Chapter

Concentrated Hydrochloric Acid Leaching of Greenland Steenstrupine to Obviate Silica Gel Formation

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Abstract

The Ilimaussaq complex in Greenland contains a rare earth bearing mineral called Steenstrupine. This mineral is a complex sodium rare earth phospho-silicate which also contains significant uranium and thorium. The mineral can be beneficiated via froth flotation to produce a mineral concentrate ranging between 15 and 23% rare earth oxide. Leaching of the mineral concentrate is required to dissolve the contained values and recover them using hydrometallurgy. Steenstrupine contains high amounts of acid soluble silica which can result in the formation of silica gel during leaching. Laboratory scale testwork was performed to determine which leaching conditions offer the control of silica and high extraction of values such as rare earth elements and uranium. A range of leach parameters where investigated to determine which are most significant to leach performance. Optimised parameters consisting of acid strength, residence time and nature of the operation were determined as significant. In conclusion the operating range identified produces high rare earth extractions while yielding a leach residue with suitable solid liquid separation performance. This process may be applied to other rare earth bearing minerals which contain high proportions of soluble silica.

Keywords: rare earth elements, leaching, silica gel, hydrometallurgy, steenstrupine, hydrochloric acid leaching

1. Introduction

1.1 Project information

The Ilimaussaq complex is located within the Kommune Kujalleq, the municipality of southern Greenland (**Figure 1**). It is a peralkaline intrusion which contains rocks which are extremely enriched in sodium, iron and high fields strength elements including rare earth elements.

Within the complex is the Kvanefjeld deposit which consists of a measured resource (JORC 2012) of 143 million tonnes @ 1.2% REO, 303 ppm U3O8 and 0.24% zinc [1].

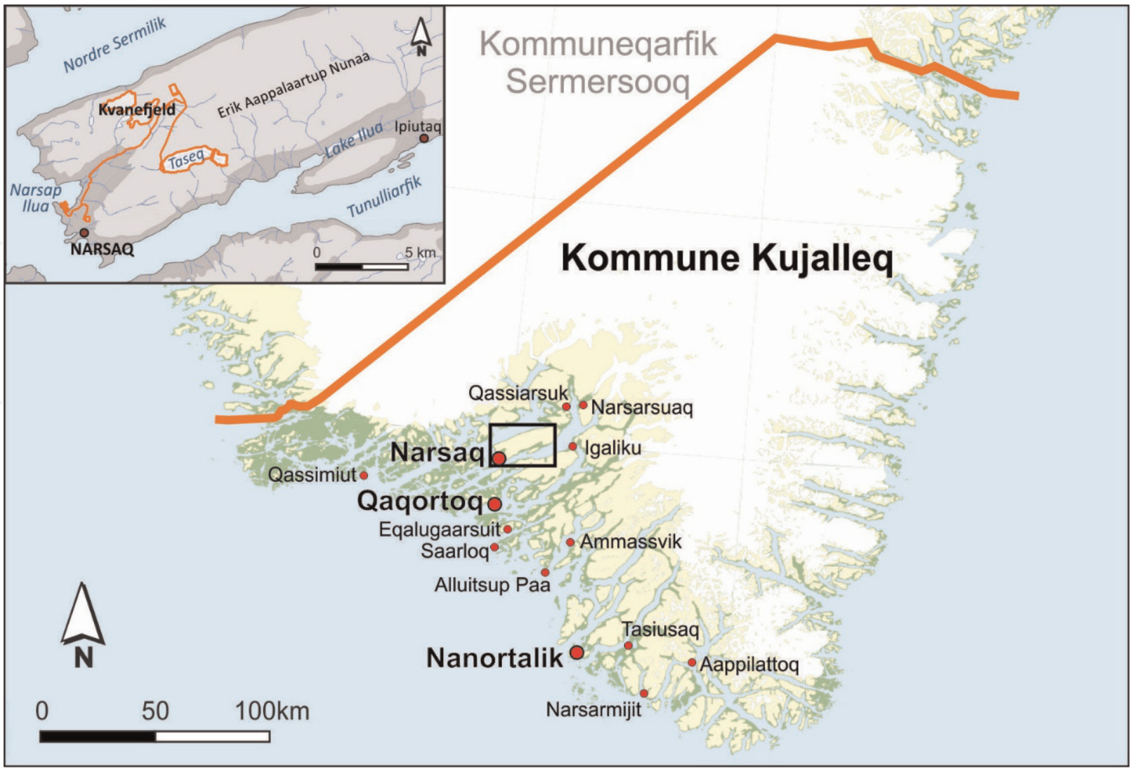

Figure 1.
Map of Kommune Kualleq showing local population centres and project infrastructure.

