




Recycling of Yttrium and Europium from Microwave-Roasted Waste Cathode Ray Tube Phosphor Powder

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Waste cathode ray tubes (CRT) containing a high quantity of yttrium and europium have great potential for the resource circulation of rare earths through an efficient recycling process. This study examined how the removal of zinc from a CRT that had been microwave-roasted at various temperatures (600–800°C) affected the various mineral phases of rare earths. The parameters like acid medium, concentration, temperature, and time were varied to yield about 99% leaching efficiency using 2.0 mol/L HCl at 5% pulp density for 60 min of leaching performed at 90°C. The apparent activation energy values ($E_{a(Y)}$, 22.1 kJ/mol, and $E_{a(Eu)}$, 16.7 kJ/mol) show that the leaching follows an intermediate-controlled mechanism by following the logarithmic rate law. Finally, the mixed oxalate precipitation at the stoichiometric ratio of RE- $E^{3+}:C_2O_4^{2-} = 1:1.5$ gave high-purity (Y,Eu) $(C_2O_4)_3$ salt.

INTRODUCTION

Cathode-ray tubes (CRT) containing one or more electron guns have been widely used as picture tubes in television sets and computer monitors as well as in memory devices.¹ In the recent past, the use of CRT has been superseded by new technological devices like liquid crystal display (LCD), light-emitting diode (LED), and plasma display technology.² As a result, their bulk disposal in the form of waste electrical and electronic equipment (WEEE) has gained momentum. As estimated, China generates 2400 million units of CRT,³ while 50,000–150,000 tons of CRT are collected every year in Europe,⁴ and it accounts for 58% of the total regulated e-waste in the US.⁵ Among the bulky components of waste CRT, other than funnel glass, panel glass is a major fraction.³ This panel glass remains covered by a luminescent powder that produces images by excitation with an electron

beam.⁶ The luminescent powder usually consists of rare earth elements (REEs) such as red phosphor (Y₂O₃ or Y₂O₂S:Eu³⁺) along with green and blue colors producing ZnS:Cu and ZnS:Ag, respectively, and thin Al film as an electroconductive substance.⁷ Therefore, the recycling of waste CRT, in particular panel glass, has greater importance for yttrium and europium recovery given their technological relevance, high economic values, and negative supply in the commodity market.^{8–10} Although many papers have focused on REE extraction by the recycling of fluorescent lamp phosphor waste,^{11–13} less attention is paid to REE recovery from CRT waste phosphor.

The literature survey reveals that the high-temperature incineration of CRT waste has been used in practice for melting glass and sending the residual mass for landfilling, which also creates severe environmental issues.¹⁴ However, for the recycling of REE values, hydrometallurgical routes are widely explored,⁶ with acid leaching at the forefront for dissolving REEs in the acid solutions.^{15,16} Due to the presence of sulfide substances, leaching is commonly required to use high

(Received August 22, 2023; accepted October 19, 2023;
published online November 9, 2023)

concentrations of acids, which cause large effluent generation and gaseous emissions, including H_2S .¹⁷ Tian et al.³ used $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ leaching to control H_2S emissions. Subsequently, zinc was recovered as ZnS in another study by Innocenzi et al.¹⁸ Forte et al.⁶ applied roasting at 850–1000°C before leaching to convert the sulfide into oxide compounds, which was followed by two-step leaching in acetic acid and methanesulfonic acid solutions. However, Dexpert-Ghys et al.⁷ used NaOH/NaClO roasting to aim for selective dissolution of zinc in water, and then thermal treatment was given to the residue at 1300–1400°C to obtain $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$. Alternatively, Pan et al.¹⁹ used NH_4Cl roasting to leach out YCl_3 and EuCl_3 in water, whereas Onal and Binnemans⁵ applied sulfation roasting in the presence of ZnSO_4 to leach REEs in water.

In the aforementioned studies, roasting has been found to be a bottleneck due to being an energy-intensive and time-consuming process, which has an adverse effect on the leaching and selectivity of REE extraction. Therefore, we applied microwave-assisted roasting, which has been found to be favorable to sulfide minerals compared to silicates,²⁰ thus allowing all zinc to be converted to leachable ZnO and ZnSO_4 at neutral pH (5.5 using dil. H_2SO_4) and leaving REEs in the residual form. However, REEs also form different compounds that can have an effect on their subsequent leaching. In this study, we report the leaching behavior of water-leached residue after microwave roasting of CRT powder at different temperatures, with an emphasis on REEs' dissolution kinetics and mechanism.

