Hydrometallurgical Treatment of an Eudialyte Concentrate for Preparation of Rare Earth Carbonate

REE recovery yields of up to 85.5% achieved

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This study was a small part of the EURARE project concerned with the processing of eudialyte concentrates from Greenland and Norra Kärr, Sweden. Eudialyte is a potential rare earth elements (REE) primary resource due to its good solubility in acid, low radioactivity and relatively high REE content. The main challenge is avoiding the formation of silica gel, which is non-filterable when using acid to extract REE. Some methods have been studied to address this issue and, based on previous research, this paper examined a complete hydrometallurgical treatment of eudialyte concentrate to the production of REE carbonate as a preliminary product. Dry digestion with concentrated hydrochloric acid (10 M) and subsequent water leaching of the treated eudialyte concentrate resulted in high REE extraction while avoiding gel formation. Experiments were performed at a small scale to obtain the optimal parameters. After the first two stages, 88.8% REE was leached under the optimal conditions (HCl:concentrate ratio 1.25:1, digestion time 40 min, water:concentrate ratio 2:1, leaching temperature 20–25°C and leaching time 30 min). After obtaining the pregnant leach solution, preliminary removal of impurities by a precipitation method was examined as well. When adjusting the pH to ~4.0 using calcium carbonate, zirconium, aluminium and iron were removed at 99.1%, 90.0% and 53.1%, respectively, with a REE loss of 2.1%. Finally, a pilot plant test was performed to demonstrate the feasibility and recovery performance under optimal parameters. The material balance in the upscaling test was also calculated to offer some references for future industrial application. A REE carbonate containing 30.0% total REE was finally produced, with an overall REE recovery yield of 85.5%.

1. Introduction

Rare earth elements (REE) are known as 'industrial vitamins'. They are vital components in the fields of metallurgy, military, petrochemical, glass, ceramics, agriculture and new materials science (1–2). The main REE sources in nature contain the minerals bastnasite, monazite, loparite and xenotime as well as ion-absorption clays. A set of mature technologies for REE extraction from these minerals have been developed in recent decades (3–8). However, some countries do not possess enough high-grade deposits and have to import or search for alternatives (9). For example, the EURARE project has recently studied the exploitation of promising REE resources in Europe (10). Eudialyte, as an unconventional REE resource, is one of the most potentially economic raw materials for REE production because of the large amounts of these deposits, low radioactivity and high chemical activity (11–13).

Eudialyte is the most prominent index mineral for agpaitic rocks, but the crystal chemistry has great variation (14–18). The accepted formula of eudialyte group minerals in academia is $N_{15}[M(1)]_6[M(2)]_3[M(3)][M(4)]Z_3[Si_{24}O_{72}]O'_4X_2$ with N = Na, Ca, K, Sr, REE, Ba, Mn, H_3O^+; M(1) = Ca, Mn, REE, Na, Sr, Fe; M(2) = Fe, Mn, Na, Zr, Ta, Ti, K, Ba, H_3O^+; M(3, 4) = Si, Nb, Ti, W, Na; Z = Zr, Ti, Nb; O' = O, OH, H_2O; X = H_2O, Cl, F, OH, CO_3^{2-}, SO_4^{2-}, SiO_4^{4-} (14). Many eudialyte mineral deposits have been reported around the world, which include the Lovozero deposit in Russia, Ilímaussaq complex in South Greenland, Mont Saint-Hilaire deposit in Canada, Norra Kärr alkaline complex in Sweden, Paschim Banga nepheline complex in India and Pajarito deposit in New Mexico (19–24). The REE content in these deposits is 1–3%, including a high heavy REE (HREE) proportion, at a little over 50% (15). Owing to its great compositional variability and sensitivity to hydrothermal re-equilibration, some portion of eudialyte is altered, forming a suite of secondary minerals, including catapleiite (Na-Zr silicate), aegirine, pectolite, analcime, alkalifeldspar, nacareniobsite-(Ce) (REE-Nb silicate) and britholite (Ca-REE-silicates) (25). For example, the Kringlerne eudialyte concentrate contains britholite along with catapleiite.

To date, research on REE extraction from eudialyte has been mainly confined to laboratory studies and hydrometallurgical treatments, especially acid decomposition which has been extensively studied. Despite being easily dissolved by acids, the main challenge with this processing has been how to avoid silica gel formation, which is a gelatinous and non-filterable phase (26–29). Lebedev *et al.* have studied a two-stage decomposition process for REE recovery. High temperature leaching with concentrated sulfuric acid (H_2SO_4) was used to decompose the eudialyte. Then, following washing of the slurry with sodium sulfate solution, the REE remained in the insoluble residue as double sulfate salts. The residue was then washed again and REE recovered after converting the sulfates into nitrates or chlorides. However, a large excess of acid was required here to prevent silica gel formation (27). Another idea for avoiding gel formation is the introduction of sodium fluoride, because fluoride ions (F^-) can promote the coagulation of silica acid in solution such that silica content in the pregnant would be lower and the slurry thus filterable (30). F^- can promote eudialyte decomposition, but it is also toxic.

Recently, Davris *et al.* have reported that a digestion treatment at 100°C (called a 'fuming'

process) successfully prevents silica gel formation. REE are transferred to solution in the subsequent leaching process with water. However, the high temperature makes the processing more complicated and increases the extraction cost (31). Furthermore, Vossenkaul *et al.* have studied hydrometallurgical processing of eudialyte bearing concentrates to recover REE *via* a low temperature dry digestion. Highly concentrated acids without external heating are used in this dry digestion, which also avoids silica gel formation. The pasty material is subsequently water leached, achieving high REE recoveries (29).