Greenland Minerals Limited (GML) proposes to develop a mine and integrated minerals processing facility for Kvanefjeld. In addition to producing significant quantities of REE products, the project will also produce, as by-products, small but commercially valuable quantities of uranium, zinc concentrate and fluorspar.

The rare earth elements, in the Kvanefjeld deposit, are contained predominantly in the mineral steenstrupine, which has the chemical formula provided below:

$$Na_{6.7}H_xCa(REE)_6(Th, U)_{0.5}(Mn_{1.6}Fe_{1.8}Zr_{0.3}Ti_{0.1}, Al_{0.2})(P_{4.3}Si_{1.7})O_{24})(F, OH).nH_2O.$$

It contains all 15 rare earth elements, radionuclides, phosphorous and soluble silica. Rare earths are also found to a lesser extent in the minerals townendite, monazite and vitusite [2].

Beneficiation of steenstrupine from the gangue minerals is possible via the froth flotation method. Froth flotation can be used to produce a steenstrupine mineral concentrate which typically grades 14–23% rare earth oxide [3, 4]. Gangue minerals such as felspars, feldspathoids, agerine and amphiboles are extensively rejected and report to the tailings stream. This paper will focus on the hydrometallurgical treatment of flotation concentrates as this is the typical process for rare earth recovery. The inventive aspect is in the definition of process variables which result in a leaching process which produces high extractions while exhibiting good solid liquid separation characteristics of leach residues.

1.2 Mineral leaching issues

As steenstrupine is a very rare mineral there is little research and commercial precedent for it’s treatment [5]. It contains a high degree of acid soluble silica and nonrefractory rare earth elements which makes it's leaching characteristics unusual. Historical leaching tests were performed on ore samples which contained steenstrupine [6]. This early work showed steenstrupine and some associated gangue minerals are susceptible to silica gelling. Steenstrupine and it's associated gangue silicate minerals contain acid soluble silica which dissolves into solution with acid leaching [7, 8].

Other researchers have found silica issues with another alkaline rare earth mineral Eudialyte which also contains significant soluble silica [8–10]. Silica soluble minerals which leach under acidic conditions will release silica into solution. If not carefully controlled, dissolved silica can precipitate rapidly forming a network structure in solution resulting in the solution gelling and exhibiting poor solid liquid separation [11–13].

Some leaching methods have been applied to steenstrupine which include both sulphuric and hydrochloric acids [1, 3, 7]. In 2012 GML developed a method which uses a two stage sulphuric acid leach to extract the values and control silica [7]. Since 2017, GML has been developing a different leaching method which utilises concentrated hydrochloric acid in a single stage leaching process [14]. Previous steenstrupine hydrochloric leach tests investigated low solids loading, relatively dilute acid solution and only batch operation [8]. This was effective in leaching rare earths from steenstrupine however a pyrometallurgical pre-treatment was required. In addition the leach density was moderately dilute.

This paper presents the results of investigating the leach performance at higher solids loading and concentrated acid concentrations without a pre-treatment step. The viability of the process is demonstrated by comparing batch leach results with those of continuous leach results.

2. Samples utilised

2.1 Low grade flotation concentrate

The low grade flotation concentrate was generated from Kvanefjeld ore (Lujavrite) during continuous flotation pilot plant testwork. Mineralogical examination (QEM-SEM) identified steenstrupine, arfvedsonite, aegirine, analcime, K feldspar and plagioclase as the major components of the concentrate [3]. Elemental analysis of the concentrate used a combination of fusion inductively coupled plasma optical emission spectroscopy (ICP-OES) and inductively coupled plasma mass spectroscopy (ICP-MS). The chemical analysis was performed by the commercial SGS Laboratory in Perth, Australia who are certified and ISO accredited. The accuracy of the assays is considered very good with an accuracy of +/− 5% (**Table 1**).