EXPERIMENTAL

The CRT phosphor powder of size – 100 mesh (as supplied by a local recycler in India) was used for microwave roasting in a Milestone PYRO Advance at different temperatures between 600°C and 800°C for 15-min duration (after reaching the desired temperature) under the conditions defined elsewhere.²⁰ Notably, the composition of the CRT phosphor powder that underwent to the microwave-assisted roasting process at different temperatures was 26.4% Zn, 12.6% Y, 8.2% Si, 1.05% Al, 1.08% Eu, and 14.6% of S. After cooling down to room temperature, the roasted mass was used for neutral leaching at pH 5.5 (maintained by adding 10% H_2SO_4) and a pulp density (PD) of 5% for 2 h; > 99% zinc could be leached in all cases, and the residues were collected after filtration and drying at 80°C for 6 h. These residues were characterized for their REE contents by a wet-chemical digestion process and analyzed using inductively coupled plasma spectroscopy (ICP-OES, Thermo Scientific). The REEs in residual samples were determined to be 21.1% Y and 1.8% Eu for roasting at 600°C, 21.63% Y, and 1.86% Eu for roasting at 700°C and 18.9% Y and 1.7% Eu for roasting at

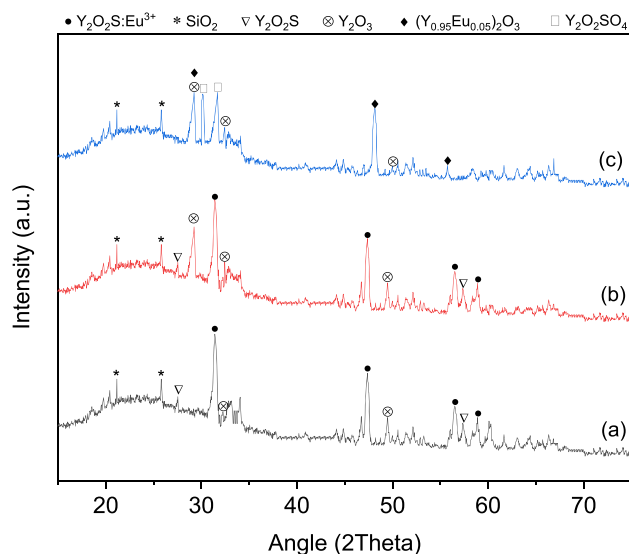


Fig. 1. XRD patterns of the residues after neutral leaching (pH 5.5) of the microwave roasted CRT powder at different temperatures: (a) 600°C, (b) 700°C, and (c) 800°C.

800°C. The x-ray diffraction (XRD) patterns acquired for the samples are shown in Fig. 1, showing the presence of different phases of REEs in the residues caused by the effect of temperature in microwave roasting of CRT powder. As can be seen the mineral phases of $\text{Y}_2\text{O}_2\text{S}:\text{Eu}^{3+}$ [JCPDS file no. 24-1428], SiO_2 [ICDD file no. 083-1833], $\text{Y}_2\text{O}_2\text{S}$ [JCPDS file no. 24-1424], and Y_2O_3 [JCPDS file no. 01-71-5970] were prominently observed with the residue of roasting at 600°C, whereas the residual sample of roasting at 700°C showed that the formation of Y_2O_3 phase increased with the dominant existence of $\text{Y}_2\text{O}_2\text{S}:\text{Eu}^{3+}$ that could be converted to $\text{Y}_2\text{O}_2\text{SO}_4$ (JCPDS file no. 53-168) and $(\text{Y}_{0.96}\text{Eu}_{0.04})_2\text{O}_3$ (ICDD file no. 025-1011) when roasted in microwave at a higher temperature of 800°C. These residues were used in this study to examine the leaching behavior of REEs in acid solutions.

All the leaching experiments were conducted in a 250-mL two-neck flat-bottom flask fitted with a condenser in the middle. The heating was supplied externally using the hot plate, while stirring was provided by a magnetic paddle at a constant mixing speed of 250 rpm; 5 g of sample and 100 mL of the prepared acid solution of desired concentration were taken into the leaching flask and stirred for 1 h at a pre-decided temperature (variable in the range of 30–90°C). Notably, to determine the kinetics, experiments were carried out for a particular duration each time at a particular temperature. Then, the slurry was filtered to separately collect the filtrate it to analyze the concentration of REEs therein in the leach liquor using ICP-OES. Furthermore, the leaching efficiencies of yttrium and europium were calculated as follows:

$$\% \text{ Leaching} = \frac{m_o}{m_{in}} \times 100 \quad (1)$$

where m_{in} and m_o are the metal content (in grams) present in the input sample before leaching and in the output leach liquor, respectively.