Both strategies above favoured hydrochloric acid (HCl) over H_2SO_4 in REE recoveries. The low temperature digestion studied by Vossenkaul mainly focuses on the mechanism of suppressing silica gel formation. HCl at 10 M has been found to be a good choice, resulting in high REE extraction and preventing gel formation. Furthermore, some parameters have been studied in our group using experimental design and neural network modelling (32), but a complete processing treatment to yield a REE carbonate product has not been available. Also, more information regarding the pilot scale test needed to be determined, such as the structural materials of the reactors and material balance, and some unfavourable phenomena during the test need to be taken into account. In view of these conditions, this paper focuses on describing the whole process. Further optimisation and an upscaling test were performed to verify the process feasibility and observe REE behaviour. Finally, another aim of this study was to produce a REE carbonate for further treatment.

2. Materials and Methods

In this study a eudialyte concentrate from TANBREEZ ore was the raw material. TANBREEZ ore was exploited on southern Greenland, with the name TANBREEZ being an acronym of tantalum (Ta), niobium (Nb), REE and zirconium oxide (Z). After mining, the ore was sent for crushing and beneficiation, in which eudialyte concentrate was separated through a partly magnetic selection. The chemical composition and particle distribution of the eudialyte concentrate used in laboratory conditions are shown in **Table I** and **Figures 1** and **2**.

ALS Metallurgy (Australia) conducted the compositional analysis, using X-ray fluorescence (XRF) analysis and REE analysed in solution after dissolution. The solution elements were measured by inductively coupled plasma mass spectrometry

Table I Chemical Composition of Eudialyte Concentrate from TANBREEZ Ore

Elements	Si	Al	Fe	Ca	Na	K	Mg	Ti	Mn	Nb	Zr	TREE[a]
wt%	23.10	3.20	6.04	5.70	9.86	0.80	0.02	0.21	0.30	0.06	6.46	1.60

[a]TREE: total rare earth elements
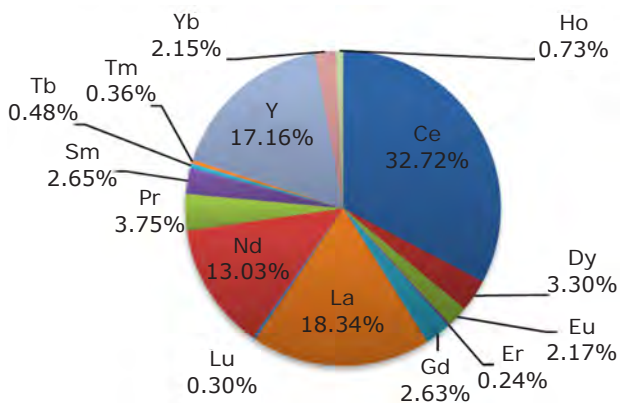
Fig. 1. Distribution of REE in eudialyte concentrate

Fig. 2. Particle size distribution of the eudialyte concentrate

(ICP-MS). The mineral composition and microstructure were determined by quantitative evaluation of materials by scanning electron microscopy (QEMSCAN).

The mineral distribution in the eudialyte concentrate illustrated by false colour imaging is shown in **Figure 3** and the phase quantification is summarised in **Figure 4**. Eudialyte a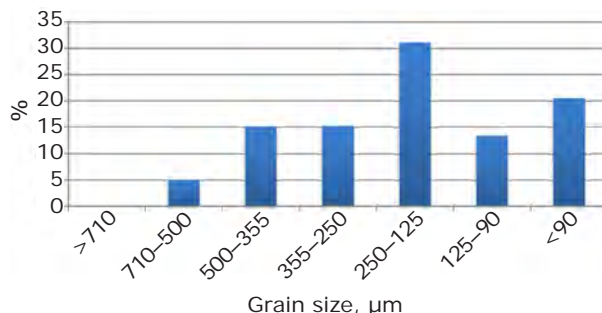ccounted for 67.05% of material in the eudialyte concentrate and the concentrate contained some other silicate minerals, such as arfvedsonite, nepheline and feldspar.

3. Research Procedure

3.1 Experimental Procedure in Laboratory

The flowchart for the treatment of eudialyte concentrate is shown in **Figure 5**. The mass of
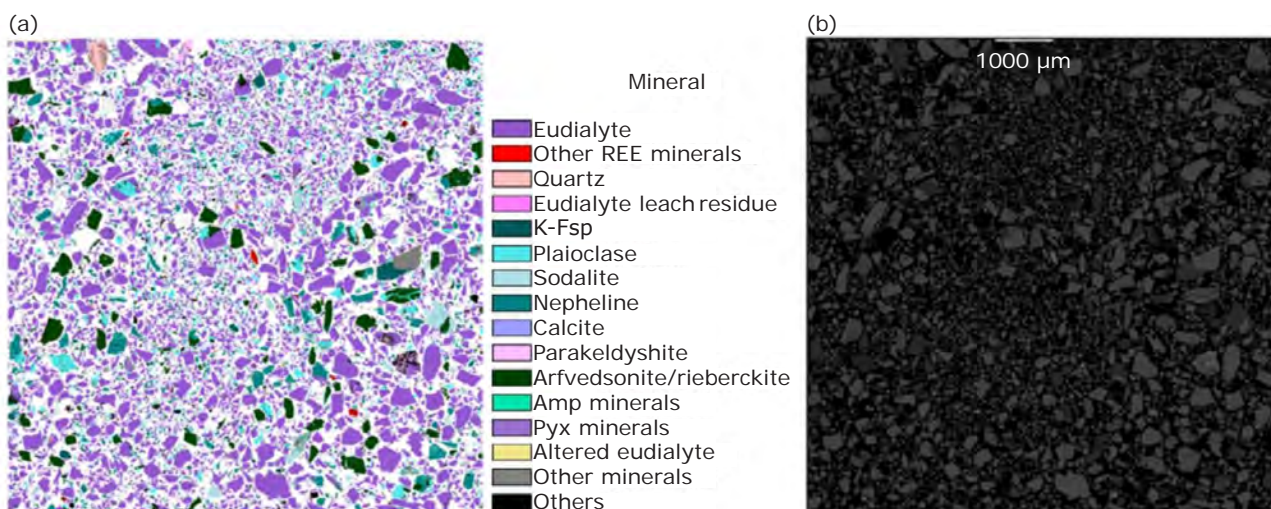

