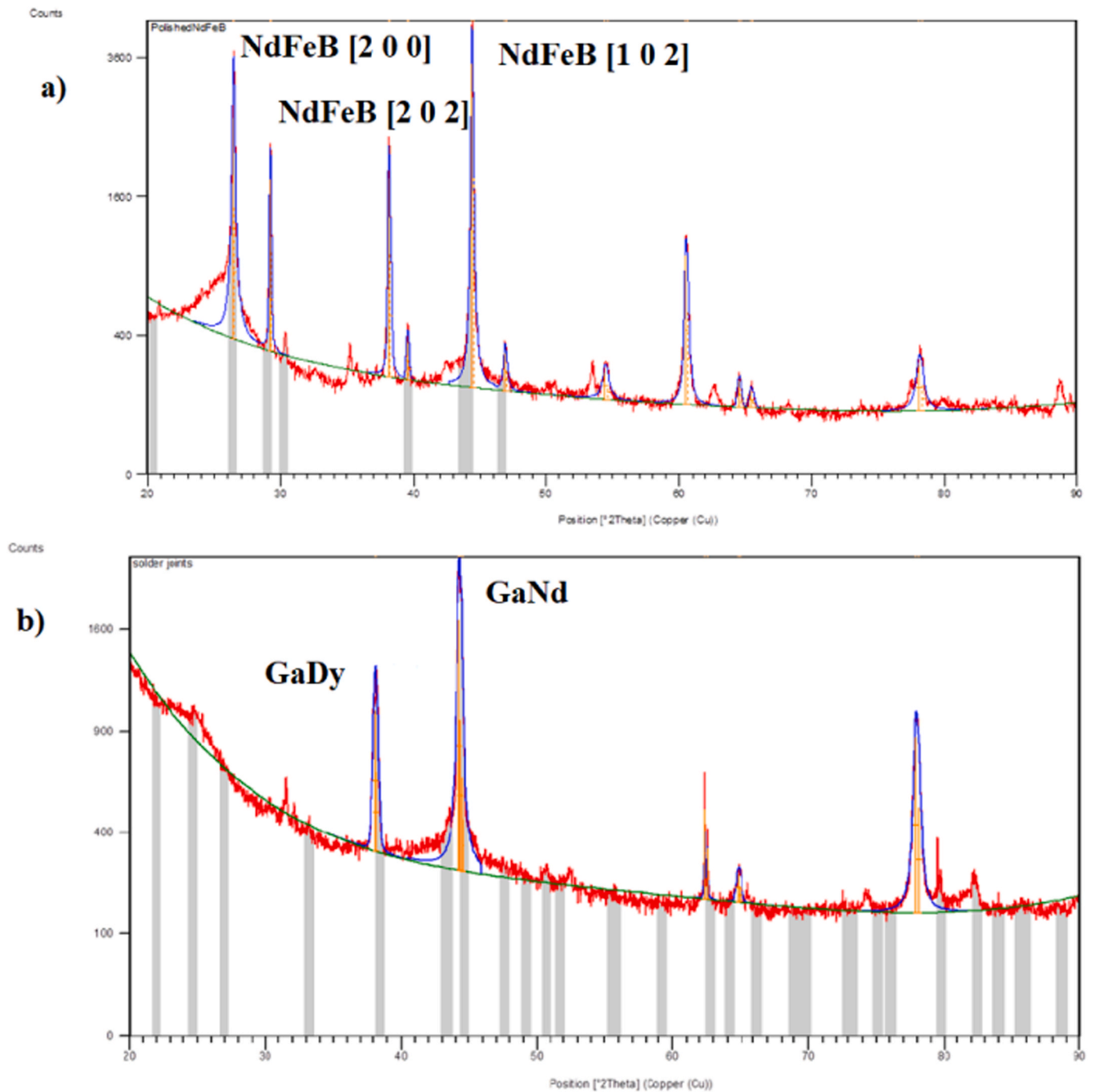
Fig. 1. a) XRD analysis result of the magnet specimen showing NdFeB b) XRD results obtained for the solder joint extracted from printed circuit board showing GaNd and GaDy.

