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Prevention of Silica Gel Formation for Eudialyte Study Using New Digestion Reactor

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Abstract: This work includes a combined hydrometallurgical treatment of a eudialyte ore sample with a subsequent preparation of leaching residue using mechanical separation methods. Hydrometallurgical treatment contains dry digestion with sulphuric acid at room temperature and filtration of the obtained product. The objectives of adopting these procedures are to test a new digestion reactor in order to prevent silica gel formation from the eudialyte ore. The obtained results revealed that silica gel formation is prevented during dissolution with sulphuric acid. A high leaching efficiency of light rare earth elements (La, Ce, Nd, Y) was reached using the dry digestion process with sulphuric acid, where the starting molarity was 12 mol/L. After the filtration process, magnetic separation is studied as the main method to recover weakly magnetic minerals like amphiboles and pyroxenes from the leaching residue in the magnetic fraction and feldspars in the nonmagnetic fraction. A new combined research strategy was developed for the production of different concentrates such as the one bearing Zr, Hf, and Nb.

Keywords: hydrometallurgy; dry digestion; silica gel prevention; magnetic separation; eudialyte; aegirine; feldspar

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1. Introduction

Eudialyte is a unique mineral and nowadays is known as a potential source of rare earth and high-field-strength elements in the absence of radioactive elements such as uranium and thorium, which is the case for bastnasite, xenotime, monazite and steenstrupine. Significant resources of eudialyte and accompanied zirconosilicates are represented by Ilimaussaq and Tanbreez (Greenland), Norra Kärr (Sweden), Khibina and Lovozero (Russia), Mont Saint-Hilaire and Kipawa (Canada), Pilanesberg (South Africa), Saima (China) and Poços de Caldas (Brazil) [1,2]. Besides eudialyte and accompanied complex Zr and Ti silicates, the eudialyte ore consists of mafic minerals like amphiboles and pyroxenes and of alkali feldspars and feldspathoids like nepheline [3]. According to the current state of the art, dry and wet high-intensity magnetic separation (WHIMS) is widely proposed as the main method to recover weakly magnetic minerals with amphiboles, pyroxenes and eudialyte for the magnetic fraction, and feldspars and nepheline for the non-magnetic fraction [4]. Moreover, various marketable products could be additionally obtained from eudialyte ores as by-products like nepheline-syenite and feldspars.

A study on the extraction of REE as critical metals from eudialyte has been mainly confined to laboratory studies and combined hydrometallurgical treatments, producing different precipitation products such as rare earth carbonate and rare earth oxalate. Despite being easily dissolved by acids such as hydrochloric acid and sulphuric acid, the main challenge with this processing has been how to avoid silica gel formation, which is a gelatinous and non-filterable phase [5]. Lebedev et al. [6,7] have studied a two-stage

decomposition process for REE recovery. High-temperature leaching with concentrated sulphuric acid (H_2SO_4) was used to decompose the eudialyte. Then, following washing of the slurry with sodium sulphate solution, the REE remained in the insoluble residue as double sulphate salts. The residue was then washed again and REE recovered after converting the sulphates into nitrates or chlorides. Ma et al. [8] have studied the effect of digestion time on REE extraction 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. 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 [9]. F^- ions can promote eudialyte decomposition, but this is also toxic, and it can destroy glass equipment.

The valorisation of various waste is very important for the sustainable development of a green and circular economy [10–14]. Therefore, this study proposes a mechanical separation of valuable mineral products from leaching residues with the goal being to minimise the volume of the tailings storage. The most valuable minerals of the leaching residues are feldspars and mafic minerals, represented by pyroxenes like aegirine-augite groups. Due to the differences in the specific magnetic susceptibility of aegirine-augite groups and feldspars, the magnetic separation could also be considered as the main method for the treatment of solid leaching residues [15,16]. Feldspars are used in the glass and ceramic industries as filling materials [17,18]. Aegirine could be used as pigments, mineral wools, refractory, ceramic and glass industries (glazes, black glass, decorative glassware), fillers, coatings, lubricants for hot metal forming, weighting agents or as low-melting flux for pyrometallurgical refinement of copper from impurities [19–21].

The first aim of this study is the extraction of rare earth elements using dry digestion process in special constructed reactor preventing the formation silica gel formation from a eudialyte ore sample from Norra Kärr deposit. This digestion reactor was developed from a 1 L laboratory reactor to an increased 100 L one at the Institute for Process metallurgy and Metal recycling of the RWTH Aachen University in Germany [22,23] in contrast to some bioleaching processes [24], where the extraction efficiency for the single REEs was a maximum of 35% for zircon concentrates but up to about 70% for the eudialyte concentrate. A large number of original research articles covering techniques including solvent extraction, ion exchange, precipitation and other emerging technologies have been reviewed in detail and critically assessed to understand the role and behaviour of specific impurities in REE processing (including Al, Ca, Mg, Fe, Si, Th, U, Ti, Zr, Hf, Cr, Mo, Mn, Co, Ni, Cu, Zn, Sn, Pb and Bi) [25]. The second aim of this work is to is to investigate the possibility of producing valuable mineral products like aegirine and feldspar products from solid leaching residue using mechanical separation methods in the final step.