Fig. 3. QEMSCAN analysis of eudialyte concentrate: (a) false colour image after evaluation (K-Fsp: orthoclase and microcline); (b) initial BSE-image
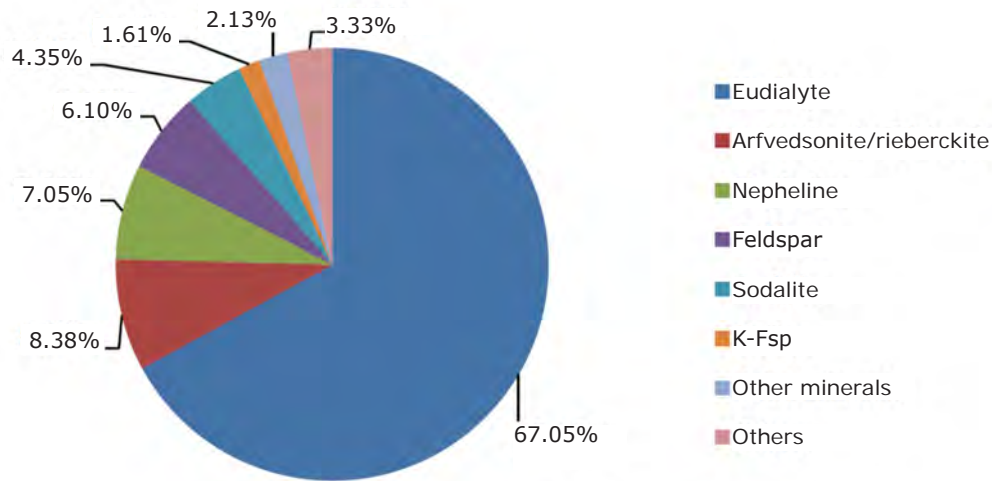
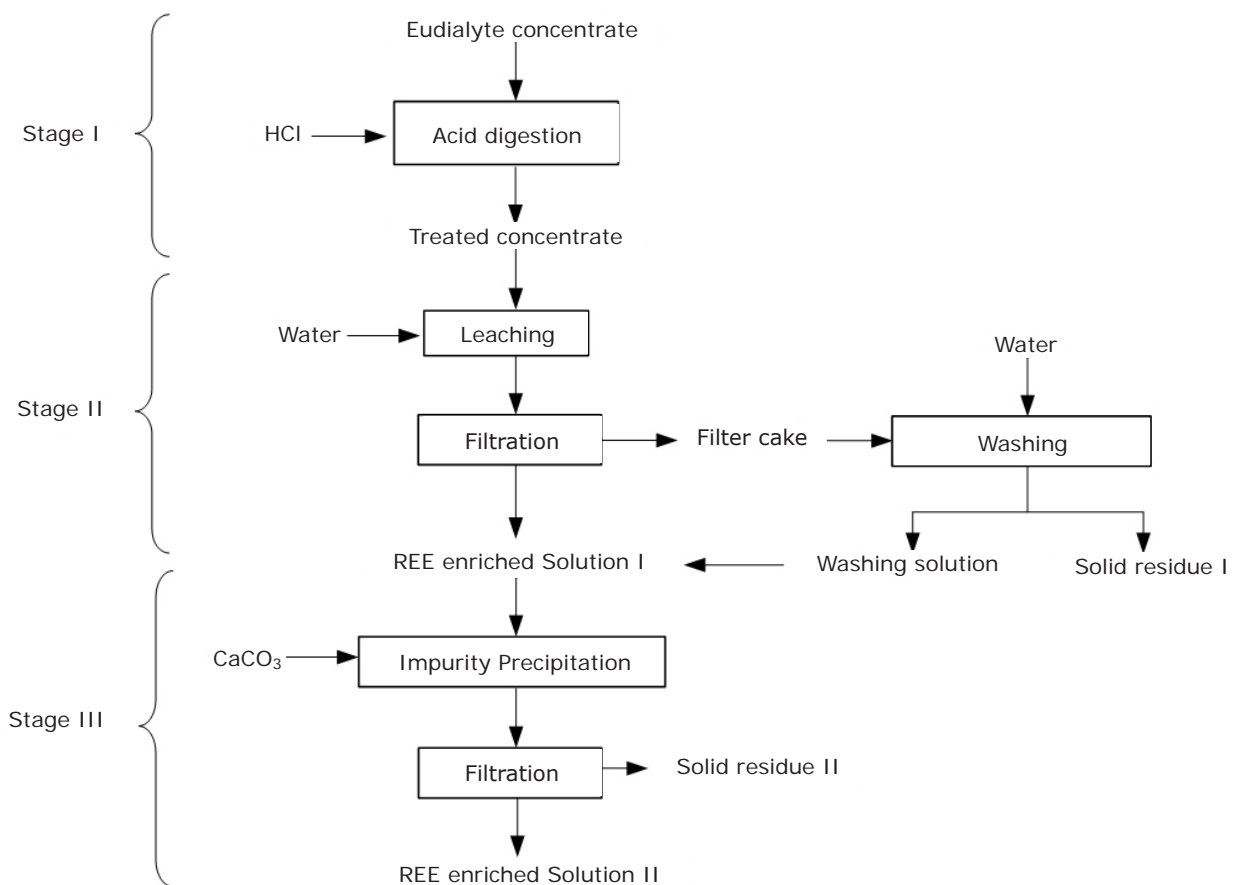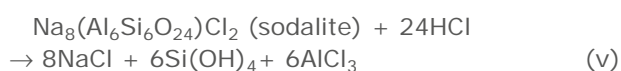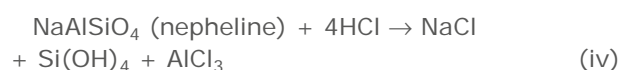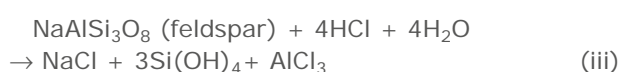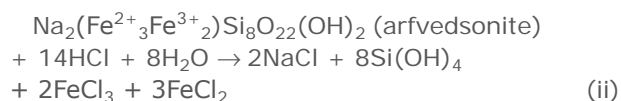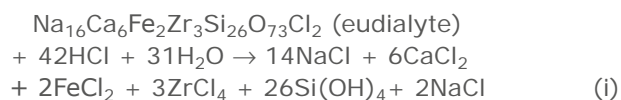Fig. 4. Mineral distribution in eudialyte concentrate