RESULTS AND DISCUSSION

Effect of Acid Media

The role of acid media on yttrium and europium leaching from Zn-depleted CRT powder was studied with three different acid solutions of HCl, H₂SO₄, and HNO₃, each of 1.0 mol/L concentration. Results presented in Fig. 2 show that the leaching trend of REEs increased regarding the roasting temperature in all acids used. This behavior can be explained by the variation in mineral phases of REEs during roasting at different temperatures. As Fig. 2 shows, sulfidic phases were present at temperatures of 600°C and 700°C, which could be eliminated by forming the soluble oxides and oxysulfate phases at 800°C. On the other hand, the maximum leaching efficiency from each temperature's residue could be observed with HNO₃ that reached > 78% yttrium and 72% europium for the residue of roasting at 800°C. The highest leaching with HNO₃ can be corroborated by the higher oxidizing capacity of nitrate ions compared to the chloride and sulfate ions.^{11,12} Using H₂SO₄ and HCl, the leaching efficiency of REEs varied in the range of 53–68% and 59–74%, respectively. However, a slightly higher leaching with HNO₃ was avoided because of the relatively higher toxicity of NO_x compared to other gaseous species of sulfur and chlorine. The use of HCl in further study was chosen because of its better efficiency than when using H₂SO₄. The least efficiency with H₂SO₄ could be correlated with the

probable formation of surface passivation by the sulfur species of REEs in the sulfate media.

Effect of HCl Concentration

The role of acid concentration in REEs' leaching was examined at a varied concentration range of 0.5–2.0 mol/L HCl. Results presented in Fig. 3 show that the leaching trend of REEs increased with higher concentrations of acid used, regardless of the roasting temperature of the samples. As can be seen, the leaching of yttrium improved from 54% to 73% and that of europium increased from 50% to 70% by varying the acid concentration from 0.5 mol/L to 2.0 mol/L HCl for the sample roasting at 600°C. The maximum yields of yttrium and europium were 92% and 90%, respectively, for the sample at a roasting temperature of 800°C and leached in a 2.0 mol/L HCl solution compared to 76% and 75% for the sample roasted at 700°C and using the same concentration of acid. This indicates that the incomplete conversion of oxy-sulfides at 600°C and 700°C hinders the leaching process because of the refractoriness of sulfur atoms, whereas the converted oxide and oxysulfate at 800°C favor the leaching process via the reaction, as follows:



Effect of Temperature

The role of temperature on yttrium and europium leaching from Zn-depleted CRT powder was investigated in the range of 30–90°C while keeping other

