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Recycling of SmCo Magnets by Removal of Iron via Oxidative Leaching

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Abstract

In the production of SmCo permanent magnets with excellent temperature stability, corrosion resistance, and oxidation resistance, samarium (Sm), one of the rare earth elements (REEs), and cobalt (Co) are employed. Cobalt (Co) is a crucial component in tool materials, nickel-based alloys, tablet and smartphone batteries, and electric car batteries. REEs and Co have been listed as critical raw materials by the European Union Commission for many years. Due to the ever-growing demand for Co and REEs in technological applications, the recovery of these elements from secondary sources has garnered significant interest. There are two types of SmCo magnets, one of which contains a high amount of iron, approximately 15.2%. This paper focuses on the recycling of Fe-bearing SmCo. In this study, an oxidative leaching process with nitric acid was developed to eliminate iron through in situ hydrolysis and to dissolve REEs and Co. The influence of experimental conditions on the formation of an amorphous iron compound through the hydrolysis of Fe^{3+} in a nitric acid environment was thoroughly examined based on a Taguchi orthogonal array. The optimal parameters for oxidative leaching were determined to be an acid concentration of 3 mol/L, a solid-to-liquid ratio of 1/10, and a process temperature of 60 °C.

Keywords Recycling · Recovery · Hydrometallurgy · Leaching · Rare earth elements

1 Introduction

Rare earth elements (REEs) possess superior magnetic, optical, and electrical properties that cannot be replicated, and they play a pivotal role in enhancing product performance and diversifying product offerings. China currently holds a monopoly on the supply of rare earth elements [1–3]. Cobalt (Co) has emerged as a vital component in electric vehicles, tablet and smartphone batteries, nickel-based alloys, and tool materials. It is also a crucial element in lithium-ion batteries, constituting 10–20% of the cathode material [4–6]. In 2023, the EU updated its Critical Raw Materials (CRM) list, identifying key elements for the years ahead [7]. With REEs and Co included in the critical raw material list, the recovery of these elements from SmCo magnets is becoming increasingly important to meet the demand for them.

Hydrometallurgy is the most common method for recovering Sm and Co from SmCo magnets in the literature [8, 9]. There are different types of SmCo magnets, one of which includes iron, cobalt, and REEs, while the other includes only cobalt and REEs. SmCo magnets contain a high amount of Sm and a low amount of other REEs. To recover REEs and Co, the magnets are initially leached with inorganic acids such as sulfuric acid, nitric acid, hydrochloric acid, or perchloric acid [10, 11]. In this process, both REEs and Co, as well as Fe, are dissolved and exist in the form of metal ions. Depending on the subsequent processing steps, different acids can be employed. For example, nitric acid or hydrochloric acid are preferred for solvent extraction processes, while sulfuric acid can be used for selective precipitation [12–14]. REEs can be almost completely precipitated as oxalate or sulfate double salts; however, cobalt and iron are precipitated along with REEs, resulting in a low-purity product [15–17]. Several studies have investigated the recycling of SmCo using organic solvents and ionic liquids in detail [15, 18, 19]. The main barrier to their widespread adoption is that many of these processes are highly complex and expensive, making them inefficient for industrial-scale operations [19–21].

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There have been few studies on the recycling of SmCo in the literature. Onoda et al. recycled Sm and Co from a Sm-Co nitrate solution. The pH value of the solution was controlled by adding sodium hydroxide solution and nitric acid. The research showed that when the pH ranges from 2 to 4, Sm can be separated and precipitated as samarium phosphate, while cobalt cations remained in the filtered solution [22]. In another study, Zhou et al. separated Co and Sm from Sm-Co alloy waste based on the differences in the solubility of samarium sulfate and cobalt sulfate in a sulfuric acid solution. They reported that a small amount of Sm was dissolved in the sulfuric acid solution due to the low solubility of samarium sulfate. The optimal leaching parameters were determined to be a liquid–solid ratio of 6:1, a leaching temperature of 60 °C, a leaching time of 75 min, and an H_2SO_4 concentration of 4 mol/L. Under these conditions, Co can be completely extracted in the leach liquor, while Sm can be extracted to about 24.1%. A high amount of Sm was separated as leach residue. Subsequently, the small amount of Sm in the leach liquor was separated by employing Na_2SO_4. In the proposed flowsheet, 93.4% of Sm was successfully recovered under the specified conditions [14]. There are no existing studies on the detailed examination of Fe-bearing SmCo magnets in the literature.