Acid soluble silicon concentration in the concentrate was determined by diagnostic leaching of concentrate with 70 g/L Hydrochloric acid (HCl) for 2 hours at ambient

La	Ce	Nd	Pr	Y	U
2.08	3.74	1.30	0.40	0.68	0.18
Al	**Fe**	**Na**	**P**	**Si**	**Th**
3.91	9.27	8.29	2.19	19.5	0.52

Table 1.
Low grade concentrate assays (% of mass).

La	Ce	Nd	Pr	Y	U
4.00	8.00	2.60	0.80	1.40	0.34
Al	**Fe**	**Na**	**P**	**Si**	**Th**
1.5	6.00	8.50	4.00	15.0	1.00

Table 2.
High grade concentrate assays (% of mass).

temperature at 1 and 3% w/w solids. The leach liquor was recovered and filtered through 0.45 microns before diluting 1:10 in deionised water and submitting for assay by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES). The low solids density resulted in low silicon tenors in solution which prevented silica gelling issues. The acid soluble silicon concentration in the concentrate was determined to be 38 g of silicon per kilogramme of concentrate.

2.2 High grade flotation concentrate

Batch testwork on Kvanefjeld ore using an improved flotation process [3] generated a limited quantity of high-grade flotation concentrate which was available for leach testwork. The concentrate elemental composition was determined using a combination of sodium fusion and inductively coupled plasma. The elemental assays are shown in **Table 2**.

The acid soluble silicon concentration in the concentrate was determined to be 32 g of silicon per kilogramme of concentrate solids.

3. Leaching methods

3.1 Batch leach tests

High acidity and high temperature are known to assist with the precipitation of silica in a solid crystalline form rather than polymerising to form silica gel [11–13]. Higher leach slurry densities offer higher acid concentrations in solution and lower mass throughputs. Lower mass throughputs reduce construction costs for the leaching equipment. Concentrated HCl was selected to produce a high solids density and high acidity in the leach. The use of concentrated HCl will increase the temperature of the leach due to the heat generated by acid dilution.

The program focused on leaching low grade concentrate under various atmospheric conditions with hydrochloric acid to determine metal dissolution and the relative filtration performance of the resultant leached slurry. The general acid leach test procedure is shown in **Figure 2**. All acid doses are quoted in grams of anhydrous HCl per gram of dry concentrate.

The concentrate was leached by manually mixing concentrate and concentrated HCl (20–29% w/w HCl) for 15–30 minutes. Dilute HCl (10 g/L HCl) is then added to generate a slurry which is sufficiently fluid to enable it to be poured onto a vacuum filter. The quantity of 10 g/L HCl added to the leach slurry was varied so that the mass of slurry and the slurry % solids to the filter was constant regardless of the acid concentration or acid dose used in the leach stage. The diluted slurry was then vacuum

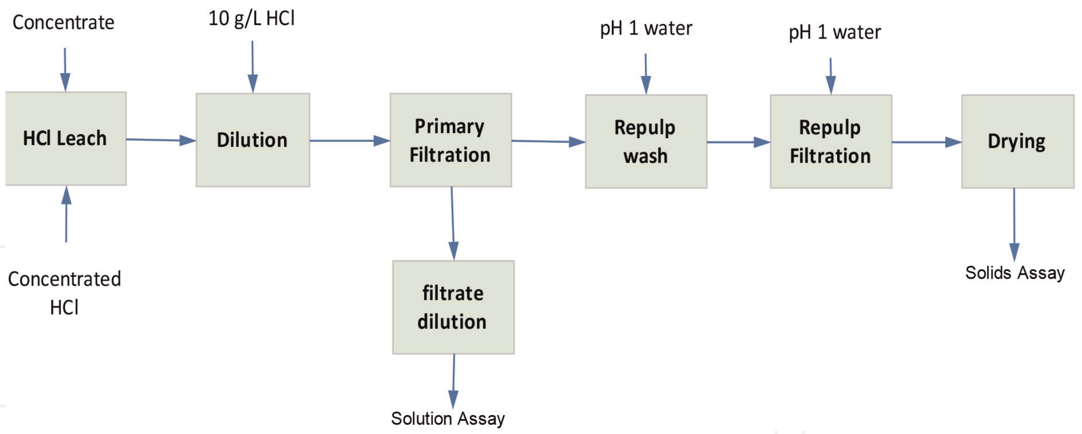

Figure 2.
Batch laboratory work flow.

filtered and the cake form time determined. Filter cake form time was used as a proxy for filtration rate.