3. Results and discussion

3.1. XRD results

According to the X-Ray Diffraction (XRD) analysis results, the crystalline phase of the E-waste samples was identified and their chemical composition determined [8]. The obtained XRD graph, presented in Fig. 1a, showed a stick pattern that was compared to a reference pattern for $Nd_2Fe_{14}B$. The peaks were then compared to reference standards and the compound identified as a phase superposed to $Nd_{4.4} Fe_{7.7} B_{17.8}$ with a tetragonal crystal structure [13]. The compound $Nd_2Fe_{14}B$ consisted of a tetragonal crystallographic structure, this leads to the elucidation of

the study of interest as the same crystalline structure was found [14]. Table 2 represents the details of the compounds identified through the analysis.

Table 2
Compound details identified using XRD analysis.

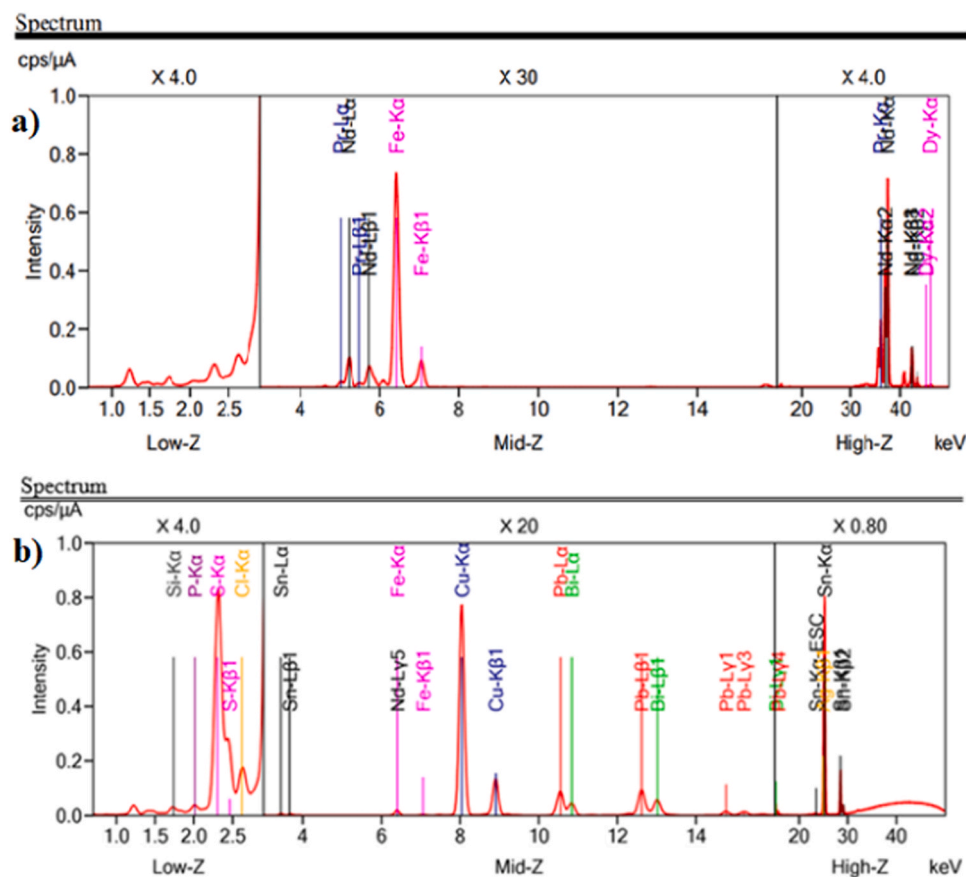| Compound Identified	Boron Iron Neodymium
Reference Code	00–043–1066
Empirical Formula	B17.8Fe77.8Nd4.4
Chemical Formula	Nd4.4Fe77.8B17.8
Crystal Structure	Tetragonal

Fig. 2. a) XRF spectrum results for the magnet specimen removed from the e-waste. b) XRD Spectrum results for the solder joint samples extracted from PCB.