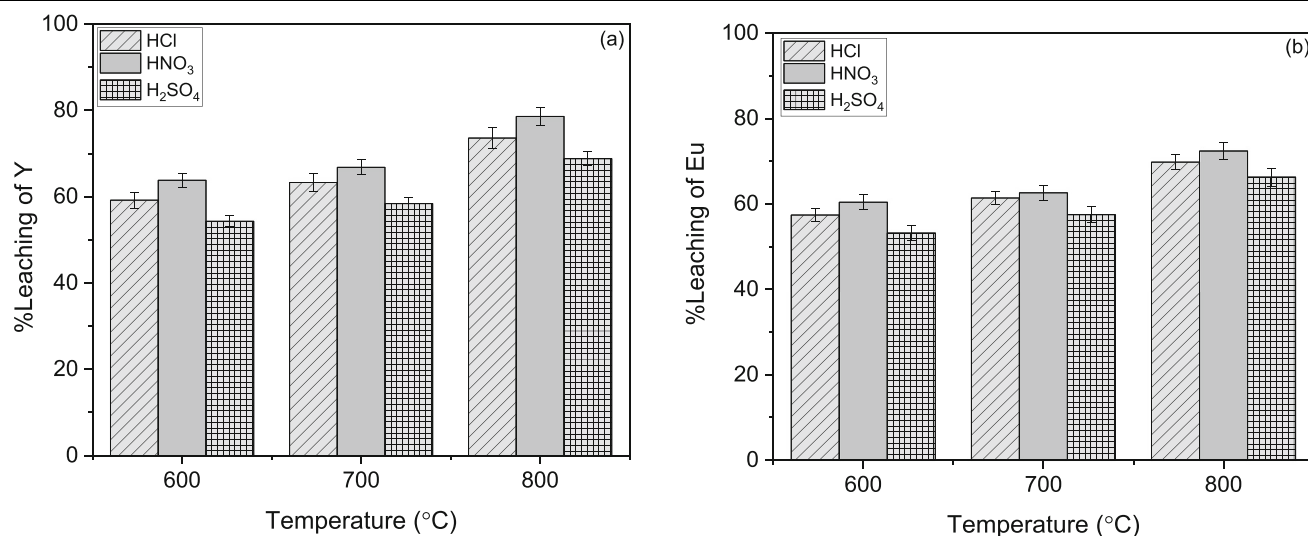


Fig. 2. Leaching efficiency of (a) yttrium and (b) europium from Zn-depleted residues of roasted CRT at different temperatures, while leaching performed in different acid media at a fixed concentration = 1.0 mol/L, temperature = 30°C, PD = 5%, mixing speed = 250 rpm, and time = 1 h.

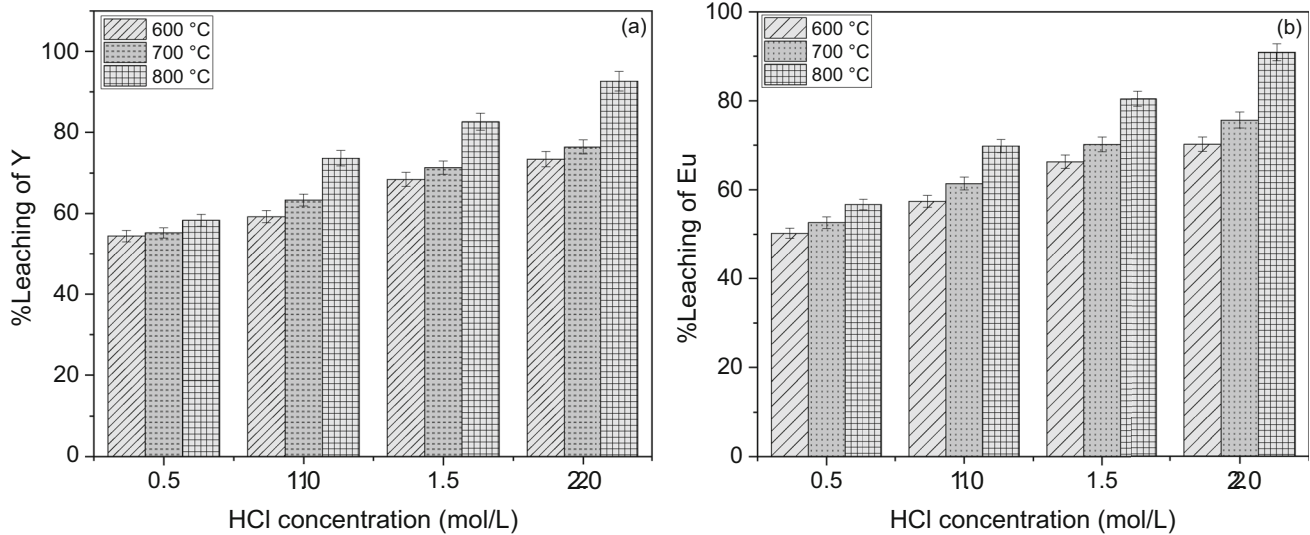


Fig. 3. Leaching efficiency of (a) yttrium and (b) europium from Zn-depleted residues of roasted CRT at different temperatures, while leaching performed in concentrations of HCl solution at temperature = 30°C, PD = 5%, mixing speed = 250 rpm, and time = 1 h.

