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Leaching the Unleachable Mineral: Rare Earth Dissolution from Monazite Ore in Condensed Phosphoric Acid

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Abstract: Monazite is a poorly soluble mineral of rare earth phosphate. It is an ore of the rare earths which is difficult to break down; in industry either concentrated sulphuric acid or caustic soda is used to attack finely ground monazite at between 140 °C and 400 °C. In these processes, the rare earths are converted into different solid compounds, undergoing an incomplete conversion. Here we show a new process for a direct and much faster breakdown of monazite by simple dissolution under milder conditions. Condensed phosphoric acid was used to dissolve rare earths (up to 96 g/L) from unground monazite sand from four sources. Greater than 99% of light rare earths dissolved within 30 min at 260 °C. The cooled solution can be diluted to an extent with water to reduce viscosity for analysis or further processing. This method of dissolution avoids the use of strong acids/bases and reduces the risk of dusk exposure from fine grinding of particles.

Keywords: rare earth; leaching; phosphoric acid; condensed; monazite; sand

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1. Introduction

Rare earth elements (also known as rare earth metals, lanthanides, lanthanoids, or lanthanons) are generally taken as the series of increasing atomic numbers from lanthanum to lutetium, as well as yttrium and scandium. Rare earth metals can be obtained from ores of phosphate minerals such as monazite. Monazite is a solid solution which is produced globally as a by-product of heavy mineral sand operations. It comprises mainly the lighter rare earths in terms of atomic mass, from lanthanum to gadolinium, with some yttrium as well [1]. Monazite in its ideal form has one rare earth element for every phosphate but may also have a significant quantity of calcium and radioactive thorium [2], which, alongside the refractory nature of the ore, poses a challenge to the production of rare earths from monazite.

Presently, there is a strong demand for the lightweight rare earth magnets used in the motors of electric vehicles. These magnets contain neodymium and praseodymium. The market prices for neodymium and praseodymium are much higher than those for cerium and lanthanum found in the same ores [3]. The prices of cerium and lanthanum are currently less than the price of mixed rare earth carbonate [4] which has not undergone separation into individual rare earths. It could be argued that the industrial separation of lighter rare earths is largely driven by the demand for the magnet metals, neodymium, and praseodymium.

There are presently two conventional methods used in industry for the breakdown of monazite. One method, known as caustic digestion, involves the mixing of fine particles of monazite with 70% sodium hydroxide at temperatures higher than 140 °C to produce a solid rare earth hydroxide [1]. The other method, known as sulphuric acid baking, mixes fine particles of monazite with 93% sulphuric acid at between 200 °C and 400 °C to produce solid rare earth sulphates [1]. Both methods involve converting the rare earths on the surface of the particle from one solid and ultimately forming another solid, which can subsequently be dissolved.

These reactions typically require fine particles to maximise surface area and subsequent recoveries of the rare earths. Generally, monazite must be ground finely [5], unless the ore is naturally very fine. The monazite must also be attacked for an extended residence time of multiple hours [1] to allow time for the conversion reaction. Fine grinding and extended residence times mean that a grinding mill and larger vessel sizes are needed on processing plants, increasing operational and capital costs. A further problem with finely ground monazite is the safety hazard posed by the monazite dust. Monazite is weakly radioactive mainly due to the presence of thorium and uranium. The radiation given off by thorium is particularly dangerous if the monazite dust is inhaled or ingested [6].

A third key disadvantage of the conventional processes are the safety and environmental hazards introduced by the chemicals added to the process. The concentrated strong acids or bases are extremely hazardous at all temperatures. Sulphuric acid in particular decomposes upon heating to a gas [7]. It also adds sulphates to the tailings which may then be introduced to the environment. The conventional processes also require further steps of neutralisation and carefully controlled filtration [1] to ameliorate the extreme conditions and handle the solids produced, respectively.

To avoid the key issues of the current industrially applied processes, an alternate direct leaching process has been investigated using condensed phosphoric acid (a moderate acid) in place of the strong acids or bases which have conventionally been used. The aim of this investigation is to present more rigorous evidence relating to the dissolution process outlined in an application under the Patent Cooperation Treaty with a priority date of 21 November 2019 [8]. Whereas the application [8] roughly shows the optimum temperature range for dissolution at about 265 °C, this investigation seeks to test condensed phosphoric acid on unground monazite sand from a variety of different sources, with redundancies in the analysis for an understanding of error.

Condensed phosphoric acid is also known as "strong" phosphoric acid in the literature. It is a mixture of orthophosphoric acid and the polyphosphoric acids. Heating a mixture of orthophosphoric acid and water to higher than about 180 °C will result in pyrophosphoric acid due to the condensation reaction of two orthophosphoric acid molecules [9].

$$2H_3PO_4 \rightarrow H_4P_2O_7 + H_2O \tag{1}$$

Condensation reactions continue with higher temperatures to produce more "condensed" phosphates as shown in Table 1 [9].

Table 1. Condensation of phosphoric acid with temperature [9].