Fig. 1b corresponds to the XRD graph for the solder joints examined. Background noise were reduced to improve the results; phase identification was undertaken to find compounds consisting of REEs, neodymium ($GaNd_3$) and dysprosium (Ga_2Dy) compounds were phase identified. $GaNd_3$ consists of a cubic crystal structure whereas Ga_2Dy has a hexagonal crystal system (Jain et al., 2013).

The XRD analysis was able to identify the crystal structure and phase of the magnet sample, but it was not effective in determining the composition of the solder joints.

3.2. XRF results

The elemental composition of the E-waste components was studied using X-ray fluoroscopy with the Rigaku NEX DE, which proved to be an important tool. Fig. 2a displays the spectrum of elements present in the magnet. The equipment can emit X-rays at low (6.5 kV, 402 μA), mid (35 kV, 305 μA), and high (60 kV, 200 μA) energy levels, which enables penetration of the different energy levels present in the sample. The Rigaku NEX DE X-ray fluoroscopy machine was calibrated to focus on the elements of interest, namely neodymium and dysprosium as rare earth elements, as well as iron, which comprises the majority of the magnet. The machine emits low, mid, and high levels of X-rays (6.5 kV, 402 μA; 35 kV, 305 μA; and 60 kV, 200 μA, respectively) to allow for penetration of the sample at different energy levels. The obtained elemental composition values for the magnet through multiple tests are Fe – 73.4 % by wt., Nd – 25.4 % by wt., and Dy – 1.2 % by wt. The graph in Fig. 4 shows that neodymium, iron, and traces of praseodymium were found at mid X-ray energy, while dysprosium was detected only at high energies.

The findings of this study are consistent with those of previous research on the presence of rare earth elements (REEs) in e-waste magnets. For instance, in a separate investigation, scholars explored the extraction of REEs from scraps of permanent magnets using $FeO-B_2O_3$ flux treatment. The weight percentage composition of the permanent magnet was determined to be Fe - 61.60 %, Nd - 30.73 %,Pr - 4.39 %, and B - 0.96 % by weight [15]. The congruence between the primary data obtained and the secondary data available in the literature provides reassurance regarding the reliability of the findings. Specifically, the elemental composition results obtained in this study align with those reported in previous research on the extraction of REEs from e-waste magnets. The relative error margin between the Nd and Fe values in the primary data and the corresponding values in the secondary data are 17.3 % and 16.1 %, respectively. Both the primary and secondary data indicate that iron is the most abundant element in the magnet, followed by neodymium. Hence, the experimental outcomes can be deemed credible and valid, substantiating the success of the endeavour to identify rare earth elements.

The process of obtaining results for the solder joints through the Rigaku NEX DE was challenging due to the uneven surface and small size of the samples, making it difficult to find the appropriate orientation. Fig. 2b demonstrates the effects of these challenges, as a greater range of elements is found in comparison to the spectrum of the magnet. Despite conducting three tests on the sample, dysprosium was only detected in one of the tests and neodymium was not detected at all. The analysis revealed the following weight compositions: copper (Cu) – 64.3 %, lead (Pb) – 15.4 %, tin (Sn) – 11.3 %, sulphur (S) – 8.62 %, dysprosium (Dy) – 0.362 %, and neodymium (Nd) – 0 %.