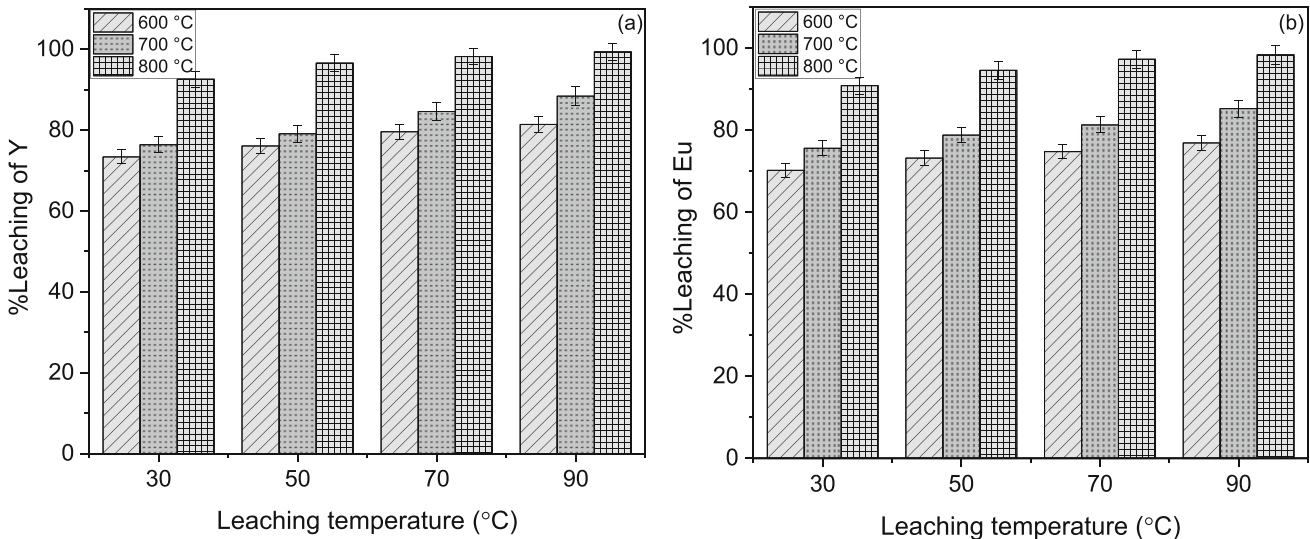


Fig. 4. Leaching efficiency of (a) yttrium and (b) europium from Zn-depleted residues of roasted CRT at different temperatures and leaching performed as a function of temperature by keeping acid concentration = 2.0 mol/L HCl, PD = 5%, mixing speed = 250 rpm, and time = 1 h constant.

parameters constant. Results in Fig. 4 clearly show progress in the leaching efficiency of yttrium and europium by raising the temperature from room temperature (30°C) to 90°C. The effect of leaching temperature was more prominent for the sample roasted at a higher temperature (800°C), reaching about 99% efficiency for both rare earths by leaching performed at 90°C compared to around 90% efficiency at 30°C. It clearly indicates that the solubility of REEs' oxide and oxysulfate mineral phases formed at a roasting temperature of 800°C is greatly increased in a heated solution. Leaching of the samples roasted at lower temperatures, i.e., 600°C and 700°C, also showed an improvement of 8–12% regarding the rise in dissolution temperature, albeit

a little lower than the efficiency achieved for the roasted mass at 800°C. Leaching for the lower temperature roasted mass was observed to be 81% and 88% yttrium and 77% and 85% europium, while the sample used had roasting temperatures of 600°C and 700°C, respectively. Commonly, the progress in REEs' dissolution regarding leaching temperature can be corroborated by the increasing rate of solid-liquid mass transfer.^{21,22}

Kinetics and Mechanism

Looking at the influential role of temperature on REEs' leaching, their dissolution kinetics were evaluated by taking the zinc-depleted sample for roasting at 800°C. For this, leaching was performed