Fig. 5. Proposed flowchart of treatment for REE extraction from eudialyte concentrate

eudialyte concentrate used in each small-scale experiment was 500 g. The first step was the mixing of the eudialyte concentrate with 10 M HCl (dry digestion) to decompose the concentrate and form metal chloride salts and a silica precipitate, thus avoiding silica gel formation. The setup for the digestion process is shown in **Figure 6**. The reactions can be expressed as per Equations (i)–(vi):

$$Na_{16}Ca_6Fe_2Zr_3Si_{26}O_{73}Cl_2 \text{ (eudialyte)} + 42HCl + 31H_2O \rightarrow 14NaCl + 6CaCl_2 + 2FeCl_2 + 3ZrCl_4 + 26Si(OH)_4 + 2NaCl \quad (i)$$

$$Na_2(Fe^{2+}_3Fe^{3+}_2)Si_8O_{22}(OH)_2 \text{ (arfvedsonite)} + 14HCl + 8H_2O \rightarrow 2NaCl + 8Si(OH)_4 + 2FeCl_3 + 3FeCl_2 \quad (ii)$$

$$NaAlSi_3O_8 \text{ (feldspar)} + 4HCl + 4H_2O \rightarrow NaCl + 3Si(OH)_4 + AlCl_3 \quad (iii)$$

$$NaAlSiO_4 \text{ (nepheline)} + 4HCl \rightarrow NaCl + Si(OH)_4 + AlCl_3 \quad (iv)$$

$$Na_8(Al_6Si_6O_{24})Cl_2 \text{ (sodalite)} + 24HCl \rightarrow 8NaCl + 6Si(OH)_4 + 6AlCl_3 \quad (v)$$

$$Si(OH)_{4(aq)} \rightarrow SiO_{2(s)} + 2H_2O_{(l)} \quad (vi)$$

After dry digestion, an injection of water led to further leaching of REE. The extraction efficiency was increased by washing the filter cake after filtration, thus obtaining a leaching solution and solid residue. At this point, Stages I and II in **Figure 5** were complete. In Stage III, some common impurities, such as Fe, Al and Zr, were removed by adding $CaCO_3$ to neutralise the pregnant leach solution. High removal efficiency and low REE loss was achieved by controlling the pH. After filtration, a REE enriched Solution II was obtained.

3.2 Upscaling Test

Results from laboratory procedures were validated at one demonstration plant (**Figure 7**) at the IME, RWTH Aachen and a test under the determined optimal process parameters could also provide data references for industrial applications. It should be noted that only one filtration with a filter press was applied after the neutralisation process in the test, such that the solid residues I and II shown in **Figure 5** were combined.

Two glass reactors (40 l each) were used for the dry digestion process (**Figure 8**). These reactors were of borosilicate glass, which has a high corrosion resistance, a smooth and nonporous surface and poses no environmental risk. Each reactor was equipped with a stirrer with an acceleration torque of up to 330 N m and made of either polyvinylidene fluoride or polypropylene, both of which possessed high chemical and abrasion resistance; the speed of the stirrer was adjustable *via* a control knob. The equipment (**Figure 9**) used after dry digestion

Fig. 6. Setup for dry digestion in laboratory

Fig. 7. Demonstration pilot plant at the IME Process Metallurgy and Metal Recycling, RWTH Aachen, Germany

Fig. 8. Dry digestion reactors and filter press at the pilot plant

Fig. 9. Sketch of equipment for leaching, neutralisation and filtration at the pilot plant

processing included a leaching unit (2 × 100 l), neutralisation unit (3 × 10 l and 1 × 8.5 l), stirred collecting tank (250 l) and separation unit (filter press). After leaching and neutralisation, the slurry was collected into the large stirred collecting tank (250 l) and the small filter press (MFP 300, Andritz Separation, Andritz AG, Austria) used to achieve solid-liquid separation. The filter press had four 300 × 300 mm chambers, the operating pressure reached 6 bar, the maximum capacity was ~6 kg wet filter cake and the pH of the slurry was required to be ≥3.0 (**Figure 8**).