2. Materials and Methods

2.1. Materials

The original sample of low-grade eudialyte ore from the Norra Kärr deposit in Sweden was used for various geometallurgical investigations during the multidisciplinary Siemens research section (S-FB): Rare Earth—Green Mining and Separation. The remaining processing products from geometallurgical investigations (sensor-based sorting, magnetic and electrical separation, flotation) were mixed, homogenised and split into subsamples for hydrometallurgical investigations in this work. Initially, the eudialyte ore sample was crushed by a jaw crusher (Type MN 931/10. KHD Humboldt Wedag AG, Cologne, Germany) and then ground by a roll crusher (Type LWBP 2/2, Karl Merz Maschinenfabrik GmbH, Heschingen, Germany) in a circuit with a 180 µm sieve. After hydrometallurgical treatment in new digestion reactor, the pulp was filtered using a filter press to obtain the filter cake (leaching residue) and the filtrate (REE-bearing solution). The leaching residue

was then used for magnetic separation tests. The experimental process flowsheet is given in Figure 1.

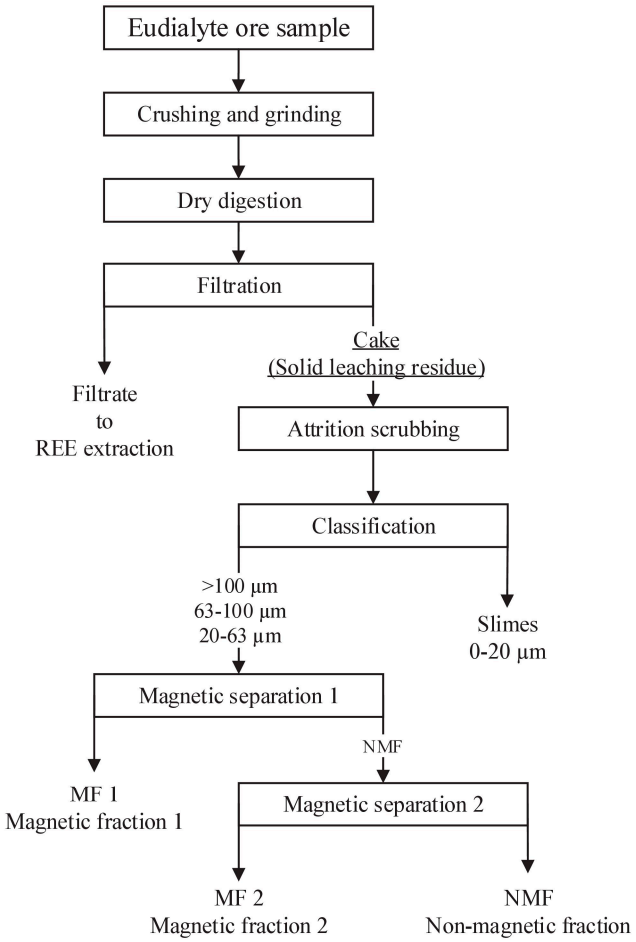


Figure 1. Experimental process flowsheet.

The chemical composition of the low-grade eudialyte ore sample is given in Table 1.

Table 1. The chemical composition of eudialyte ore sample (wt.%).

Si	Na	K	Al	Zr	Ti	Ca	Fe	Mn	P	LREE (La–Sm)	HREE (Y, Eu–Lu)
25.15	6.85	3.16	8.65	0.79	0.12	1.31	3.27	0.14	0.045	0.2823	0.1514

The molar proportion of (Na + K)/Al ratio in the investigated sample is 1.15 (>1). The sample of investigated eudialyte ore belongs to agpaitic rocks. About 35% of REE belong to HREE. Zr and REE are mainly distributed in eudialyte. According to the mineralogical analysis, the eudialyte ore sample consists of eudialyte (9%), feldspars (50%), mafic minerals (16%) like aegirine-augite series and feldspathoids (23%) like nepheline and natrolite. The micas minerals (biotite and muscovite) are detected as trace phases with a content below 2%. The metal distribution of sieve fractions is shown in Figure 2.

The particle size distribution of the milled eudialyte ore sample shows that 80% of the particles are smaller than 120 µm. Nearly 30% and 15% of the particles are less than 63 and 20 µm, respectively. Over 70% of Zr and REE in the eudialyte ore sample is distributed in the fraction below 63 µm. Sulphuric acid, 95.0%–98.0%, 258205-2.5 L, Sigma-Aldrich Chemie GmbH, Schnelldorf, Germany, for dry digestion process. Tap water was used for leaching process after dry digestion process.