The results from the XRF analysis indicate that the solder joint contains predominantly copper and lead, with negligible amounts of rare earths. Neodymium is known to be added as a microalloying element to solder joints to enhance their wetting and spreading characteristics. However, its addition is typically in small quantities (<1 %)
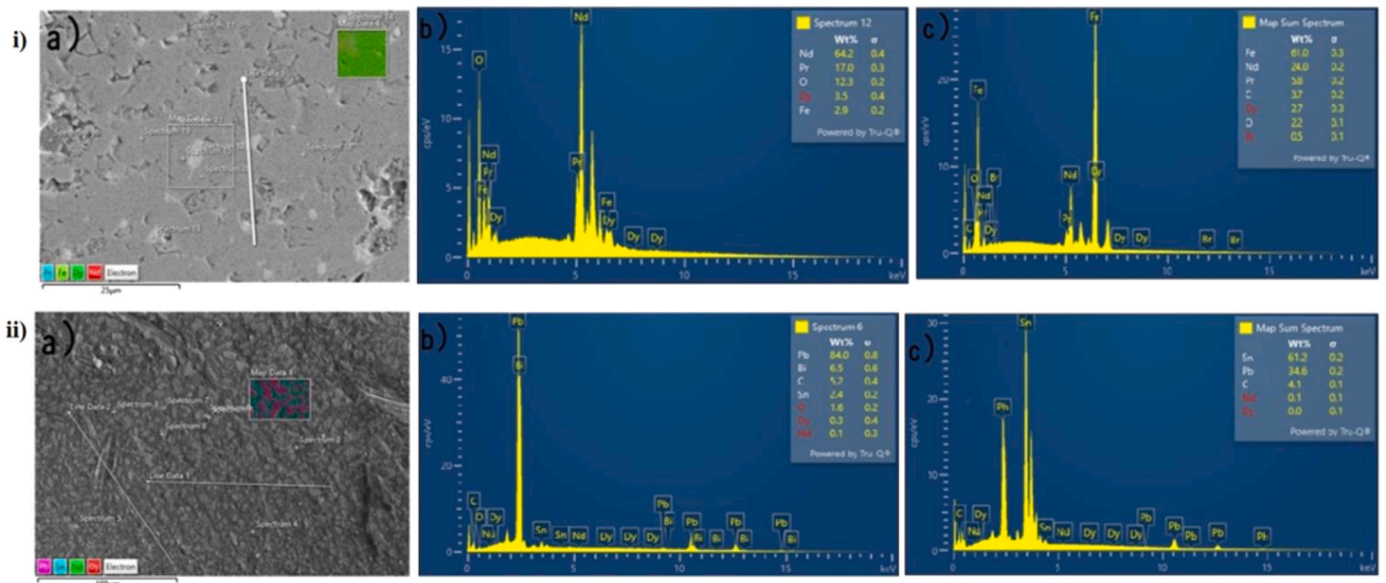
Fig. 3. i) EDS results of the magnet sample-a) showing the selected area on the SEM image b) composition for the square area selected c) composition for the point selected- 3ii) EDS results of the solder joint specimen – a) showing the selected area on the SEM image b) composition for the line data 1c) composition for the line data 2.

due to its rarity and high cost. It is possible that the XRF analysis did not detect the presence of neodymium in the solder joint because the quantity of the element was too low to be detected by the equipment.

Overall, the XRF process has proven to be successful in detecting the presence of REEs in the E-waste components, which is a primary objective of this study. The obtained results for the permanent magnet are consistent with the findings of previous research studies, which validates the accuracy and reliability of the experimental process. However, despite multiple tests, the concentration of rare earths in the solder joint is significantly low, indicating that the addition of neodymium as a microalloying element to improve the wetting and spreading characteristics of the solder is not a common practice in the manufacturing of the electronic devices from which the E-waste was obtained.

3.3. SEMh and EDS results

Fig. 3i displays the EDS image captured by the SEM of the magnet. The SEM offers various scanning modes such as point, line, and map scans. As depicted in the figure, 20 distinct scans were performed to obtain a variety of outcomes. Additionally, Fig. 3i exhibits the chart derived from a point scan of the white globule visible in the SEM image. At this point, the maximum values of Nd and Dy were recorded (64.2 % and 3.5 % by wt., respectively, with negligible error margins of <1The EDS image in Fig. 3i illustrates that the white globules on the surface of the magnet contain a significant amount of rare earth elements (REEs), particularly neodymium (Nd) with a weight percentage of 64.2 % and dysprosium (Dy) with a weight percentage of 3.5 %. Additionally, praseodymium, another REE, was identified with a weight percentage of 17 %. These results suggest that these globules are rich in REEs despite the sample being ground and polished, as oxidation still occurs due to atmospheric oxygen exposure, resulting in the presence of oxides. Fig. 3i also shows that the dominant element in the region is iron (61 % weight), with moderate amounts of REEs such as neodymium (24 %), praseodymium (5.8 %), and dysprosium (2.7 %). The obtained results are credible and accurate because the primary data aligns closely with those provided by an established manufacturer, with minimal margin of error. Additionally, the SEM results also support the XRF values obtained, further validating the accuracy of the experiment.