4. Results and Discussion

4.1 Results in Laboratory Conditions

In Stages I and II, four factors were investigated, including the HCl:concentrate ratio, water:concentrate ratio, leaching temperature and dry digestion time. Previous research had demonstrated that leaching time was not a key factor (32) and it was easily studied in pilot plant tests by taking continuous samples. As a result, the laboratory leaching time was maintained at 1 h. It should be noted that the quantity of HCl used and leaching temperature represented the main process costs. The excess acid also required a more basic agent for neutralisation when recovering REE from the leaching solution. The chosen parameters were proposed from experience as well as previous experimental work at the EURARE project.

4.1.1 Effect of HCl:Concentrate Ratio on REE Extraction

The effect of the acid amount was evaluated by varying the acid:concentrate ratio (l:kg) from 1:1 to 1.5:1 (**Figure 10**) and it was observed that REE extraction efficiency increased as acid increased. However, for ratios from 1.25:1 to 1.5:1, no significant increase in REE extraction was found. Taking into account that the acid represented one of the main costs in this process, the optimal ratio of acid:concentrate was set at 1.25:1.
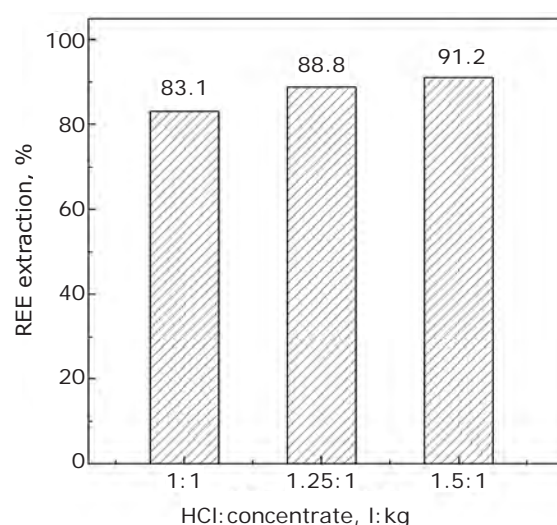

Fig. 10. Effect of HCl:concentrate (l:kg) ratio on REE extraction

4.1.2 Effect of Dry Digestion Time on REE Extraction

The effect of digestion time on REE extraction was tested using an acid:concentrate ratio of 1.25:1. The treated concentrate was subsequently leached at room temperature and a water:concentrate ratio of 2:1 for 1 h. The digestion process was performed without external heating, but the reaction temperature reached 70–80°C because of exothermic effects. Also, highly concentrated acid could have enhanced the reaction. Thus, the extraction efficiency reached a high level in a short time, increasing from 82.2% to 88.8% when the digestion time increased from 20 min to 40 min, with no further increase noted with longer times (**Figure 11**). Therefore, 40 min was considered sufficient time for the digestion process.

4.1.3 Effect of Water:Concentrate Ratio on REE Extraction

After acid digestion under the optimal conditions, the effect of water:concentrate ratio during leaching was determined. Addition of more water resulted in a higher liquid:solid ratio and the extraction efficiency increased correspondingly. The extraction efficiency thus increased with increased water:concentrate ratio (**Figure 12**). Considering the volume of the resulting slurry, a water:concentrate ratio (l:kg) of 2:1 was chosen as the optimal ratio and was applied in the upscaling test.
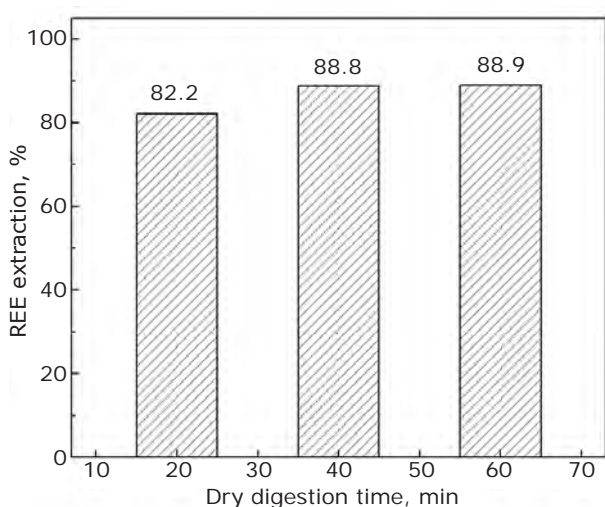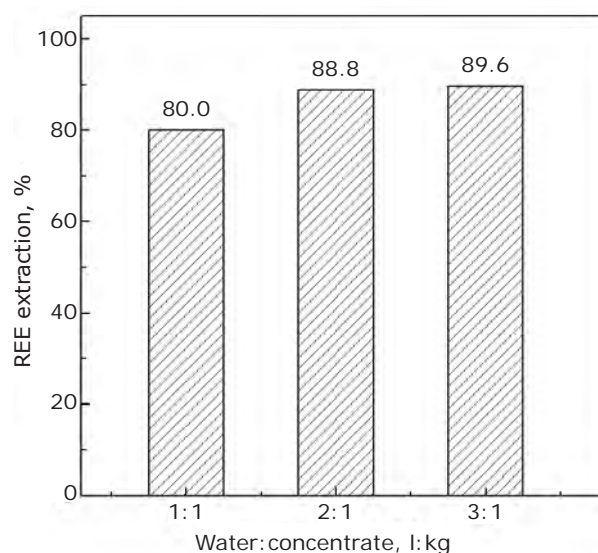