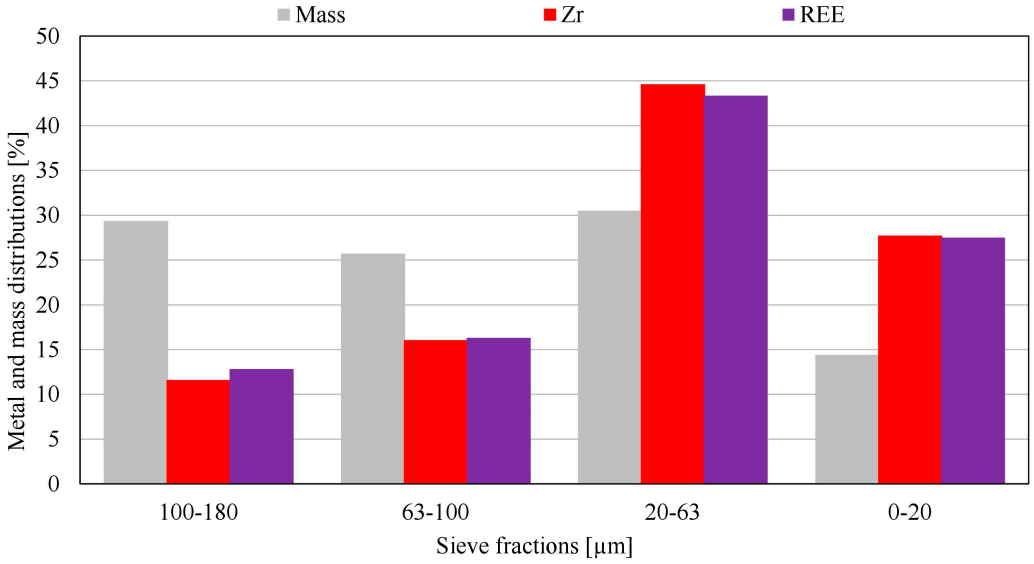


Figure 2. Particle size distribution and distribution of metals in sieve fractions of the eudialyte ore sample.

2.2. Methods

2.2.1. Sample Characterisation

The eudialyte ore and leaching residue were physically and chemically characterised. X-ray diffraction analyses of the eudialyte ore, solid leaching residue and mineral products were performed with an X-ray diffractometer (D2 Phaser, Bruker AXS, Karlsruhe, Germany), equipped with DIFFRAC.EVA Software, Version 5.0 and DIFFRAC.TOPAS Software, Version 5.0 for mineral identification and quantification, respectively. The solid samples were analysed via XRF (Panalytical Axios 2005, Malvern PANanalytical GmbH, Kassel, Germany), while the composition of liquid samples was determined via inductively coupled plasma optical emission spectroscopy (ICP-OES, Spectro Arcos 2013, SPECTRO Analytical Instruments GmbH, Kleve, Germany). The wet sieve analysis was provided using a sieve shaker (ASM 400 Siebtechnik GmbH, Mülheim a.d. Ruhr, Germany) to obtain the particle size distribution. An optical visualisation of mineral products was performed with a digital microscope (Keyence VHX-5000, Osaka, Japan). A pH meter (PCE-PHD1, PCE Deutschland GmbH, Meschede, Germany) was used to measure the pH value during attrition scrubbing.

2.2.2. Hydrometallurgical Treatment

The development of a modular reactor for the dry digestion of high-silicate ores and concentrates to avoid gel formation using eudialyte ores, as an example, is being carried out by the Institute for Process Metallurgy and Metal Recycling at RWTH Aachen University and the konzept GmbH, Engineering Services, Düren, realised together. In this study, a new reactor is developed for a process that has already been confirmed on a laboratory scale to avoid gelling (dry digestion) during leaching. Injection in the reactor is possible up to a scale of 75 kg (100 L), as shown in Figure 3a.

Special nozzle systems were used for acid injection aiming at leaching of metals and water injection for a dilution of suspension and additional leaching, as shown in Figure 3b. Furthermore, it should be proven that the modular system technology is robust in terms of chemical resistance and reproducibility of the results achieved. The innovation compared to process alternatives lies in the complete avoidance of external and cost-intensive heating energy as well as the previously impossible implementation of the dry digestion process in large solution volumes. Before gel formation, it is possible to convert the silicate components into crystals, which are filterable and stable. These results shall enable the cost-effective production of rare earth oxides.

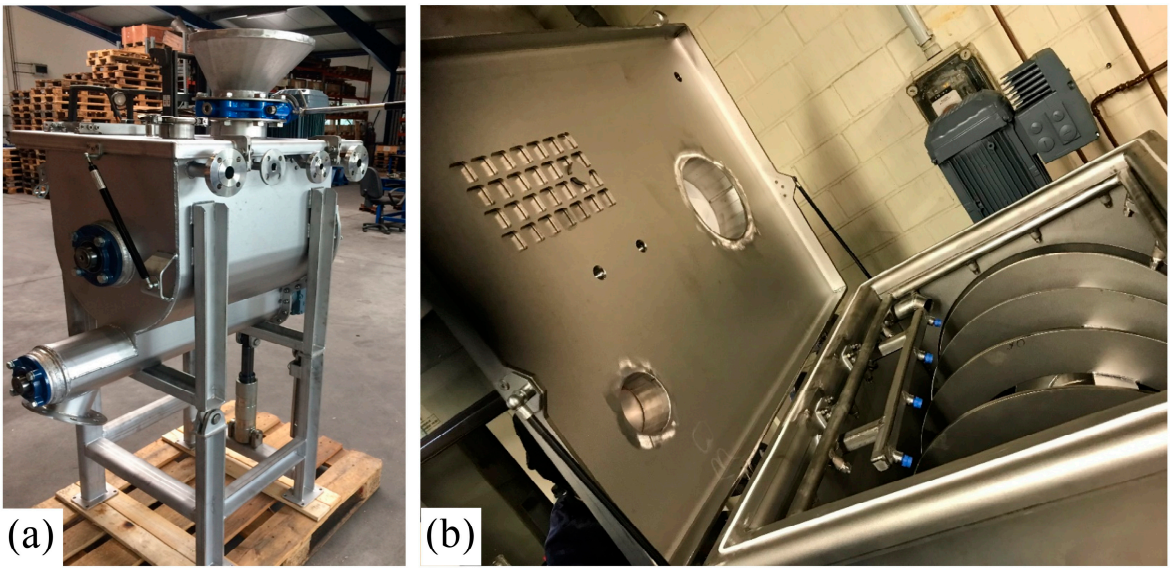


Figure 3. Dry digestion reactor (**a**); nozzle systems (blue) (**b**).