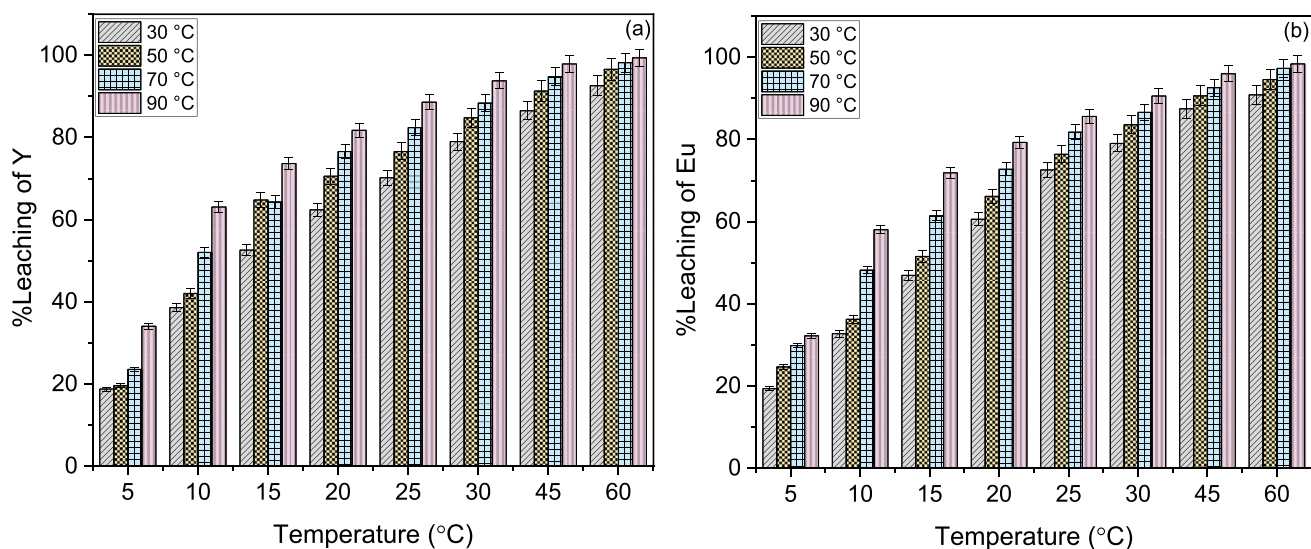


Fig. 5. Leaching efficiency of (a) yttrium and (b) europium from Zn-depleted residues of roasted CRT at 800°C, while leaching performed as a function of time at different temperatures = 30–90°C, acid concentration = 2.0 mol/L HCl, PD = 5%, and mixing speed = 250 rpm constant.

as a function of time (up to 60 min) at all the varied temperatures in the range of 30–90°C. Results in Fig. 5 show the progress of leaching with time at all temperatures. The dissolution of both metals was < 20% at the initial 5 min of leaching at 30°C, which was > 30%, while leaching was performed at 90°C. With the prolonged leaching process for 60 min, the dissolution efficiency reached about 90% at 30°C and > 98% at 90°C.

Furthermore, these data were tested with the commonly applied heterogeneous mass transfer model of the shrinking core model with chemically controlled and diffusion-controlled models, expressed as:^{21,22}

$$1 - (1 - x)^{1/3} = k_c t \quad (4)$$

$$1 - 3(1 - x)^{2/3} + 2(1 - x) = k_d t \quad (5)$$

where x = leaching fraction at certain time, t in min; k_c = chemically controlled rate constant; k_d = diffusion-controlled rate constant. However, the data fitted to these models and then, the logarithmic rate law was examined, expressed as:^{23,24}

$$-\ln(1 - x) = k_1 t \quad (6)$$

where k_1 = the logarithmic rate constant. The plots of $-\ln(1-x)$ versus t in Fig. 6a and b depicted linear fittings with R^2 value > 0.98 as shown in Table I. This could reveal that the leaching of yttrium and europium from zinc-depleted roasted CRT essentially relies on the real-time experimental conditions instead of the hypothetical models assuming the shape and size of a particular type, which actually differ from each other in their appearance.²³

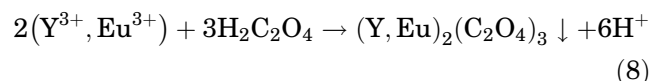
Furthermore, the obtained slope values were used to determine the apparent activation energy (E_a) using the Arrhenius equation, expressed as:^{21,22}

$$k_r = A e^{\left(\frac{-E_a}{RT}\right)} \quad (7)$$

where k_r = rate constant, A = Arrhenius constant, E_a = apparent activation energy, R = universal gas constant (8.314 kJ/mol), and T = absolute temperature (in K). Using the slope values from Fig. 6a and b as the rate constants, the values of the resultant linear plots of $\ln k_r$ versus $1/T$ (shown in Fig. 6c) were further put in Eq. 7 to calculate the apparent activation energy of yttrium to be 22.1 kJ/mol and europium to be 16.7 kJ/mol. The obtained values between > 12.5 kJ/mol and < 41.5 kJ/mol indicate that the leaching process was governed by intermediate control.²⁵ This can be explained by the slow rate of chemical reaction at low temperatures, which gets accelerated with a temperature rise.²¹ In other words, it shows that the lixiviant solution first diffused at the interface through the particle surface to reach REEs oxide and oxysulfate minerals to perform the chemical reactions following Eqs. 2 and 3, thus making yttrium and europium leach out into the bulk solution.

Co-precipitation Recovery of REE Intermediate

The high affinity of REE complexation with oxalate ions with very low solubility limit (K_{sp} values 1×10^{-26} – 1.23×10^{-29})²⁶ often leads their recovery mainly as intermediate oxalate salts. The co-precipitation reaction can be commonly written as:²⁷