Fig. 3ii shows the EDS image taken of the solder joint sample, which

Table 3

Rare Earth Elements recovery methods extracted from literature review [17–23].

Method to recover REE	Advantages	Disadvantages
Pyrometallurgical Processes	Less hazardous waste produced in comparison to hydrometallurgical Increased reaction rates due to high temperatures Efficient at recovering substantial amount of REE	High energy consumption due to operating temperatures of 800–1000 C High initial investment
Hydrometallurgical processes	No atmospheric SO2 emissions	Toxic and hazardous by products produced Large quantities of water required High operation cost Ammonium bicarbonate is used in the process to precipitate REE, difficult to treat the wastewater
Electrochemical Processes	Less energy intensive and reduced chemical consumption Low cost and fast Can use renewable energy to achieve a sustainable approach	Expensive Electrolyte may lead to corrosion of apparatus Large production areas Only suitable for mass production work
Separation by Crystallization and Precipitation	High purification can be obtained quickly Operates at a lower energy	Only purifies one component at a time Yield is limited by phase equilibria
Diffusion Dialysis	Energy efficient Clean process as it does not involve addition of chemicals Small space requirement	High membrane cost Low processing capability & processing efficiency

has a general composition that contrasts with that of the magnet. Multiple scans were conducted to investigate the presence of REE throughout the sample, and the results of a point scan are also shown in the figure. At this particular point, lead (Pb) was the most dominant element, with a weight percentage of 84. A minute amount of REE was detected, with Nd having a weight percentage of 0.1 % and Dy having a weight percentage of 0.3 %. The figure also shows the map scan taken of
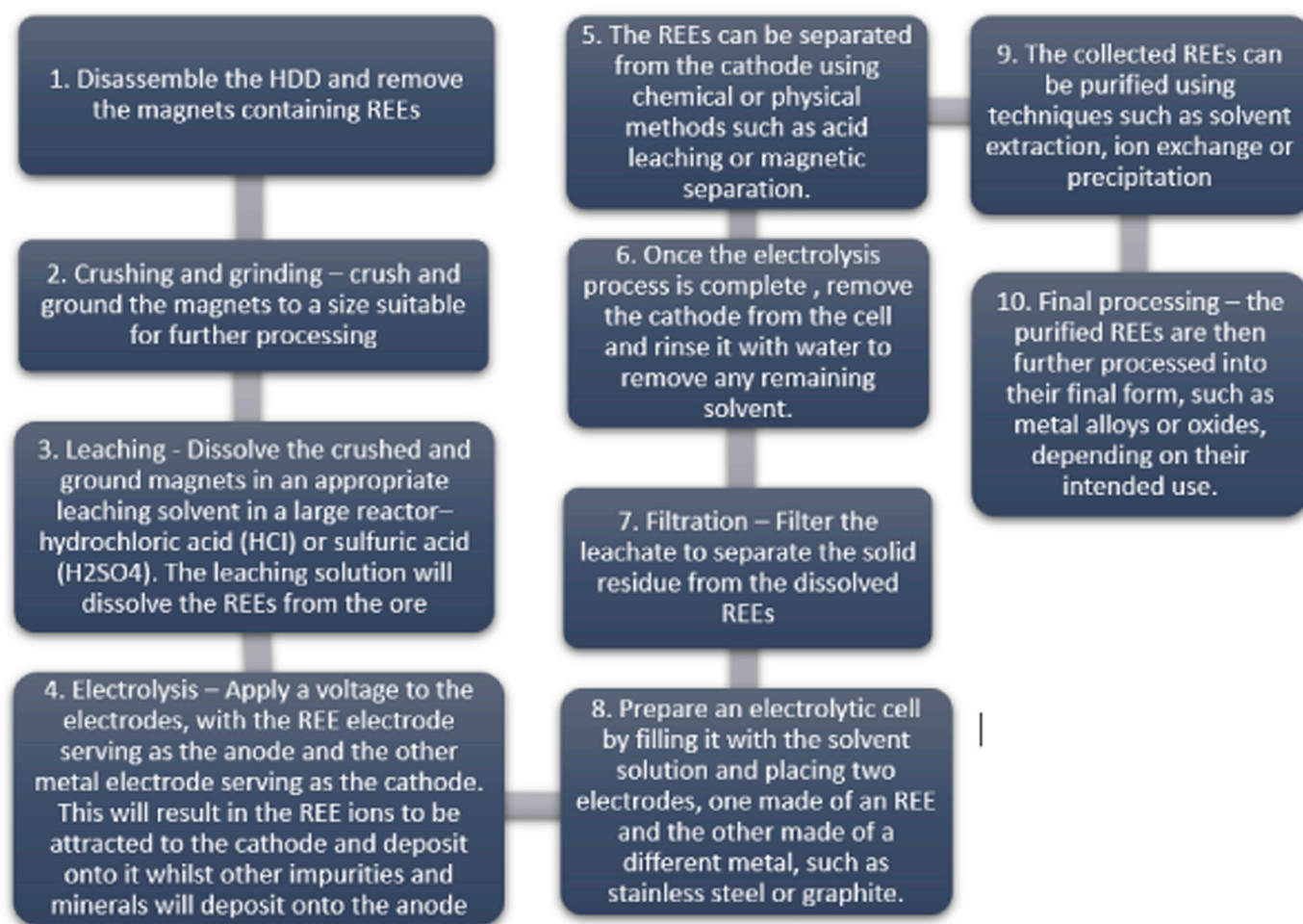
Fig. 4. Flow chart for the steps in extraction of Rare earth elements identified from the e waste starting from disassembling, separation, extraction and processing.