2.2.3. Magnetic Separation

Prior to the magnetic separation, the attrition scrubbing of leaching residue was provided in a laboratory-scale flotation machine (Type D-12, Denver Equipment Company, Littleton, CO, USA) in a 3 L beaker with 60% solids content. Impeller speed was set to 1600 rpm. Then, the treated leaching residue was sieved, dried and sampled for the magnetic separation tests, which is a widely used physical method to separate minerals with different magnetic properties. These tests were performed with a Frantz isodynamic magnetic separator (S.G. Frantz Company Inc., Tullytown, PA, USA), which is a device to accurately separate mineral grains according to their specific paramagnetic susceptibility. Three sieve fractions (>100, 63–100 and 20–63 µm) were used for the magnetic separation tests under various magnetic field strengths (0.5 and 1.6 T) at an orientation of magnetic system with a constant inclination angle (side slope of 15°, and forward tilt of 15°). As a result, two magnetic fractions (MF1 and MF2) and one non-magnetic fraction (NMF) were separated from each sieve fraction of 200 g.

3. Results and Discussion

3.1. Hydrometallurgical Treatment

The new dry digestor reactor, as shown in Figure 3, was used for hydrometallurgical treatment of eudialyte ore at room temperature using highly concentrated sulphuric acid. The value of 12 M is the starting molarity from the dry digestion stage. So, the 96% H_2SO_4 is diluted with water to achieve the desired 12 M. The tested experimental parameters for a treatment of eudialyte ore are shown in Table 2.

Table 2. Experimental parameters for hydrometallurgical treatment in dry digestor reactor.

Parameter	Unit	Value
Initial Material	kg	20
Used acid (H_2SO_4)	%	96
Concentration	mol/L	12
Duration of dry digestion	min	120
Solid/liquid in dry digestion (H_2SO_4)	-	1:2
Solid/liquid in leaching (H_2O)	-	1:5
Duration of leaching	min	60

The obtained results under these conditions are shown in Figure 4.

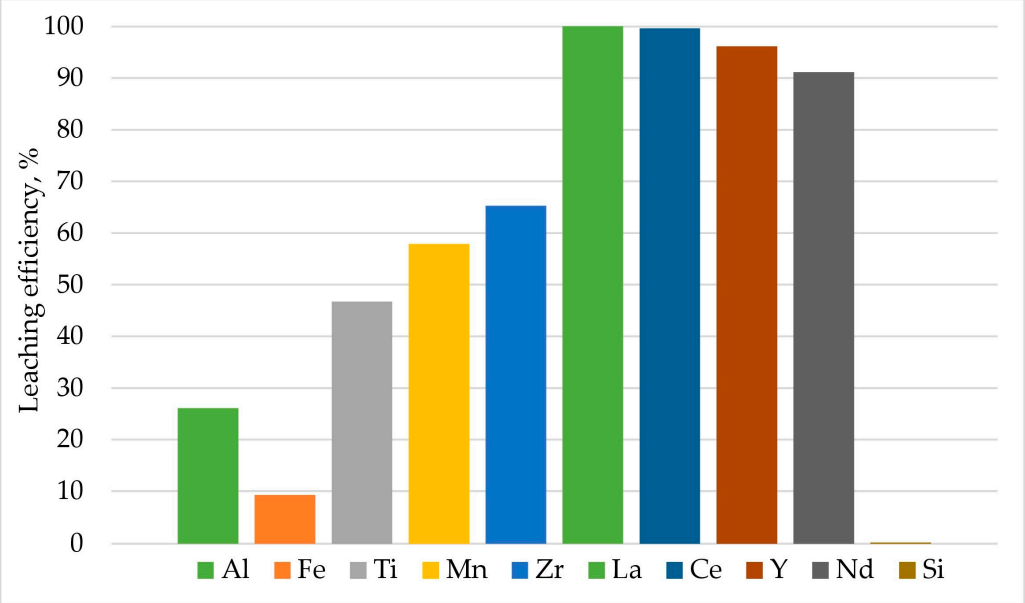


Figure 4. Leaching efficiency after hydrometallurgical treatment.

The obtained results have shown the high leaching efficiency (higher than 90%) at room temperature for rare earth elements (Nd, Y, Ce and La). At the same time, the leaching efficiency for aluminium and iron amounted to 10% and 25%, respectively. The minimal extraction achieved was for silicon (about 0.2%). This is the main reason that silica gel formation was prevented. The leaching efficiency for titanium, zirconium and magnesium amounted to between 45 and 65%., In principle, it is possible to receive higher Zr and, in the same turn, Hf yields. It is a question of the material composition and final pH in solution. As we lower the concentration of the sulphuric acid in the "washing" or "leaching" stage with a factor of 1:10, the final concentration is somewhat low compared to all the soluble elements in the input material. The final pH is about 2–3, and the Zr in the solution starts to precipitate. Therefore, if it is aimed to achieve higher Zr yields, it is possible to reach it starting with a higher acid molarity, change the s/l ratio or the dilution factor. But the main objective of this study is the extraction of the REE, and there is not high interest for this research, which is studied by Ma et al. [23]!