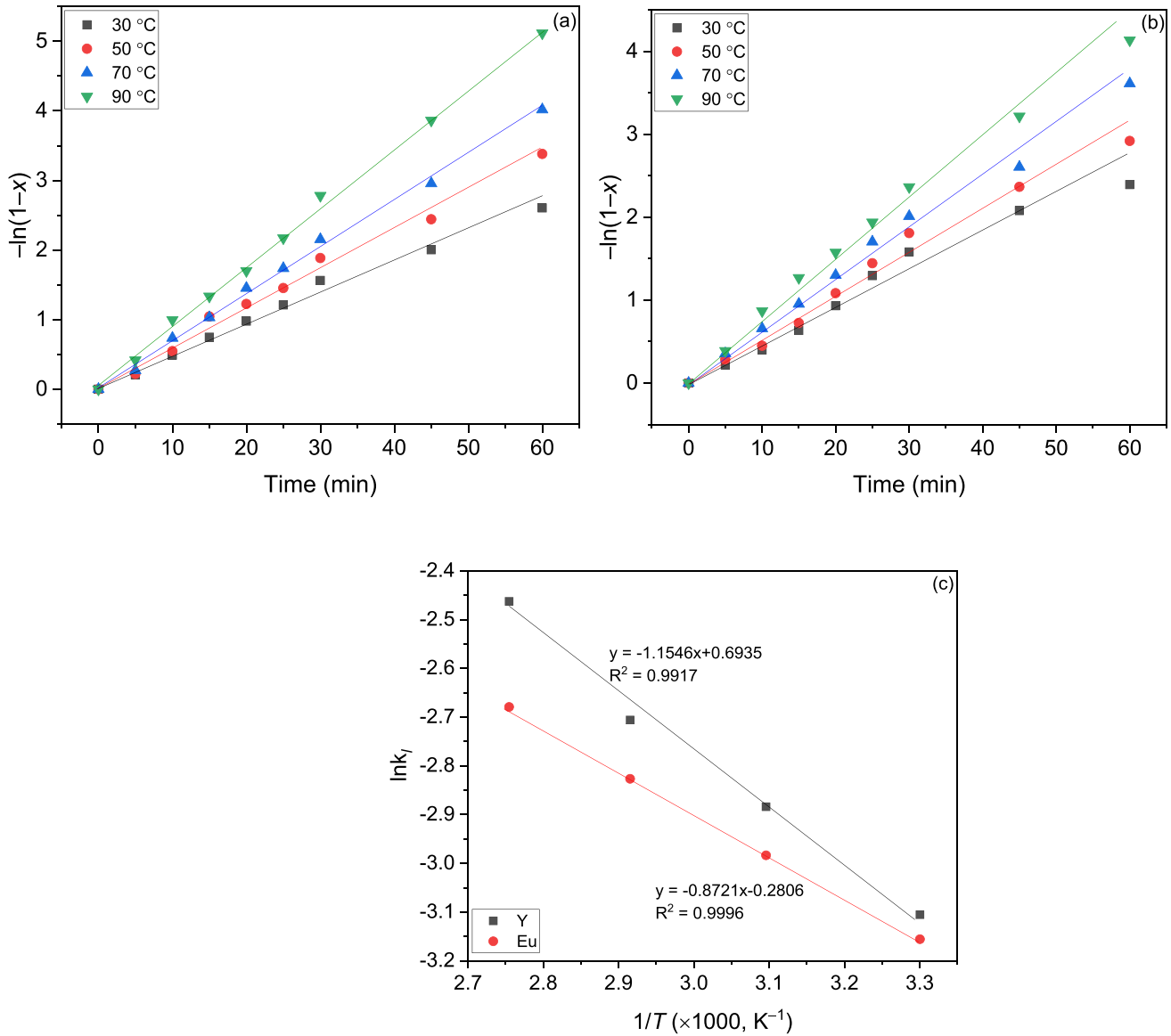


Fig. 6. Plots of logarithmic model, i.e., $-\ln(1-x)$ versus time for (a) yttrium and (b) europium from Zn-depleted residues of roasted CRT at 800°C, while leaching performed as a function of time at different temperatures = 30–90°C, acid concentration = 2.0 mol/L HCl, PD = 5%, and mixing speed = 250 rpm constant. (c) The Arrhenius plot of $\ln k_f$ versus $1/T$ for the leaching of yttrium and europium.

Table I. Obtained slope values and regression coefficient values obtained by the straight-line equations of the linear fitting of the logarithmic kinetic model

Temperature (°C)	Yttrium leaching		Europium leaching	
	Slope (k) values	R^2 values	Slope (k) values	R^2 values
30	0.0448	0.989	0.0426	0.988
50	0.0559	0.994	0.0506	0.997
70	0.0668	0.982	0.0592	0.989
90	0.0852	0.980	0.0686	0.989