the solder, which once again shows almost no REE detected. This may be because the REE quantity present is below the resolution of the technology. The general composition of the solder consists of tin (Sn – 61.2 %) and lead (Pb – 34.6 %).

Indeed, the SEM with EDS has been a valuable tool for the composition analysis in this study. By providing magnified inspection of the samples, it was able to identify significant REE presence within the magnet, whereas the solder contained negligible amounts. This understanding of where REE is present is crucial in assessing whether it is feasible to sustainably recycle the component.

### 3.4. Extraction methods of REE

One of the principal objectives of this study was to determine the most feasible and viable process to recover REE from E-waste on a large-scale. China currently supplies approximately 90 % of the worlds REE, China's decision to tighten export quotas have increased the global interest to sustainably recover their own REEs. REE recycling has significant advantages over mining of REE including energy saving, water and chemical consumption and the global interest to become more sustainable. These factors make finding an appropriate large-scale method necessary [16]. An in-depth literature review was conducted on various recovery processes were compared and shown in the Table 3.

The most sustainable approach for extracting rare earth elements (REE) has been determined to be the electrochemical process. Compared to pyrometallurgical methods that involve temperatures as high as 1000 °C, electrochemical processes are less energy intensive. The electrolysis component of the electrochemical process requires electrical

energy, which can be obtained from renewable sources such as wind and solar power. By investing in renewable energy sources, industries can minimize their reliance on limited fossil fuels to power the process, resulting in a lower carbon footprint that is environmentally sustainable. Additionally, the electrochemical method is a clean and rapid process that does not generate any toxic byproducts, as evidenced by research studies [17–20]. Furthermore, the electrochemical method is well-suited for mass production work with large production areas, making it an ideal option for large-scale recovery projects. The process for these methods is illustrated in Fig. 4, while Fig. 5 provides a graphical representation of the extraction of Nd and Dy [9].