3.2. Magnetic Separation

After filtration using filter press (ANDRITZ, Cologne, Germany) under working pressure approx. 5–6 bar below 50 °C, the filter cake (leaching residue) was sampled and wet sieved. Figure 5 shows the particle size distribution of the original filter cake.

According to the granulometric distribution in Figure 5, approximately 58% of the particles in the original filter cake are smaller than 100 µm. Preliminary visual examination of sieved fractions using the digital microscope shows that the mineral particles are cemented (Figure 6a) or coated (Figure 6b).

To liberate the aggregates of cemented mineral particles as well as to clean the mineral surfaces for further separation, the leaching residue was scrubbed in the flotation cell for 15 min and wet sieved. The pH value of pulp during the attrition scrubbing was measured at 3. After attrition scrubbing, the visual examination shows that the mineral particles were liberated and cleaned (Figure 6c). Moreover, after attrition scrubbing, the amount of slime particles smaller than 20 µm was raised from 14 up to 35%, while the amount of particles of over 100 µm was reduced from 42 to 15% (as shown in Figure 5). Due to the negative impact of slimes on physical separation processes, the fraction smaller than 20 µm was separated by wet sieving and was not further considered for the experiments in this work. The yield of the deslimed fraction is over 65%. The chemical composition of the leaching residue after desliming is listed in Table 3.

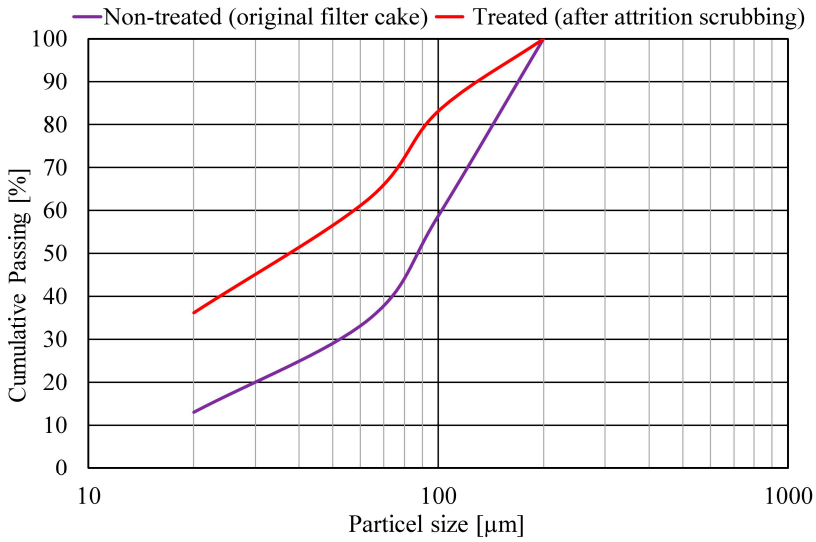


Figure 5. Particle size distribution of treated and non-treated leach residue.

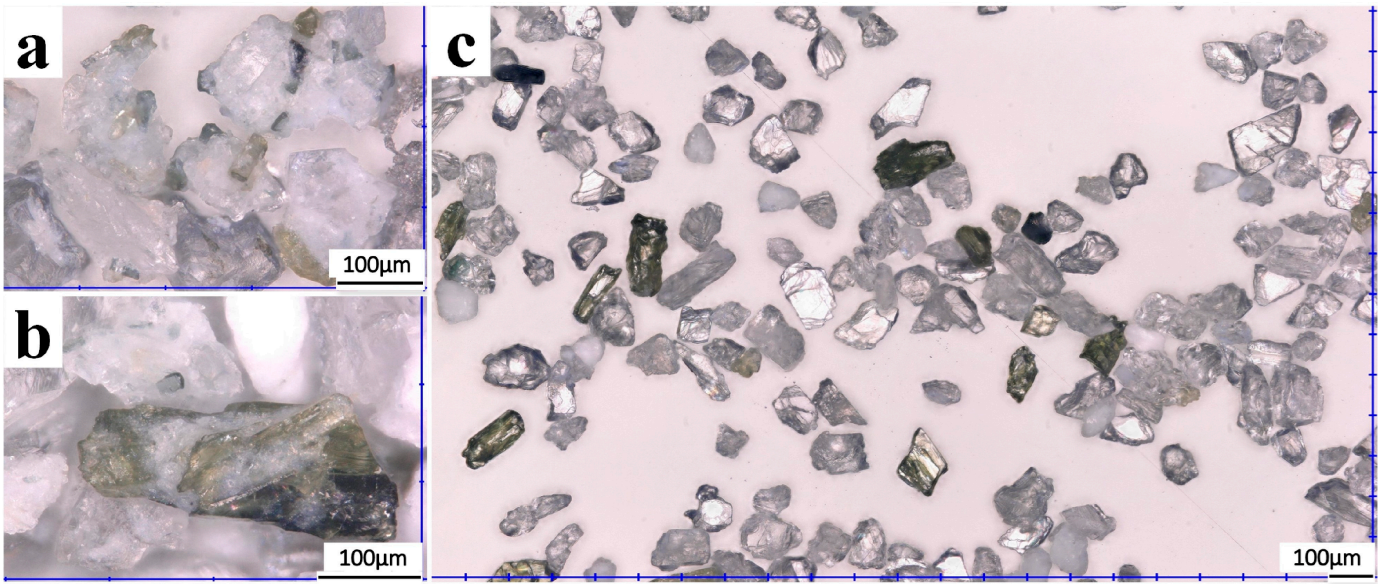


Figure 6. Optical microscope images of original filter cake (**a**,**b**) and treated leach residue (**c**).