The filtrate was collected and a subsample immediately diluted 1:10 with 1 M HCl, to stabilise the sample prior to submitting for elemental analysis. The filter cake was dispersed in D.I water which had been acidified to pH 1 (HCl) and vacuum filtered. Once the surface moisture had disappeared from the filter cake, additional pH 1 wash water was applied to the cake to remove entrained dissolved salts. The solids were then dried to constant mass at 80°C and submitted for elemental analysis.

3.2 Continuous leach tests

Continuous leach tests were conducted once suitable conditions for concentrate leaching were identified from the batch testwork program. Dry concentrate was continuously fed (vibratory feeder) to a 5.8 L stirred baffled reactor. Concentrated (25% w/w) HCl was continuously pumped in via a peristaltic pump to achieve the required acid dose. The reactor temperature was maintained at 80°C using indirect steam through titanium steam coil. A thermocouple was inserted into the slurry and indirect steam added through a solenoid valve connected to an automatic controller.

The reactor had a nominal residence time of between 69 and 97 minutes depending on feed flowrates. Samples were taken from the reactor overflow and prepared for assay as previously described.

3.3 Continuous leach tests

Analysis of the solid samples was performed using a four-acid digest followed by ICP-OES and ICP-MS finishes. The digest solutions were appropriately diluted before being analysed. Solid samples were also analysed by sodium peroxide fusion digest, followed by an analysis of the resulting solutions by ICP-OES and ICP-MS. Solids were assayed using sodium fusion to ensure any refractory minerals were dissolved. Good agreement was achieved between the four acid digest and peroxide fusion methods and suggests that any refractory minerals present in the sample was minimal.

Elemental assays of liquors were determined by ICP-OES and ICP-MS. The liquors were diluted 1:10 with 1 M HCl prior to submission to stabilise silicon in solution.

Free acid was determined by titration with standard NaOH using Calcium EDTA as a complexing agent, the procedure was modified from that described in [15] and

Figure 3.
Continuous leach set up.

required correction for phosphoric acid in the sample, based on the phosphorous assay of the solution (**Figure 3**).

4. Results and discussion

4.1 Batch test results

Four tests were performed to examine the impact of acid dose on metal dissolution and solid liquid separation of the leached slurry. In each test concentrate was mixed with 29% w/w HCl for 30 minutes. The impact of acid dose on filtration rates is provided in **Table 3**.

The metal leach extraction for the four batch tests are shown in **Table 4**. They show very high extraction of rare earth elements. Gangue elements such as aluminium and iron are lower. This is due to the deportment of these gangue metals in the

Test	Acid Dose (g HCl/g con)	Cake Form Time (sec)	Terminal Free Acid (g/L HCl)	Gell Formed
1	0.48	96	119	No
2	0.38	180	89	No
3	0.35	282	68	Yes
4	0.32	642	47	Yes

Table 3.
Acid dose and solid liquid separation performance.

Test	Al	Fe	Na	P	Th	La	Ce	Nd	Y	U
1	64	13	64	94	90	96	98	98	96	84
2	63	11	64	93	87	95	97	97	94	87
3	63	12	64	93	84	95	97	97	95	84
4	63	11	63	91	79	95	96	96	95	85

Table 4.
Low grade concentrate metal dissolution for batch tests.

minerals leached. Most of the iron is found in the black amphibole arfvedsonite which appears to be refractory in the leach process. The refractory aluminium and sodium may be present in sodic feldspar minerals which do not leach extensively under these leach conditions.