Fig. 12. Effect of water:concentrate ratio on REE extraction

4.1.4 Effect of Leaching Temperature on REE Extraction

The experimental results of this examination suggested that the leaching temperature did not affect REE extraction, as it was mainly a process of salt dissolution, transferring target elements to solution. The results indicated that it was not necessary to conduct leaching at high temperature and thus, room temperature leaching was deemed reasonable (**Figure 13**).
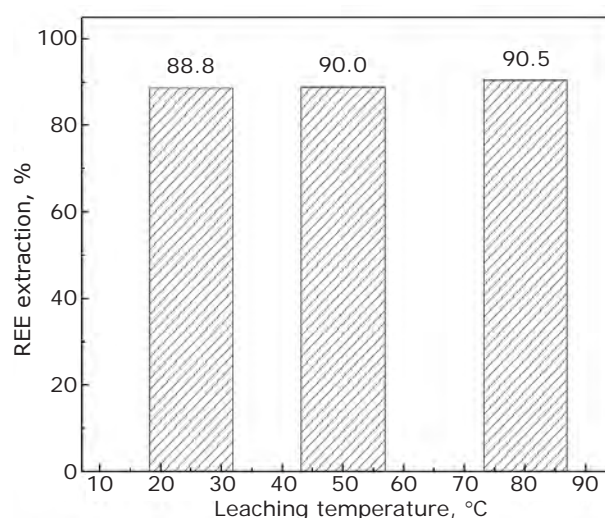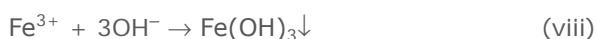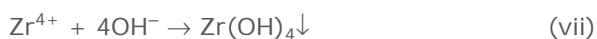

Fig. 11. Effect of dry digestion time on REE extraction

Fig. 13. Effect of leaching temperature on REE extraction

4.1.5 Results of Impurity Removal by Neutralisation

The composition of REE enriched Solution I showed that silica dissolution was very low and, in the meantime, some common impurities were leached by the acid along with the REE (**Table II**). In addition, this low pH slurry was not suitable for the filter press in the demonstration plant. Therefore, $CaCO_3$ neutralisation was studied for preliminary Fe, Al and Zr removal according to Equations (vii)–(ix):

$$Zr^{4+} + 4OH^- \rightarrow Zr(OH)_4\downarrow \qquad \text{(vii)}$$

$$Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3\downarrow \qquad \text{(viii)}$$

$$Al^{3+} + 3OH^- \rightarrow Al(OH)_3\downarrow \qquad \text{(ix)}$$

The effects of pH on Zr, Al and Fe removal and REE loss showed that, the higher the pH, the more Zr, Al and Fe precipitated (**Figure 14**). When the pH was adjusted to ~4.0, Zr, Al and Fe were removed at 99.1%, 90.0% and 53.1%, respectively and REE loss was 2.1%. Further $CaCO_3$ addition did not clearly promote impurity removal but caused more REE loss. Thus, the optimal pH for this step was set at ~4.0. After neutralisation and filtration, the final REE enriched solution was obtained for further purification.

4.2 Pilot Scale Results

An upscaling test containing 36 kg of eudialyte concentrate was carried out using the optimal parameters from the small-scale laboratory experiments. There were two reactors (40 l each) for the dry digestion process at the demonstration plant. Considering that the slurry was thick after mixing the acid and eudialyte concentrate,
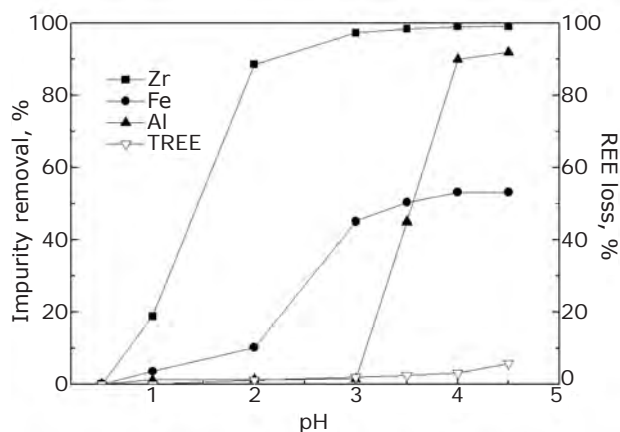

Fig. 14. Effect of pH on impurity removal and REE loss

the handling capacity was controlled at 6 kg of concentrate at a time per reactor and repeated three times every day; the parameters are shown in **Table III**. There were some unfavourable results in cases of improper operations during the

Table III Parameters for Pilot Scale Processing of TANBREEZ Concentrate

Parameter	Unit	Value
Dry digestion temperature	°C	70–80
Digestion time	min	40
Addition of eudialyte concentrate	kg	6 per reactor
Addition of HCl (10 M)	l	7.5 per reactor
Leaching temperature	°C	20–25
Leaching time	min	30
Addition of water in fuming reactor	l	12 per reactor
$CaCO_3$	kg	7.6