Table 3. Chemical composition of leaching residue after desliming (%).

SiO_2	Al_2O_3	Na_2O	K_2O	CaO	MgO	Fe_2O_3	MnO	ZrO_2	TiO_2	P_2O_5
60.6	15.15	7.2	4.58	0.77	0.45	4.82	0.16	0.27	0.092	0.31

The main components in the leaching residue are SiO_2 and Al_2O_3. Fe, Ti and Mn mostly belong to iron-bearing silicates like pyroxenes and amphiboles and are partly distributed in the leached eudialyte phase. Zr also belongs to leached eudialyte. Feldspars are the major minerals remaining in the leach residue. The results of the magnetic separation are listed in Table 4.

Table 4. Results of magnetic separation.

Products	Yield, %	Fe_2O_3		ZrO_2		TiO_2	
		Content, %	Recovery, %	Content, %	Recovery, %	Content, %	Recovery, %
Magnetic Fraction 1 (MF1)	15.64	28.20	94.11	0.39	17.09	0.34	43.22
Magnetic Fraction 2 (MF2)	4.66	3.51	3.50	3.15	41.60	0.80	30.70
Non-Magnetic Fraction (NMF)	79.70	0.14	2.40	0.18	41.31	0.04	26.08
Feed	100.00	4.69	100.00	0.35	100.00	0.12	100.00

The separation of the iron-bearing silicate minerals from the leaching residue in the magnetic fraction 1 is feasible with a magnetic field intensity of 0.5 T. Over 90% of Fe is recovered in the magnetic fraction 1 with a yield of 15.6%. The XRD pattern of the magnetic fraction is presented in Figure 7.

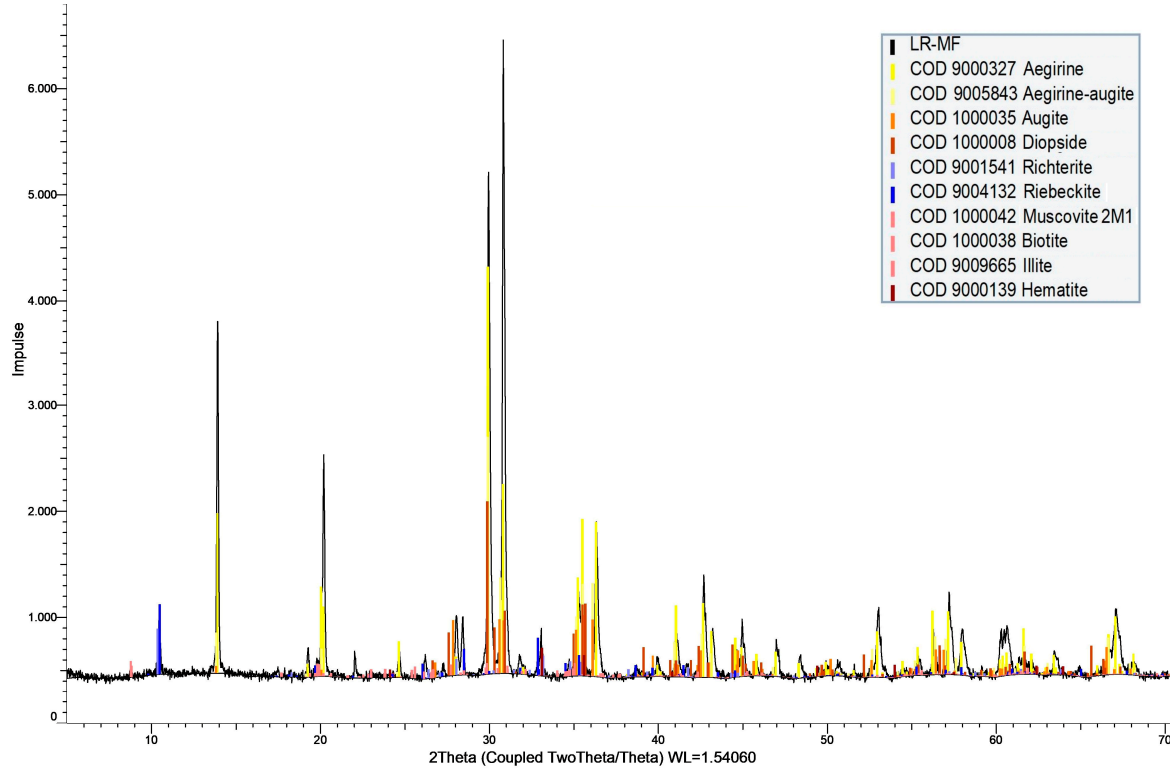


Figure 7. XRD pattern of the magnetic fraction.