Due to the high slurry density it was important to observe the viscosity of the slurry mixture after the addition of concentrate and acid. For the first 1–2 minutes of mixing the mixture would remain fluid, however after this period the free liquid would disappear and the viscosity of the mixture increased to the consistency of damp solids. The damp solid became hot and with continued mixing, formed a viscous paste which progressively became more fluid with time. These observations have also been reported when leaching eudialyte concentrates [16] with strong acid.

Figure 4 shows there is very little impact of acid dose on the dissolution of metals except thorium over the range investigated. Under all acid doses near complete extraction of rare earths and uranium was observed.

The cake form time (filtration rate) shows a strong correlation with acid dose. As the acid dose increased the cake form time decreased, demonstrating an improvement in filtration. At the higher acid doses no gel formation was observed. Both lower acid doses resulted in gel formation indicating that lower acid conditions result in favourable conditions for silica polymerisation. At higher acid doses and therefore higher acid strength in solution may result in a more crystalline silica precipitate.

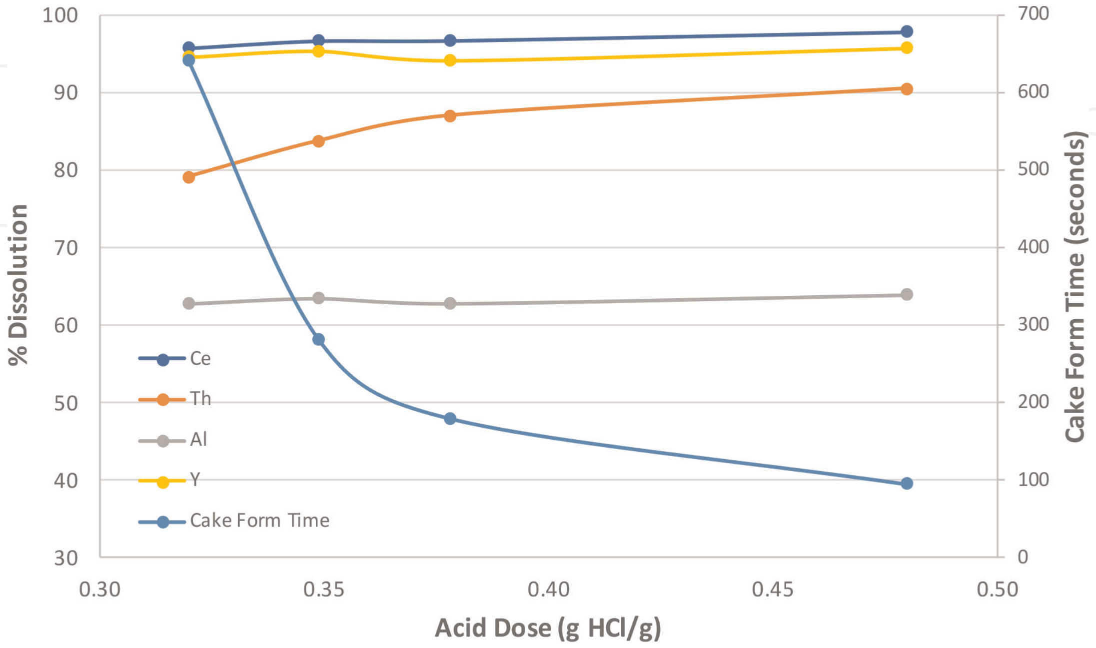

Figure 4.
Acid dose effects on dissolution and filtration.

The reduced filtration rate and the appearance of silica gel at the lower acid doses are all consistent with reduced rates of silica coagulation [12, 13, 17]. The rate of silica coagulation from solution has been shown to be strongly influenced by a number of factors including temperature, the presence of metals in solution and free acid [17, 18]. Generally, as temperature increases the rate of silica precipitation increases while the presence of aluminium in some systems appears to increase the rate of silica precipitation. In these series of tests, the aluminium tenor and the temperature were similar over the acid dose range investigated. As the terminal free acid increased the solid liquid separation improved. The results are consistent with improved silica coagulation at higher acid concentrations resulting in improved filtration.