Table II Chemical Composition of Rare Earth Element Enriched Solution I

Element	Concentration	Element	Concentration
Al	5.58 g l^{-1}	Ce	976 mg l^{-1}
Ca	7.29 g l^{-1}	Pr	108 mg l^{-1}
Fe	4.31 g l^{-1}	Nd	382 mg l^{-1}
Mn	569 mg l^{-1}	Sm	99 mg l^{-1}
Nb	<2 mg l^{-1}	Gd	80 mg l^{-1}
Zr	2.57 g l^{-1}	Dy	105 mg l^{-1}
Hf	30 mg l^{-1}	Y	610 mg l^{-1}
Si	172 mg l^{-1}	Yb	73.4 mg l^{-1}
La	445 mg l^{-1}	TREE[a]	2.88 g l^{-1}

[a]TREE: total rare earth elements

acid digestion process in the pilot scale test. First, agglomeration appeared when injecting the acid if the eudialyte concentrate was poured into the reactor first, resulting in stirring difficulties and an inadequate reaction. In addition, it was also not feasible to add the eudialyte concentrate into the reactor containing the acid, because silica gel formed quickly as the acid was very excessive in the system at the beginning of charging. Therefore, the reactors were filled alternately with acid and concentrate (1.25 l of acid and ~1 kg concentrate, alternating) until the required quantity was reached.

Once acid digestion was completed, it was not possible to transport the slurry directly because of its high solid content. For transport, 8 l of water was first added into the reactor, mixed for a few minutes and then an air compressor pump used to move the suspension by pipe to a large tank. As the reactor still contained some solid residue, another 4 l of water was injected and the remaining slurry transported.

Leaching was then carried out at room temperature for between 10 min and 60 min with samples taken periodically. The results verified that leaching time was not an important factor for REE extraction and therefore 30 min was sufficient for the leaching process (**Figure 15**). After that, limestone was used to neutralise the acidic suspension to ~4.0. Next, the suspension was pumped into a filter press and the separation of solid and liquid achieved. The
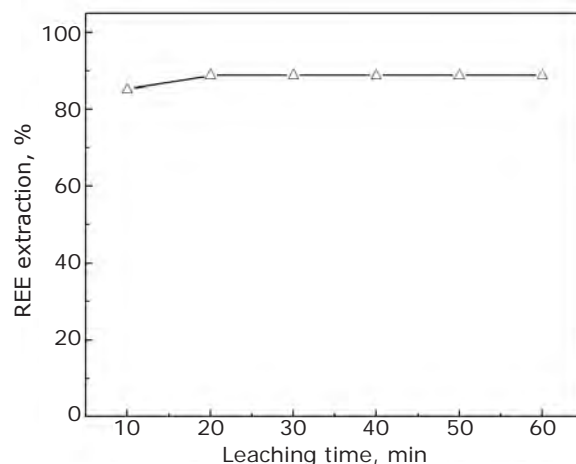

Fig. 15. Effect of leaching time in pilot scale test (other conditions shown in Table III)

final REE enriched Solution II was obtained after washing the filter cake. The material balance of the pilot scale test is shown in **Figure 16**. Analysis of the eudialyte residue indicated that 2% of the eudialyte remained in the residue (**Figure 17**); similar results have already been discussed by Vossenkaul (29). The incomplete REE recovery could have been due to the embedding of eudialyte in the eudialyte residue, in aggregation with feldspars and siliceous precipitate.

As converting REE into a solid product benefits both storage and transport, a preliminary product, REE carbonate, was finally produced in this study.
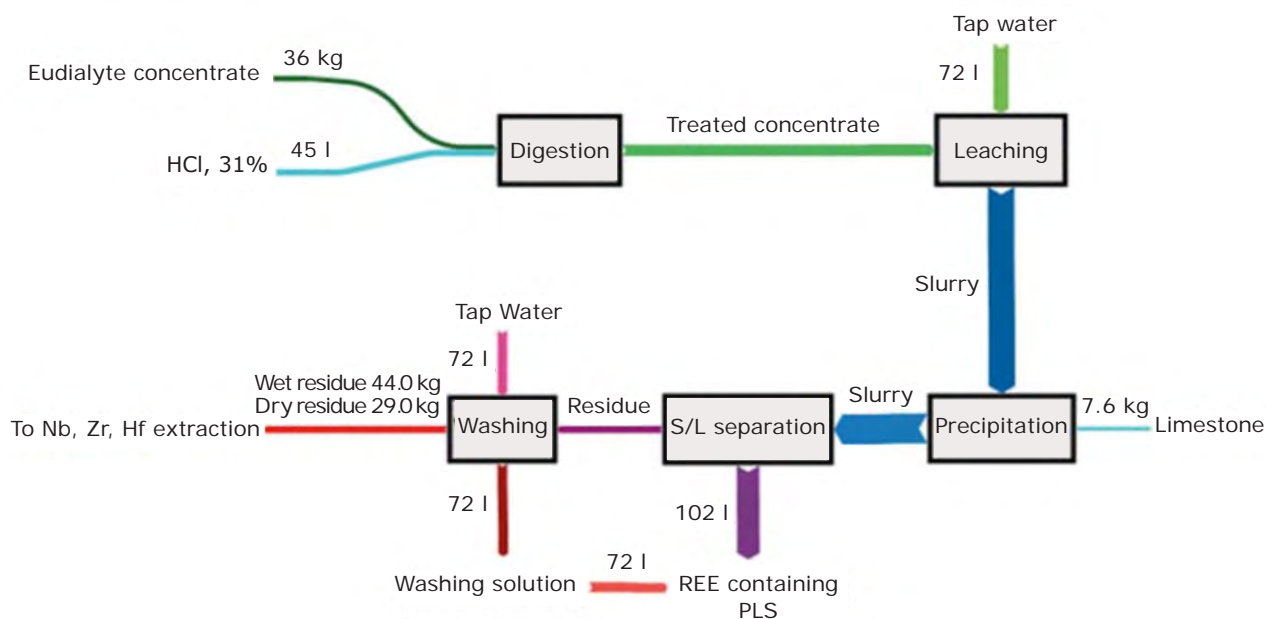