The major phases belong to aegirine-augite series (89%), alkali amphiboles (riebeckite) and micas. The aegirine particles are elongated and needle-shaped crystals (as shown in Figure 8a).

Thus, it was also confirmed that after hydrometallurgical treatment using a prototype digestion reactor, the mafic minerals preserve their magnetic properties and can be recovered in the magnetic fraction.

The separation of feldspars in the non-magnetic fraction is carried out with a magnetic field intensity of 1.6 T. The main aim of the second magnetic separation stage is to further reduce the iron content in the feldspar product. The middling magnetic fraction (MF2), obtained due to the two-staged magnetic separation, has a ZrO_2 content of 3.15% with a mass recovery of 4.6%. The middling fraction is the feed for the resource-efficient recovery of Zr and Hf, which is mentioned by Ma et al. [23]. The amounts of Fe and Ti in the non-magnetic fraction were reduced to 0.14% and 0.04%, respectively. The microscope image of the purified fraction is presented in Figure 8b. According to mineralogical analysis, the non-magnetic fraction consists of feldspars (88%) and residue zeolites (Figure 9).

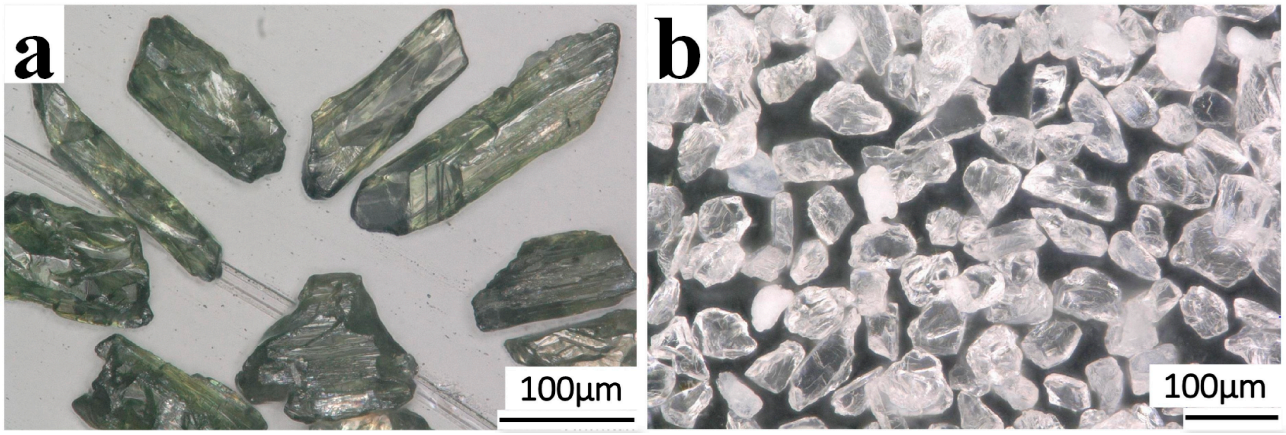


Figure 8. Optical microscope images of magnetic (**a**) and non-magnetic fractions (**b**).

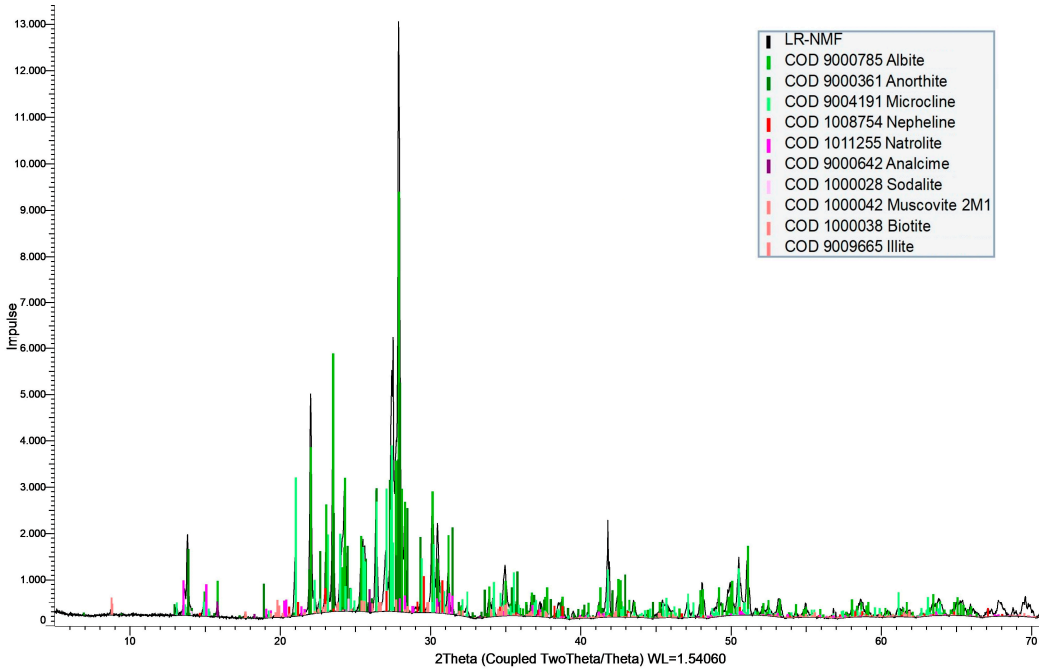


Figure 9. XRD pattern of non-magnetic fraction.