Pyrometallurgy is the most common method for metal separation and recovery in e-waste, with smelting being the preferred approach for over 70 % of waste PCBs. However, it comes with challenges such as toxic material generation, difficulty in reclaiming certain metals like aluminum and iron, and partial metal separation [33]. Hydrometallurgical techniques can complement pyrometallurgy for better metal recovery and plastic retrieval. Researchers have also suggested Vacuum Metallurgy Separation (VMS) as a method to recover Bi, Sb, Pb, and other heavy metals [32].

Hydrometallurgical procedures for e-waste involve acid or caustic leaching, followed by separation and purification methods like cementation, solvent extraction, activated carbon, and ion exchange. Acid leaching is commonly used, but the toxicity of cyanide leaching has led to the exploration of alternative chemistries like thiosulfate for precious metals extraction. Bioleaching shows promise as a leaching method, and electrowinning is the preferred approach for copper recovery from the solution after solvent extraction concentrates soluble copper and rejects
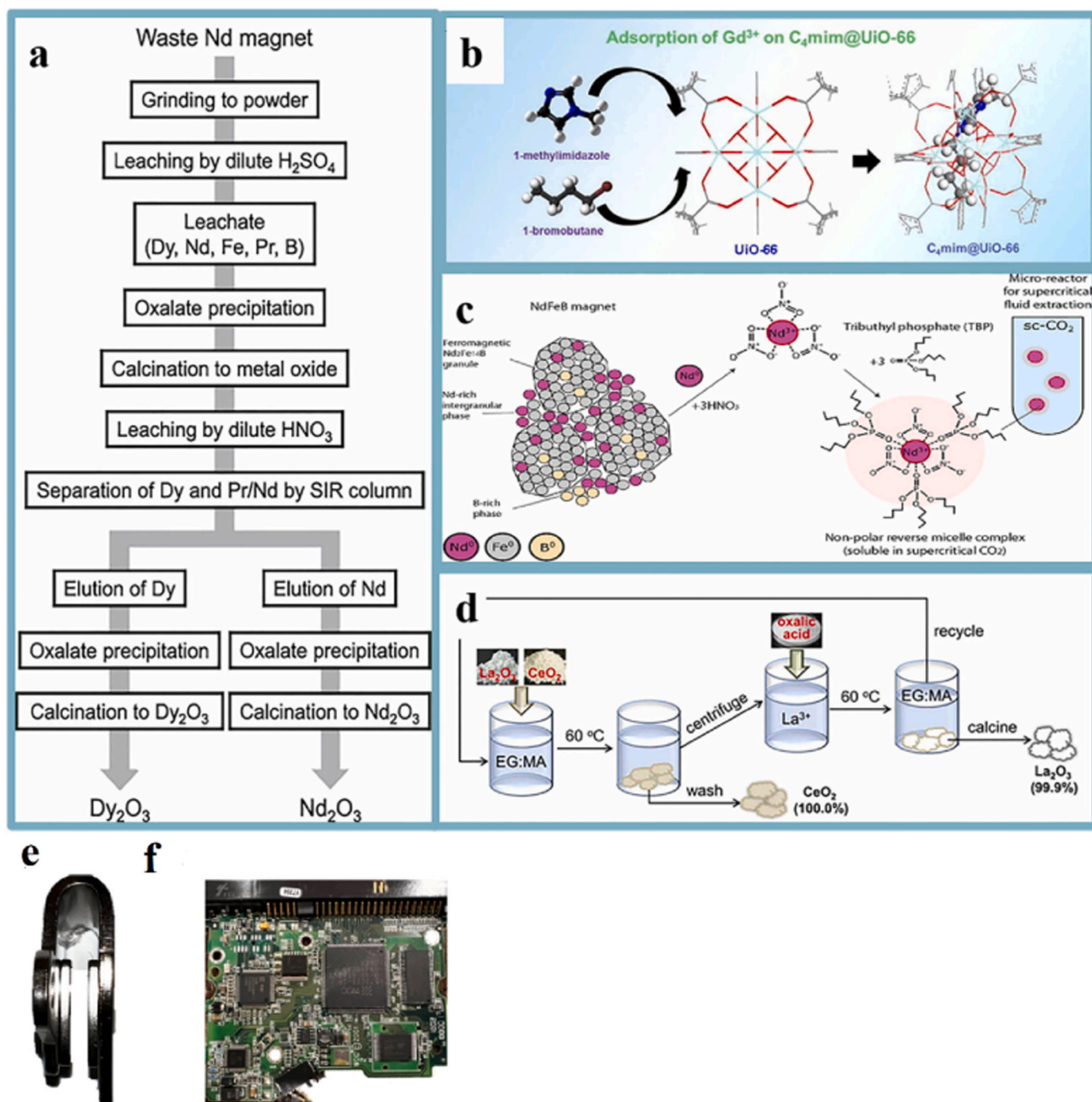
Fig. 5. (a) The comprehensive flowchart outlining the recovery process of Dy and Nd from waste Nd magnets. (b) Utilizing UiO-66 coated with ionic liquid to capture Gd3 + in solution (c) Depicting the model for supercritical carbon dioxide (sc-CO2) extraction (d) A schematic representation of the rare earth oxides separation process [9,27–30] e) Image of the magnet and f) printed circuit board dismantle from the e-waste.