Herein, we propose a novel method for recycling Fe-bearing SmCo magnets. An oxidative leaching process has been developed for the first time, in which iron is removed from the leach liquor through in situ hydrolysis in a nitric acid medium, resulting in the elimination of iron during the leaching stage and the dissolution of cobalt and REEs into the solution. We explain the underlying mechanism of oxidative leaching through chemical equations. Furthermore, we determine the optimal oxidative leaching parameters using statistical analysis combined with Taguchi plots.

2 Materials and Method

2.1 Characterization of SmCo Magnet Powders

The magnet samples used in this research were provided by Vacuumschmelze GmbH & Co. KG., located in Germany. To begin with, the magnets underwent a crushing process using a jaw crusher (Retsch BB51). Subsequently, the resulting powders were sifted using a vibrating sieve shaker (Retsch, AS200) to obtain powder samples with a particle size of less than 90 µm. The metal concentration of the magnet samples was determined through inductively coupled plasma optical emission spectroscopy (ICP-OES) using the SPECTRO ARCOS instrument from SPECTRO Analytical Instruments GmbH in Kleve, Germany. X-ray diffraction analysis was carried out on the powder samples of SmCo magnets using the X-ray diffractometer (PANalytical EMPYREAN). Morphological investigation and elemental mapping of the magnet powders were performed using scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX) analysis, utilizing the scanning electron microscope, Thermo Fisher Quattro S.

2.2 Oxidative Leaching Experiment

Taguchi orthogonal array design was used for the oxidative leaching experiments. The philosophy of the employing Taguchi design method in this study lies in minimizing the iron dissolution and maximize the extraction of Sm and Co during oxidative leaching by identifying the optimal leaching parameters. The key conditions affecting the leaching process, namely, the effects of concentration of nitric acid temperature, and solid-to-liquid ratio were systematically examined in a controlled environment. Oxidative leaching parameters, along with their levels, are detailed in Table 1.

To initiate this, a pre-determined quantity of the nitric acid solution was introduced into a 500-mL three-necked round-bottom flask, which was placed within a heating mantle. Subsequently, the magnet powders were introduced gradually into the leaching solution while being agitated using a mechanical stirrer operating at a speed of 450 rpm for a duration of 1 h. Upon completion of the leaching experiment, the round-bottom flask was removed from the heating mantle, and the resulting slurry was promptly filtered using a vacuum filtration setup. It is worth noting that due to the exothermic reaction between the SmCo magnet powders and the nitric acid, the temperature of the experiments was closely monitored, and a temperature–time graph was constructed. This graph depicted the leaching temperature on the *y*-axis and the leaching time on the *x*-axis, providing insights into the temperature variations throughout the process.

2.3 Analytic Methods

The metal concentration in the leaching solution was determined through ICP-OES analysis. To characterize the phases, present in both the leach residue and the calcine leach residue, X-ray diffraction (XRD) was recorded between 10 and 90° using Cu-Kα radiation as the X-ray

Table 1 Oxidative leaching parameters and their levels

Parameters	Levels		
	1	2	3
Acid concentration (mol/L)	1	2	3
Solid/liquid ratio (w/v)	1/10	1/20	1/30
Temperature (°C)	20	40	60

source. Furthermore, in addition to structural analysis, the leach residue was characterized using SEM equipped with EDX. The pH values of the leaching solutions were measured using a pH meter (WTW Lab-pH Meter inoLab® pH 7110). Calibration was performed with three pH buffer solutions: pH 4, pH 7, and pH 10. The leaching efficiencies of Sm, Co, and Fe were then calculated based on the metal concentrations obtained from the ICP-OES analysis. The chemical analyses allowed to assess the effectiveness of the leaching process in terms of extracting these specific metals from the initial magnet powders.

3 Results and Discussion

3.1 Characterization of the SmCo Magnet Powders

To propose a flowsheet for recycling of SmCo magnets, a thorough characterization of the magnet powders was conducted. This involved a detailed examination of the chemical composition and morphology of the SmCo magnet powders using SEM coupled EDX analysis.