Thus, the two-staged magnetic separation is a suitable method to treat the solid leaching residue from eudialyte processing. The chemical composition of the magnetic (MF1) and non-magnetic fractions (NMF) is listed in Table 5.

Table 5. Chemical composition of magnetic (aegirine) and non-magnetic fractions (feldspar) (%).

Products	SiO_2	Al_2O_3	Na_2O	K_2O	CaO	MgO	Fe_2O_3	MnO	ZrO_2	TiO_2	P_2O_5
Aegirine (MF1)	48.28	1.53	8.35	0.86	1.78	2.78	28.15	0.62	0.39	0.36	0.40
Feldspars (NMF)	64.10	17.21	7.10	5.11	0.55	0.16	0.14	0.01	0.18	0.04	0.15

In comparison to the feldspars concentrate, the aegirine concentrate is not presented in the global market as a commodity. The aegirine product composition is comparable with the composition of aegirine concentrate, obtained as a by-product in Khibina deposits from the Kola Peninsula [20]. Currently, the chemical and technical requirements for aegirine concentrates are not specified and must be determined by the potential consumer. Meanwhile, the most critical requirements for the usage of feldspars in glass and ceramic

industries are low contents of iron oxide and an alkali content (K_2O + Na_2O) as high as possible [17,18]. In comparison to dry magnetic separation, the removal of iron oxide could be improved using wet magnetic separation as well as optimised attrition scrubbing. Equally important is the particle size distribution, because coarse-grained products for glass industry are required [17,18]. The obtained feldspar product could be potentially used for the ceramic production and as filling material. Due to the lower transportation costs, the local consumer might successfully integrate the use the local products. Although the specification for internationally traded concentrates is rigorous, the local consumer might successfully integrate the use of local products due to lower transportation costs.

4. Conclusions

A combined treatment starting in a new digestion reactor with filtration, re-pulping, attrition, desliming and a two-step magnetic separation leads to the formation of concentrates of Zr, Nb and Hf from eudialyte ore and the maximal transfer of rare earth elements in the solution, as shown in Figure 10.

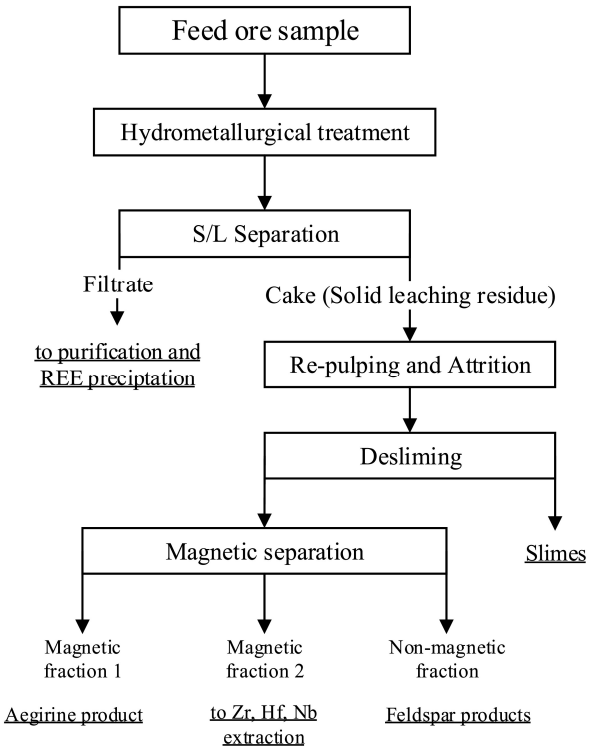


Figure 10. Proposed flowsheet for the treatment of a eudialyte ore sample and leaching residue.

During hydrometallurgical treatment with highly concentrated sulphuric acid in a new digestion reactor at room temperature, the leaching efficiency of La, Nd, Ce and Y is higher than 90%. Under the same conditions, silica gel formation is prevented. The solid leaching residue contains the value minerals and can be treated for the production of mineral products. For this purpose, the preliminary deagglomeration of particles of a solid leaching residue using attrition scrubbing is required. Then, the solid leaching residue gets deslimed, and the coarse fraction is treated using magnetic separation. The results of the magnetic separation showed that aegirine- and feldspar-bearing products can be additionally recovered using a two-staged magnetic separation. The WHIMS could be applied to recover aegirine from the slimes. The further application, sales and distribution of marketable mineral products is the challenge for the development of a circular economy. At the other side, the production of rare earth oxides from a solution after purification, precipitation and thermal decomposition is a real option in this case. This developed

strategy can be tested for the treatment of bauxite residue in order to decrease treatment costs in the future.

Author Contributions: Conceptualization, I.S.; methodology, I.S.; software, I.S.; validation, C.D. and S.S.; formal analysis, S.S. and V.S.C.; investigation, C.D. and I.S.; resources, I.S.; data curation, S.S.; writing—original draft preparation, I.S., S.S. and C.D.; writing—review and editing, I.S., C.D., S.S. and V.S.C.; visualisation, I.S.; supervision, B.F.; project administration, S.S.; funding acquisition, B.F. All authors have read and agreed to the published version of the manuscript.

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