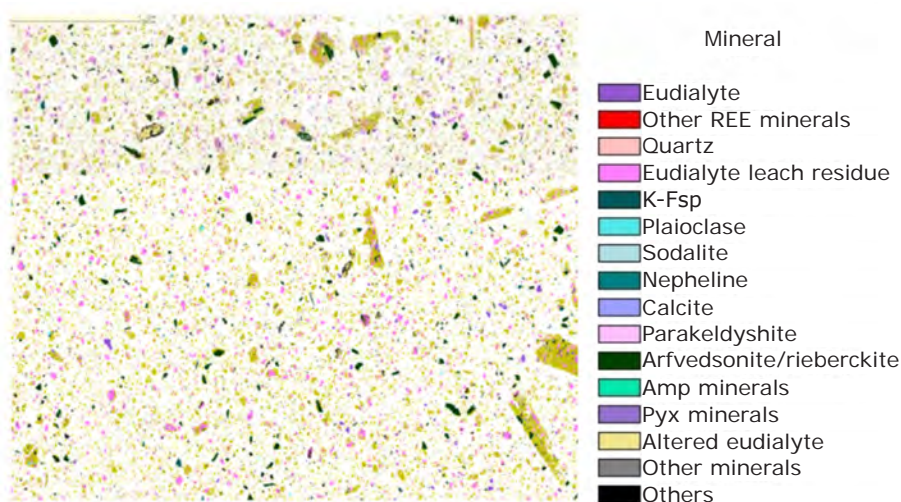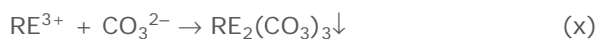Fig. 16. Material balance of the pilot scale test

Fig. 17. QEMSCAN of eudialyte residue

Sodium carbonate was added to the REE enriched Solution II to a pH of 5.7 to precipitate the REE as REE carbonate. The reaction is shown in Equation (x):

$$RE^{3+} + CO_3^{2-} \rightarrow RE_2(CO_3)_3\downarrow \qquad (x)$$

The REE precipitation efficiency was found to be higher than 98.8% at pH 5.7. This first precipitate obtained contained high Fe and Ca but, after removing them by dissolution and reprecipitation, a higher quality product was obtained (**Figure 18**). The produced carbonate contained 30.0% REE (**Table IV**), which qualified for further treatment to obtain a final pure product; solvent extraction would be utilised in the EURARE project. The overall extraction efficiency of REE to obtain the

Table IV Chemical Analysis of Obtained Rare Earth Element Carbonate			
Element	**wt%**	**Element**	**wt%**
Al	3.31	Ce	10.50
Ca	4.18	Pr	0.43
Fe	0.20	Nd	3.90
Zr	0.05	Sm	0.93
Mn	0.23	Gd	0.85
Na	0.02	Dy	1.17
Zn	0.05	Y	7.15
Si	0.03	Yb	0.24
La	5.23	TREE[a]	29.95

[a]TREE: total rare earth elements

REE carbonate product was 85.5%. Overall, it was concluded that the flowchart designed on the basis of laboratory experiments was feasible and the REE extraction performance under the optimal parameters was good.

5. Conclusion

In this study, a complete hydrometallurgical treatment of a eudialyte concentrate from TANBREEZ was successfully carried out. The flowchart proposed was capable of achieving high REE recovery, avoiding silica gel formation and lowering operation costs (no external heating).

The optimal parameters in Stages I and II were: HCl:concentrate ratio of 1.25:1 (l:kg), water:concentrate ratio of 2:1 (l:kg), dry digestion

Fig. 18. Produced REE carbonate (30.0 wt% REE)

time 40 min, leaching temperature 20–25°C (room temperature) and leaching time 30 min. Under these conditions, a high REE extraction efficiency of 88.8% was obtained. By taking advantage of the dry digestion process with high concentrated acid, the leaching time and leaching temperature were found to have no significant influence on REE extraction. In Stage III, pH was adjusted to the optimum (~4.0) with $CaCO_3$, which led to a preliminary impurity removal and met the requirement of filter pressing at the demonstration plant when scaling up.

Using the optimal parameters the upscaling test, preceding future industrial application, was conducted to verify the feasibility and efficiency of this REE extraction process. Some operational considerations were also recognised in the upscaling test. The final product, REE carbonate (up to 30.0 wt% total REE) was produced from the REE enriched solution by adding Na_2CO_3. The duration of this treatment was short and no external heating required. The overall efficiency of REE recovery reached 85.5%, which confirmed the feasibility and good performance of this process.

Acknowledgements

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