Figure 1 illustrates the results obtained from the EDX analysis of the SmCo magnet powders. This figure includes an SEM micrograph, a tabulated representation of the chemical composition of the magnet powders, and the EDX spectrum. These data provide valuable insights into the elemental composition and physical structure of the magnet powders, which are crucial for developing an effective recycling process.

The composition analysis of the SmCo magnet indicates the presence of several elements, primarily including Co, Sm, Fe, Cu, Zr, and O. A minor amount of carbon was detected, likely originating from the analytical process. Additionally, the presence of aluminum (Al) can be attributed to the use of an aluminum stub during the SEM analysis.

To ensure the accuracy and reliability of these EDX results, the chemical composition of the SmCo magnet was cross-verified through ICP-OES analysis. Table 2 provides the mass percentages of each metal identified in the SmCo magnets, offering a comprehensive overview of the elemental composition in a quantifiable manner.

The primary elements found in the SmCo magnet are Co, Sm, and Fe, constituting the major components of its composition. In addition to these main elements, there are also minor trace elements present, including Cu, Nd, Yb, and Zr.

3.2 Effect of Oxidative Leaching Parameters

The magnet powders were leached into nitric acid solution in a controlled atmosphere. The oxidative leaching experimental details and leaching efficiencies of Sm, Co, and Fe were given in Table 3.

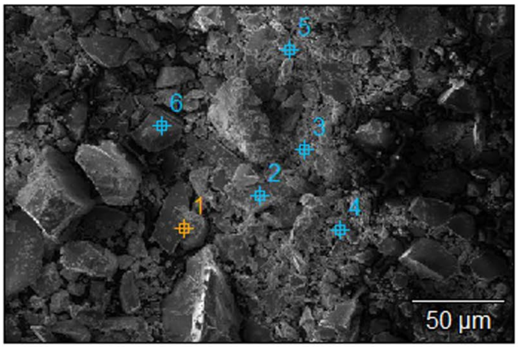
The impact of nitric acid concentration, solid/liquid ratio, and temperature on leaching efficiency was evaluated using Taguchi plots and ANOVA (analysis of variance) analysis. For the creation of these Taguchi plots, the signal-to-noise approach was chosen. For Sm and Co, the "larger is better" approach was selected. Conversely, for Fe, the "smaller is better" approach was chosen as the objective is to minimize the extraction of Fe, while achieving high extractions of Sm and Co. This is consistent with the expectation that oxidative leaching should result in high Sm and Co extractions and low Fe extraction. Figure 2 illustrates the Taguchi plots for Sm, Co, and Fe.

Table 2 Chemical analysis of the SmCo magnets determined by ICP-OES analysis

Elements	Co	Sm	Fe	Nd	Cu	Zr
wt.%	55.0	21.0	15.2	0.8	5.1	2.7

Table 3 The experimental parameters for oxidative leaching and the results of experiments based on Taguchi's orthogonal arrays

Experiment code	Molarity of acid (mol/L)	Solid/liquid ratio (w/v)	Temperature (°C)	Leaching eff. of Sm (%)	Leaching eff. of Co (%)	Leaching eff. of Fe (%)
M1	1	1/10	20	26	28	5
M2	1	1/20	40	56	60	37
M3	1	1/30	60	79	81	62
M4	2	1/10	40	63	66	4
M5	2	1/20	60	98	99	96
M6	2	1/30	20	95	96	96
M7	3	1/10	60	99	99	0
M8	3	1/20	20	92	92	94
M9	3	1/30	40	99	99	99



	C	O	Al	Fe	Co	Cu	Zr	Sm
Point 1	0.517	1.575	-	9.852	35.714	5.355	1.624	45.362
Point 2	0.417	2.702	-	13.091	33.697	4.539	2.132	43.423
Point 3	4.737	6.953	3.480	7.607	39.687	-	-	37.536
Point 4	0.552	2.604	-	12.078	31.214	4.123	1.967	47.463
Point 5	0.978	2.691	0.256	11.226	36.056	4.816	1.939	41.654
Point 6	1.574	2.534	-	17.764	29.988	4.007	2.488	40.950