most of the dissolved iron. Researchers have also investigated ammonia and Bronsted acidic ionic liquids as alternative chemistries for metal extraction [34–36].

In addition, Brewster's research explored the feasibility of scaling up the electrochemical process for industrial applications. Her findings suggest that it is possible to manufacture a stack of electrochemical cells, enabling long-term analysis. Moreover, she discovered that the cell voltages can be maintained when scaled up, indicating that the efficiencies achieved in the laboratory could potentially be replicated on a larger scale [24]. Moreover, another study suggests that the electrochemical method for REE recovery has an advantage over other aqueous-based processes. It enables the simultaneous recovery of both rare earth elements (REEs) and non-REEs in a single step, as opposed to other methods which require multiple steps [25]. However, the study also identified a limitation of the electrochemical technique in that the hydrogen evolution reaction can impact process efficiency. This issue can be addressed by carefully selecting a cathode with a low exchange current density [25]. It is worth noting that the specific electrolysis conditions for the electrochemical process may vary depending on the type of rare earth element (REE) being extracted, as well as the properties of the solvent and electrodes used. Another study conducted a composition analysis on 100 units of hard disk drives (HDDs), focusing

on the NdFeB magnets and PCB boards found within them. The analysis revealed that 19 % of the magnet was Neodymium, 72 % was iron, 4 % was Dysprosium, and 4 % was Praseodymium. In addition, the PCBs were found to contain 0.5 % Cerium and 0.2 % Neodymium [26].

4. Conclusion

Electronic waste has rapidly emerged as one of the fastest-growing waste markets globally, including the UK, and other countries worldwide. The objective of this study was to identify Rare Earth Elements (REE) in electronic waste and recommend a sustainable recovery method for their extraction. Through a comprehensive literature survey and dismantling of an electronic waste component, material characterization using XRD, XRF, SEM and EDS, and identification of a sustainable recovery method were achieved. The study focused on the magnet and solder joints within the E-waste component. The results of the EDS and XRF analysis were consistent with established secondary data, indicating the presence of Nd and Dy within the samples. The SEM and XRF results revealed a significant amount of neodymium with a noticeable quantity of dysprosium present in the magnet. The analysis also identified small amounts of REE in the solder alloys, which is a positive result. Based on the literature survey, the electrochemical recovery technique was identified as the most efficient method for REE extraction and recovery. This technique has a high efficiency rate, allowing for quick extraction of the majority of REEs within components, making it suitable for large-scale use. Overall, this study contributes to the growing body of knowledge on sustainable REE recovery from E-waste. For future works, other bioleaching methods could be identified and examine the suitability for REE recovery. Also, a comparison between all the available process could also give a good output for the area of REE recovery from e waste.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

Acknowledgements

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