Over 99% of the acid soluble silicon had precipitated under all the test conditions. Terminal free acid concentrations down to 47 g/L HCl are still capable of achieving high rare earth dissolution. A stoichiometric acid balance was performed to determine the elemental acid consumption. Most of the acid (~60%) was consumed in the dissolution of Al, Na and Fe. A significant proportion (15–25%) of the acid added is not consumed in the reaction and remains in solution as free HCl. The majority of the balance of the hydrochloric acid dose is consumed by rare earth dissolution.

4.2 Impact of addition method

Previous tests were typical batch tests where all the acid and concentrate are mixed together at the start of a test. While this is a convenient procedure it can produce different results to a continuous process which represents a commercial operation. Continuous leach tests where precipitation of reactants occurs can result in significantly more heterogeneous precipitation. Heterogeneous precipitation products can have different particle size and morphology compared to homogeneous precipitation products [19].

Four tests were performed to examine acid addition methods and more continuous leach conditions. Continuous operation was simulated by dosing the reactants to the leach in staged doses during the leach. The acid dose (0.38 g/g), acid concentration (29% w/w HCl), average leach time (30 minutes) and temperature (ambient) were kept constant between tests.

The additions to the reactor were as follows:

Test 9–100%, all acid and concentrate added at the start (baseline test).

Test 11–80% of the concentrate added at the start, remainder after 10 minutes.

Test 14–40% of the concentrate added at the start, remainder after 10 minutes.

Test 15 - a third of the acid and concentrate added every 5 minutes to the reactor.

Table 5 shows that the method of feed addition had little impact on metal extraction with constant acid dose. The filtration of the residues was significantly impacted by the timing of acid addition as observed in **Figure 5**.

The smaller incremental additions to the reactor are more representative of a continuous leach reactor. The results show that cake form time decreases (better filtration) as the proportion of sample added to the reactor at the start of the test fell. These are consistent with increases in heterogeneous precipitation and particle growth expectations. As incremental additions are made to the reactor; silica dissolves and re-precipitates on precipitated silica from earlier reactor additions, increasing silica precipitation particle size. Incremental feed addition to the batch reactor results in a substantial improvement in the filtration properties of the leach slurry.

Test	Al	Fe	Na	P	Th	La	Ce	Nd	Y	U	CFT* (sec)
1	63	12	64	93	84	95	97	97	95	84	245
2	63	12	64	92	84	95	96	97	94	83	220
3	63	13	63	92	85	95	96	96	93	82	106
4	63	12	63	92	85	95	96	96	93	82	52

**CFT = Cake Form time in seconds.*

Table 5.
Impact of acid addition method.