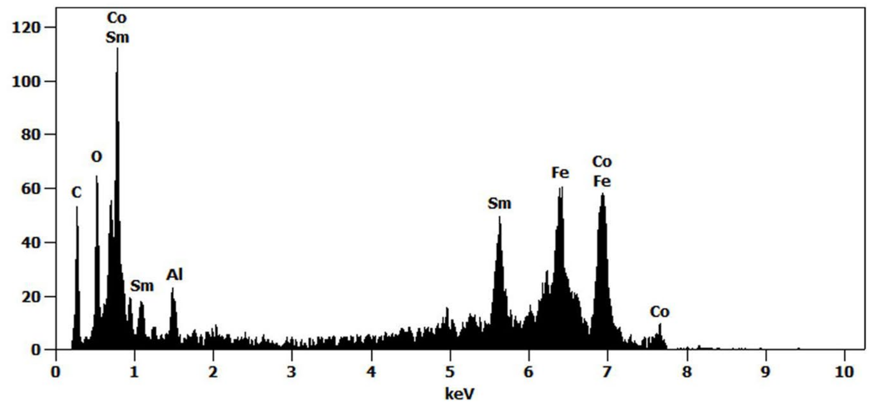


Fig. 1 **a** SEM micrograph, **b** chemical composition, and **c** EDX spectrum of SmCo magnets.

The influence of the experimental parameters on leaching efficiency was assessed through analysis of variance (ANOVA). The *F* and *P* values for each experimental parameter were calculated for Sm, Co extraction, and Fe hydrolysis, and the results are presented in Tables 4, 5, and 6, respectively. When the *P* value is less than 0.05, it indicates that the parameter has a significant effect on oxidative leaching within the selected range of values. R-Sq and $R\text{-}Sq_{(Adj)}$ values were calculated for both metal ions. R-Sq and $R\text{-}Sq_{(Adj)}$ were calculated as 97.63% and 90.51% for Sm, 97.06% and 88.24% for Co, and 95.05% and 80.22% for Fe, respectively.

A new approach has been introduced to address the removal of iron from Fe-bearing SmCo magnets during oxidative leaching. This innovative idea involves selectively leaching cobalt (Co) and samarium (Sm) while avoiding the leaching of iron. To achieve this, nitric acid is used to create highly oxidative conditions specifically targeted at iron, resulting in the hydrolysis of iron to form an amorphous Fe compound. This method appears to be a promising way to separate and retain valuable elements (Sm and Co) while effectively isolating and converting iron into a more manageable form. It showcases a strategic approach to enhance the efficiency of recycling processes for SmCo magnets.

The effect of nitric acid concentration on Sm and Co extraction and Fe hydrolysis was examined. The extraction of Sm and Co increased with increasing acid concentration between 1 and 3 mol/L while the Fe hydrolysis increasing with decreasing acid concentration. A high acid concentration can be effective in promoting the dissolution of various metals. This result highlights the importance of controlling acid concentration to achieve specific objectives in the leaching process. Based on the results of the ANOVA analysis, it appears that the selected range of acid concentrations significantly influences the extraction of Co and Sm while having no significant effect on Fe hydrolysis, as indicated by the analysis conducted at a 95% confidence interval. Similarly, the investigation of the solid-to-liquid ratio in the range of 1/10 to 1/30 revealed that this parameter does not significantly impact the extraction of Sm and Co. However, it does have an influence on Fe hydrolysis, suggesting that variations in the solid-to-liquid ratio can affect the way iron reacts and hydrolyzes during the oxidative leaching process.

The ANOVA results indicate that the most effective parameter for iron hydrolysis is the solid-to-liquid ratio, and the Fe hydrolysis increases with a decreasing solid-to-liquid ratio between 1/10 and 1/30. Based on the ANOVA results, the extraction of Co and Sm, as well as Fe hydrolysis, were not affected by changes in leaching temperatures. The '*P*' values for temperature are 0.131, 0.154, and 0.591 for Sm, Co, and Fe, respectively.