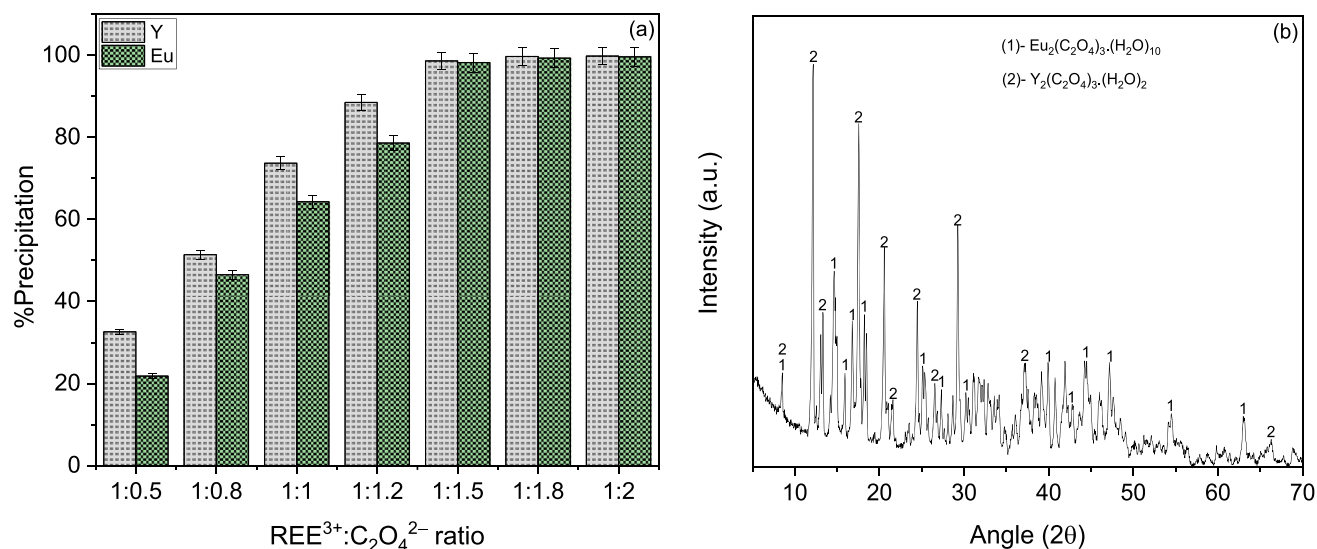


Fig. 7. (a) Precipitation behavior of yttrium and europium as a function of oxalate ion addition regarding the stoichiometric requirement. (b) XRD patterns of co-precipitated oxalate salt of yttrium and europium.

Hence, the co-precipitation was performed with different concentrations of oxalic acid as the stoichiometric ratio of $\text{REE}^{3+}:\text{C}_2\text{O}_4^{2-}$ from 1:0.5 to 1:2 at $\text{pH} \sim 0.5$ and time 30 min. As Fig. 7a shows, both REEs exhibited a more or less similar tendency to precipitate with oxalic acid that reached about 99% by adding 50% more precipitant than the required quantity of stoichiometric ratio. The XRD analysis of the precipitate (as shown in Fig. 7b) reveals the formation of oxalate salt mixture containing decahydrate of europium (JCPDS file no. 04-025-4675) and dihydrate of yttrium (JCPDS file no. 24-1419). This oxalate salt mixture of Y and Eu can be used as intermediate for many application industries.

CONCLUSIONS

This study examined how the removal of zinc from a CRT that had been microwave-roasted at various temperatures (600–800°C) affected the various mineral phases of rare earths. When the Zn-depleted residue from roasting at 800°C was leached at 90°C, 99% of both rare earths were dissolved because the REE-sulfide was completely changed into its oxide and oxysulfate mineral phases. On the other hand, the samples from lower-temperature roasting at 600°C and 700°C could be corroborated by the incomplete conversion of rare earth sulfides into the leachable phases. The kinetics data fitted well with the logarithmic model indicated for intermediate-controlled mechanisms, with the values of apparent activation energy determined to be $E_{a(\text{Y})} = 22.1$ kJ/mol and $E_{a(\text{Eu})} = 16.7$ kJ/mol. Finally, both rare earths were co-precipitated by adding oxalic acid to the leached solution at the stoichiometric ratio of $\text{REE}^{3+}:\text{C}_2\text{O}_4^{2-} = 1:1.5$, which was analyzed to form a mixture of $(\text{Y,Eu})_2(\text{C}_2\text{O}_4)_3$ with different water molecules attached to the oxalate salts. This

study offers two-fold advantages: securing the secondary supply of REEs through resource recovery from waste material and reducing environmental stress through waste treatment.

ACKNOWLEDGEMENTS

This work was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education (project no. 2023-00243477) and a grant funded by the Korea Government (MSIT) (no. 2022R1A5A1032539).

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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