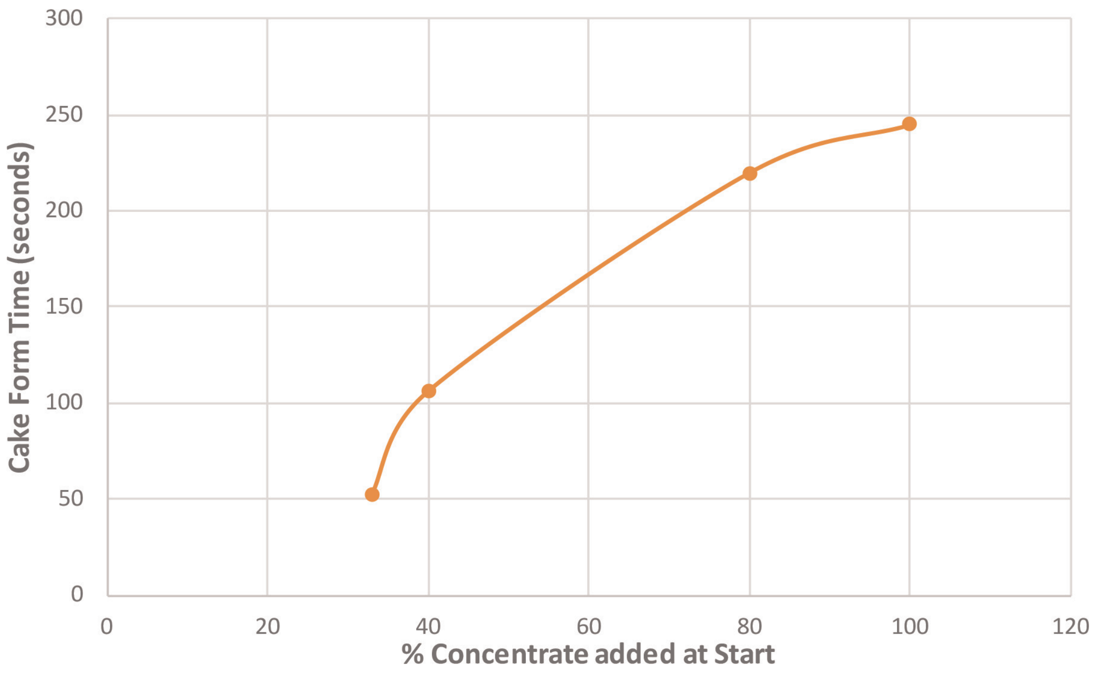

Figure 5.
Dissolution and filtration versus acid dose.

4.3 Impact of acid concentration

Three tests were completed where the acid concentration was varied while maintaining a constant acid dose of 0.38 g HCl /g. The leach mixture was maintained at 55°C in a water bath. To better reproduce the conditions observed in continuous operation the acid and concentrate were added incrementally to a stirred reactor. The first two tests were under the same conditions except the second test used a higher acid concentration (but same acid dose). The third test was a repeat of the first test except a longer residence time (slower addition rate) was employed. The results of these tests are shown in **Tables 6** and 7.

All tests gave similar metal extractions except for thorium which shows a significant drop in dissolution at the lower terminal free acid concentration (lower acid concentration in feed).

The acid strength had the greatest impact on the physical performance of the leach, with gel formation at the lower acid concentration which increased filtration time. Increasing leach residence time, from 32 to 69 minutes (Test 20) overcame the silica

Test	Residence Time (min)	HCl Conc. (%)	Cake Form Time (sec)	Terminal HCl	Gel Formed
18	32	96	119	No	Yes
19	32	180	89	No	No
20	69	282	68	Yes	No

Table 6.
Impact of acid concentration on solid liquid separation.

Test	Al	Fe	Na	P	Th	La	Ce	Nd	Y	U
18	63	11	62	91	78	95	96	96	97	84
19	63	11	63	92	80	96	97	97	96	83
20	63	12	62	91	88	95	95	96	96	85

Table 7.
Impact of acid concentration on metal extraction (% from concentrate).

gelling issues resulting in improved filtration rate. These observations are consistent with the expected impact of dissolved silica precipitating as a polymerised gel.

The cake form time for test 19 is much shorter (40 seconds) than the comparable batch test (test 2, 180 seconds). This is attributed to the elevated temperature of the acid mixing stage in test 19 and increased heterogeneous silica precipitation when the acid and concentrate are added incrementally to the reactor.

4.4 High grade concentrate test results

Leach tests with high grade concentrate were completed to confirm the general relationships between the various process factors examined in this paper. The concentrate and acid were mixed using the simple batch contact and the incremental addition procedure. The acid mixing stage was maintained at 65°C with the acid added as 25% w/w HCl. Tests were conducted to identify the impact of acid dose, acid concentration and addition method on metal dissolution and filtration rate.

Metal dissolution at the lower acid dose was significantly lower regardless of addition method. The lower grade concentrate gave significantly higher metal dissolution at the 0.38 g HCl/g dose, this is attributed to the refractory nature of the gangue minerals in the low-grade concentrate which do not consume significant quantities of acid. Gangue minerals such as feldspar and arfvedsonite are only significantly present in the lower grade concentrate.

At the lower acid dose **Table 8** reveals the incremental addition method resulted in significantly better filtration which is consistent with results for the low grade concentrate.

At the higher acid dose, rare earths, uranium and thorium recovery increased while aluminium, iron and sodium dissolution were unchanged (**Table 9**). Filtration was significantly better at the higher acid dose while the method of acid addition had no discernible impact on filtration rate. The results are consistent with the higher rate

Acid Addition Type	Acid Dose (g HCl/g)	Cake Form Time (sec)	Terminal HCl (g/L)
Batch	0.38	280	40
Batch	0.53	59	83
Incremental	0.37	75	40
Incremental	0.53	57	84

Table 8.
Impact of acid strength on the solid liquid separation of leached high grade concentrate.