The nitric acid leaching of SmCo magnets was highly exothermic, and initially, the leaching temperature could not

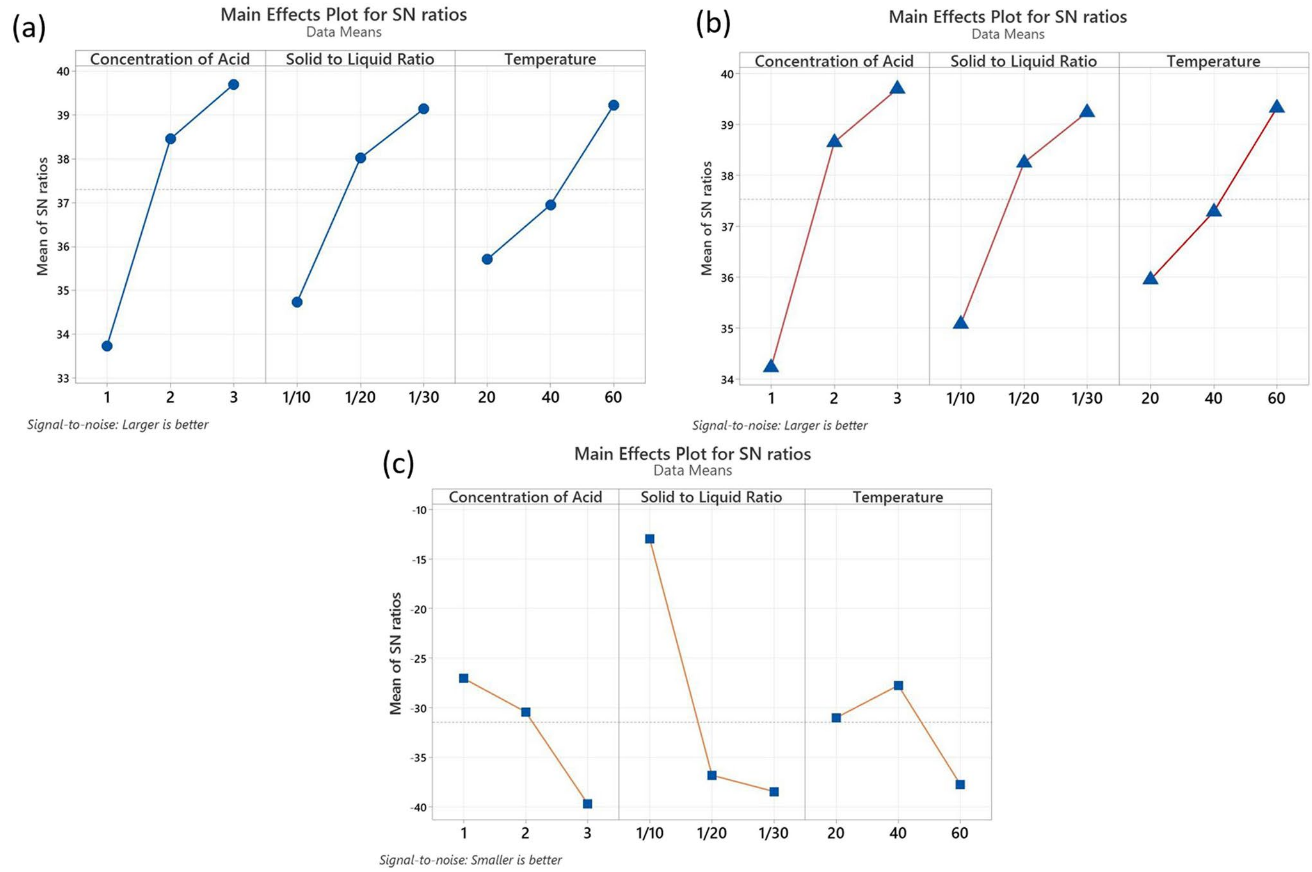


Fig. 2 Taguchi Plots for **a** Sm, **b** Co, and **c** Fe

Table 4 ANOVA analysis of the Sm extraction

Parameters	DF	Adj SS	Adj MS	*F*-value	*P*-value
Concentration of acid	2	2980.2	1490.11	24.25	0.040
Solid-to-liquid ratio	2	1257.6	628.78	10.23	0.089
Temperature	2	817.6	408.78	6.65	0.131
Error	2	122.9	61.44		
Total	8	5178.2			

Table 5 ANOVA analysis of the Co extraction

Parameters	DF	Adj SS	Adj MS	*F*-value	*P*-value
Concentration of acid	2	2660.7	1330.33	18.91	0.050
Solid-to-liquid ratio	2	1208.7	604.33	8.59	0.104
Temperature	2	774.0	387.00	5.50	0.154
Error	2	140.7	70.33		
Total	8	4784.0			

Table 6 ANOVA analysis of the Fe hydrolysis

Parameters	DF	Adj SS	Adj MS	*F*-value	*P*-value
Concentration of acid	2	1821.6	910.8	2.40	0.294
Solid-to-liquid ratio	2	12,214.2	6107.1	16.12	0.058
Temperature	2	524.2	262.1	0.69	0.591
Error	2	757.6	378.8		
Total	8	15,317.6			

be controlled. After a certain duration, the leaching temperature became stable. Despite the difficulty in selecting optimal conditions for both eliminating Fe from the system and achieving high Sm and Co extraction, based on Taguchi plots and ANOVA analyses, the optimal parameters for oxidative leaching were determined to be an acid concentration of 3 mol/L, a solid-to-liquid ratio of 1/10, and a process temperature of 60 °C. These conditions are consistent with the experimental conditions of sample M7.

3.3 The Underlying Mechanism of Fe Hydrolysis

The temperature and pH value influence Fe hydrolysis. The oxidative leaching temperature for all experiments was measured, and a graph was created, with the *y*-axis representing the oxidative leaching temperature and the x-axis

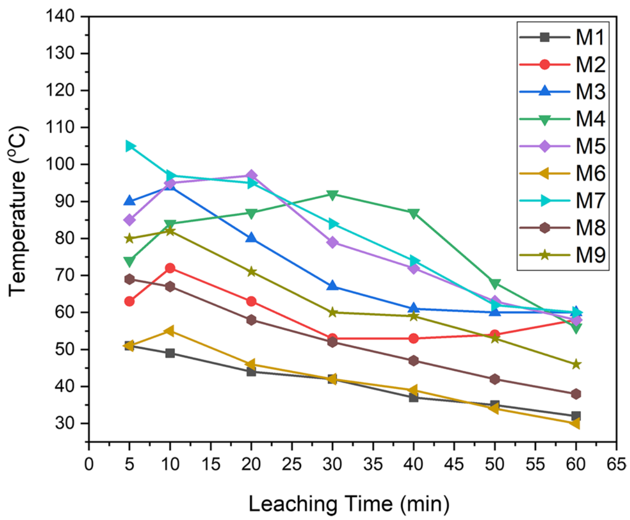


Fig. 3 Oxidative leaching temperature of SmCo magnet powders as a function of time

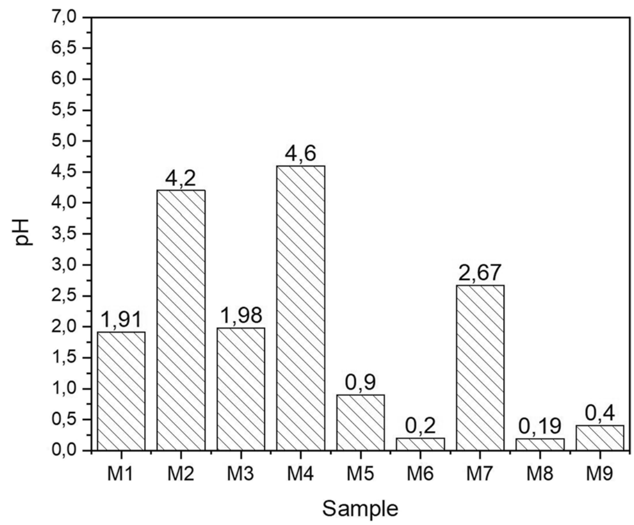


Fig. 4 pH of the leaching solutions.

representing the leaching time. Figure 3 illustrates the oxidative leaching temperature of SmCo magnet powders as a function of time.

It is expected that Fe ions leached at high temperatures mainly hydrolyze to form iron-containing compounds [23, 24]. The formation of goethite is favored by the elevated reaction temperature [25]. If the reaction occurs at a lower temperature, the formation of $Fe(OH)_{3(solid)}$ is expected. As can be seen in Fig. 3, the oxidative leaching of SmCo magnets with nitric acid is highly exothermic, with the leaching temperature of sample M7 starting at approximately 107 °C. Initially, the temperatures of the samples could not be controlled; however, after a certain duration, the leaching temperature became stable.