Addition type	Acid dose (g/g)	Elemental extraction in %									
		Al	Fe	Na	P	Th	La	Ce	Nd	Y	U
Batch	0.38	62	17	78	65	11	81	76	73	89	85
Batch	0.53	60	15	76	89	75	94	94	94	96	88
Incremental	0.37	61	15	78	67	20	81	77	74	90	83
Incremental	0.53	61	16	78	89	71	94	95	94	98	89

Table 9.
High grade concentrate metal extraction (% dissolved from concentrate solids).

of silica coagulation at the higher terminal free acid (higher acid dose) giving better filtration rates.

Metal recovery remains unchanged between tests except for thorium which shows a slight drop when a lower acid concentration was used and is attributed to the lower terminal free acid.

4.5 Continuous leach results

Dry low grade concentrate and 25% w/w HCl were continuously added to a stirred reactor maintained at 80°C. Flowrates were controlled to maintain the target acid dose and nominal residence time. The test was undertaken to determine metal dissolution and observe the viscosity of the slurry in the leach reactor.

The first continuous test targeted a nominal residence time of 97 minutes at an acid dose of 0.46 g HCl/g concentrate. The second continuous test decreased the nominal residence time to 69 minutes at an acid dose of 0.39 g HCl/g concentrate. The test results of the continuous tests and the comparable batch test are presented in **Table 10**.

Table 11 shows the metal leach extraction was similar between the continuous and batch leach tests. The filtration rates recorded in **Table 10** were consistently better for the continuous tests at similar acid dose. The continuous leach test observed a significantly lower leach viscosity compared to the batch tests. While the slurry from the batch tests had a toothpaste consistency the slurry from the continuous test was more fluid and was able to freely overflow the reactor.

In batch tests the acid/concentrate mixture initially formed a damp solid which is difficult to agitate effectively. The damp solid did not flow but as mixing continued it became more fluid achieving a consistency similar to toothpaste. If high viscosity is

Test type	Residence time (min)	Acid dose (g HCl/g)	Cake form time (sec)
Continuous	97	0.46	52
Continuous	69	0.39	91
Batch	30	0.47	96
Batch	30	0.38	180

Table 10.
Comparison of continuous and batch leach methods impacts on solid liquid separation.

Test type	Acid dose (g/g)	Elemental extraction in %									
		Al	Fe	Na	P	Th	La	Ce	Nd	Y	U
Continuous	0.46	61	12	62	91	89	94	97	98	98	89
Continuous	0.39	60	13	65	92	81	94	96	96	97	86
Batch	0.47	64	13	64	94	90	96	98	98	96	84
Batch	0.38	63	11	64	93	87	95	97	97	94	87

Table 11.
Comparison of metal extractions from batch and continuous leach conditions.

observed in the continuous operation it will result in more expensive and specialised leaching equipment. The low viscosity slurry observed in the continuous tests will allow for the use of standard Continuously Stirred Tank Reactors (CSTRs) which are low cost, low risk and industry standard (**Figures 6** and 7).

Figure 6.
Viscous leach conditions from batch test.

Figure 7.
Fluid leach conditions from a continuous test.

5. Conclusion

Leaching of steenstrupine concentrate was successfully achieved from both low grade (10% REO) and high grade (21% REO) concentrates. Leaching tests show increasing acid concentrations in solution improved the solid liquid separation and yielded high rare earth extractions. Incremental addition of leach feed to the reactor had little impact on metal dissolution but improved leach residue solid liquid separation significantly. The optimum acid dose was in the range of 0.39 to 0.53 t anhydrous HC/t of dry concentrate. Acid doses lower than 0.35 t anhydrous HCl/t dry concentrate result in poor filterability despite excellent leach extraction.

Filtration characteristics also improved when leaching was performed in a continuous reactor as opposed to a batch reactor. Increasing residence time in the leach also improved filtration characteristics at constant acid dose. Under the same acid regime the batch process gave a very viscous slurry while the continuous system gave a free flowing slurry.

High acid strength, longer residence time and continuous operation improve are the most important factors to produce a leach slurry which is suitable for solid liquid separation.

Further research could be performed to examine the impact of temperature on the solid liquid separation efficiency.

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