The pH values also influence iron hydrolysis, with relatively high pH values favoring Fe hydrolysis. Therefore, the pH values of the leaching experiments were measured and are presented in Fig. 4.

At lower acid concentrations, pH values were found to be higher, as expected. At higher acid concentrations, pH values were generally lower, except for sample coded M7. In the case of sample coded M7, the high leaching temperature and solid-to-liquid ratio might have led to the consumption of nitric acid. This observation is supported by Fig. 4 and the chemical analysis results of the leaching solutions, which show that the Fe content in the leach liquor increases as the solution becomes more acidic, except for sample coded M7.

It is expected that the Fe ions leached from the SmCo magnets at high temperatures mainly undergo hydrolysis to form iron compounds. In this study, the Fe hydrolysis under oxidative leaching conditions for Fe-bearing SmCo magnets was investigated. This investigation aims to understand the elimination of Fe through hydrolysis during the leaching process, as it appears to be a straightforward method for recycling SmCo magnets. The Fe hydrolysis reaction equations that could potentially occur in oxidative leaching are expressed as follows [26]:

$$O_{2(g)} \rightleftharpoons O_{2(aq)} \tag{1}$$

$$Fe^{2+} + 2H_2O = Fe(OH)_2 + H_2 \tag{2}$$

$$Fe(OH)_2 + \frac{1}{4}O_2 + \frac{1}{2}H_2O = Fe(OH)_{3(solid)} \tag{3}$$

$$Fe(OH)_{4^-(aq)} = \alpha - FeOOH_{(solid)} + H_2O + OH^- \tag{4}$$

Based on the reactions mentioned above, the selective leaching process for Sm and Co can be achieved because the Fe compound can be separated as a leach residue. To elucidate the structure and chemical composition of the leach residue, a detailed analysis was conducted.

Figure 5 presents the XRD patterns of the leach residue, and the calcined leach residue obtained from sample coded M7.

Under oxidative conditions, an amorphous iron compound was formed. To confirm that the leach residue primarily consists of iron phases, it was calcined at 800 °C for 3 h.

As shown in Fig. 5, the leach residue exhibited structures corresponding to Fe_2O_3 and Fe_3O_4, which are in accordance with 01–089-0598 and 01–089-0951, respectively. The diffraction pattern did not reveal any characteristic peaks of Sm and Co, implying selective leaching of Sm and Co under oxidative conditions.

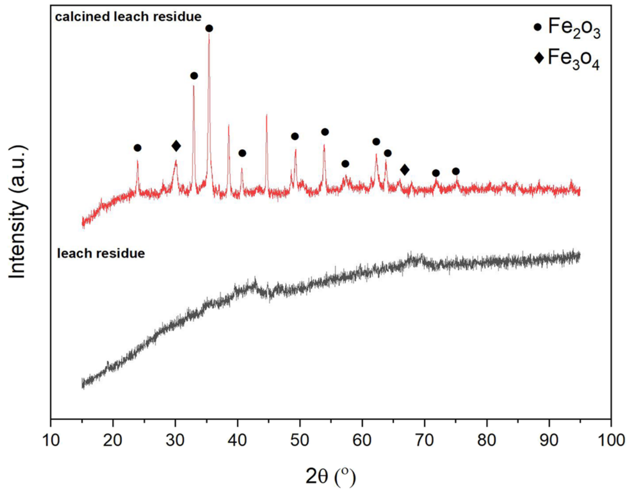


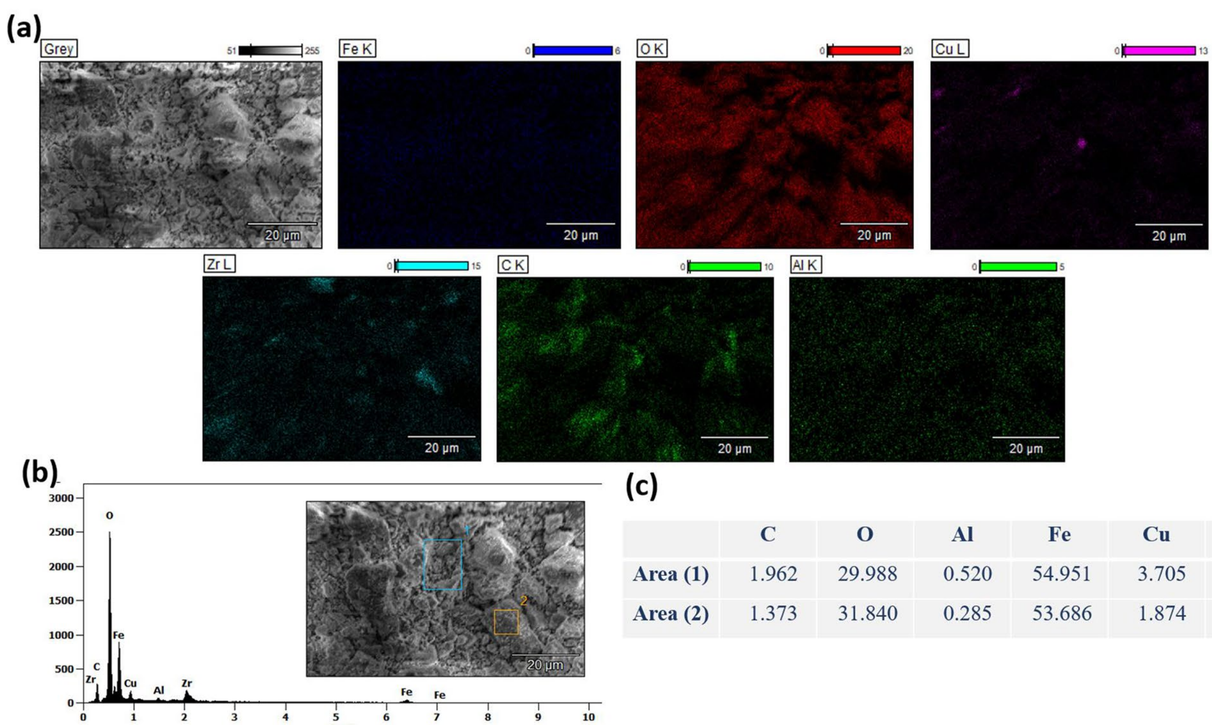
Fig. 5 XRD patterns of the calcined leach residue and leach residue obtained from sample coded M7

Elemental mapping of the leach residue was conducted using SEM equipped with EDX analysis. Figure 6 includes (a) the elemental mapping of the leach residue, (b) the EDX spectrum, and (c) the chemical composition of selected areas in the SEM micrograph.

As illustrated in Fig. 6, the leach residue contains Fe, O, Zr, and Cu. A small amount of Al was observed, due to the use of an Al stub in the analysis. After the oxidative leaching, Sm and Co were not observed in the leach residue.

4 Conclusion

This paper thoroughly investigates the behavior of iron during the oxidative leaching of Fe-bearing SmCo magnets. The research demonstrates that iron ions hydrolyze to form an amorphous iron compound during oxidative leaching, allowing for the successful separation of Sm and Co from Fe in a single step. Within the investigated experimental conditions, the range of nitric acid concentration affects the extraction of Sm and Co, but it does not significantly influence Fe hydrolysis, as confirmed by a 95% confidence interval analysis. While the solid-to-liquid ratio does not impact the extraction of Sm and Co, it does affect Fe hydrolysis. Statistical results reveal that the solid-to-liquid ratio plays a significant role in Fe hydrolysis. Under the specified conditions of acid concentration at 3 mol/L, temperature at 60 °C, and a solid-to-liquid ratio of 1/10, Sm and Co extractions reached approximately 99%. Furthermore, Fe was effectively eliminated from the system through Fe hydrolysis during oxidative leaching under these conditions. In summary, this proposed oxidative leaching method for eliminating Fe from Fe-bearing SmCo magnets and achieving high Sm and Co extraction in a single step is promising. The findings from this research provide valuable insights into the Fe hydrolysis mechanism, laying the foundation for a selective leaching process for Sm and Co.



	C	O	Al	Fe	Cu	Zr
Area (1)	1.962	29.988	0.520	54.951	3.705	8.874
Area (2)	1.373	31.840	0.285	53.686	1.874	10.943

Fig. 6 **a** The elemental mapping of the leach residue, **b** EDX spectrum, and **c** chemical composition of the selected areas in the SEM micrograph.

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Data Availability Data supporting this study are included within the article and/or supporting materials.

Declarations

Conflict of interest The authors declare no competing interests.

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