Temp (°C)	Orthophosphoric Acid (wt.%) (H_3PO_4)	Pyrophosphoric Acid (wt.%) ($H_4P_2O_7$)	Triphosphoric Acid (wt.%) ($H_5P_3O_{10}$)	Tetraphosphoric Acid (wt.%) ($H_6P_4O_{13}$)	Water (wt.%)
161	86.3				13.7
183	90.9	1.1			8.0
203	89.9	4.4			5.8
220	90.5	4.2			5.3
244	90.0	5.0			5.0
260	89.8	8.1	0.3		1.8
280	66.1	29.3	4.6		
301	53.8	39.5	6.8		
340	29.4	47.4	22.7	0.5	

Table 1 shows that up to temperatures of 260 °C, orthophosphoric acid will comprise about 90% of the condensed phosphoric acid by weight. Dehydrated orthophosphoric acid has previously been shown to precipitate rare earths such as monazite at a temperature of 160 °C [10]. A higher temperature of 260 °C would be expected to further reduce the solubility of rare earth phosphates which tend to decrease in solubility with higher temperatures [11], making monazite dissolution under these conditions appear unlikely.

The only difference in the composition of the fluid at 160 °C and 260 °C is the smaller proportion of water and the presence of a minor fraction of pyrophosphoric acid.

Pyrophosphoric acid is known to be a stronger acid (pKa 0.91) than orthophosphoric acid (pKa 2.61). It was initially hypothesised that the presence of pyrophosphoric acid may increase the acidity of the solution to allow the orthophosphate from the monazite to become protonated while dissolving the rare earths. However, it is only a moderate acid, and stronger acids such as hydrochloric and nitric acids are ineffective at dissolving monazite to a high concentration of rare earths. Since acid strength alone is unlikely to be sufficient for the dissolution of monazite, it is hypothesised that there may be a role for complexation between the pyrophosphate species and the rare earth ions. Unfortunately, no studies could be found on pyrophosphate complexes in solution conditions with a pH lower than 1.

2. Materials and Methods

Three concentrates of monazite sand were obtained from Sakorn Minerals in Thailand with three different origins: Perth and Bunbury within Western Australia, and Mozambique. Another stock was obtained separately and was a mix of monazite and zircon sand with an origin from the east coast of Australia within Queensland. From each stock, three separate samples of 3 g each were obtained by grab sampling with a spatula and set aside for characterisation. X-ray diffraction was used for mineral identification. Solid assays were performed by lithium borate fusion followed by dissolution and analysis by ICP-MS.

About 56 g of analytical grade orthophosphoric acid (85% *w*/*w*) was added to a 50 mL borosilicate beaker. A 30 mm Teflon magnetic stir bar was also added to the beaker. The beaker was put on a heater stirrer with stirring on a minimum to circulate the solution and prevent temperature gradients. The leach solution was heated as quickly as possible to a target temperature of 260 °C to dehydrate the solution and form condensed phosphoric acid with a volume of about 28 mL. The reason for the fast heating of the solution is that the walls of the borosilicate beaker may decompose in the condensed phosphoric acid; after more than about five hours a precipitate will form in the solution even if no other substance had been added to the phosphoric acid in the beaker.

Once the solution reached the target temperature of 260 °C, the stirrer was turned up to 600 rpm and either 6.0 or 8.0 g of monazite concentrate was added to the solution. After the leaching time had elapsed, the stirrer and hotplate were turned off and the leachate was decanted into another larger borosilicate beaker leaving a solid residue behind in the original beaker. The decanted solution and the solid residue were left to cool to ambient temperature. Once cooled, about 60 mL of ultra-pure water from Pureau was poured on top of the acidic, decanted solution, which was now quite viscous. The two phases were then mixed together to form a greenish solution. Part of this solution was mixed with the solid residue of the leach and poured back into the main solution. This was to help to remove and recover any viscous, leaching residue remaining with the solids. More ultra-pure water was added and mixed until the total volume of the solution was close to 100 mL.

Enough water needs to be added so that the heat generated by mixing the water and acid can be absorbed without a temperature rise sufficient to cause a precipitation reaction. When the above method is used, the temperature rises from ambient temperature to about 60 to 70 °C and the solution remains stable without any precipitation of the rare earths. The solution can then be left to cool without precipitation. It is important to note that a mixture of water and condensed phosphoric acid will have a boiling point higher than 100 °C. Mixing a small amount of water and condensed phosphoric acid may cause a temperature high enough to rapidly reverse the condensation of the phosphoric acid and cause the rare earths to precipitate.

It should also be noted that the diluted leaching solution is not thermodynamically stable. The solution will deteriorate, and the rare earths will precipitate after some time. It has been observed that a concentration of rare earths of about 90 g/L in the leaching solution, diluted as above, will lead to precipitation within about a week at ambient temperature. A lower concentration of rare earths tends to remain stable for a longer period.

The actual final diluted leach solution volume was recorded and then a sample of the leach solution was extracted, filtered, and set aside for analysis by ICP-MS. The solid residue was washed three times with ultra-pure water and placed onto a weighing tray for drying. Once dry, it was weighed and set aside for analysis.

Measurements were checked using a mass balance on the masses of each element in the feed compared to the leaching solution and solid residue combined, and then the percentage dissolution of each element was calculated using two independent methods. One method was to calculate the amount of dissolution from the compositions of the leaching solution and the solid residue. The other method was to calculate the dissolution by the difference between the solid feed and the solid residue.

3. Results and Discussion

The compositional data for each of the monazite-containing sands, and results for the deportment of the rare earths between the leach solution and the residue for each of the ores, are presented below.

3.1. Perth Monazite Sand

3.1.1. Feedstock Analysis

Monazite sands from the Perth monazite feedstock were analysed via XRD (to identify the minerals present) and solid assay (to identify the elemental composition of the ore). Three independent samples of the ore were analysed with XRD. The diffractogram for one of the samples is presented in Figure 1. The diffractograms for the other samples are very similar so have not been included herein.

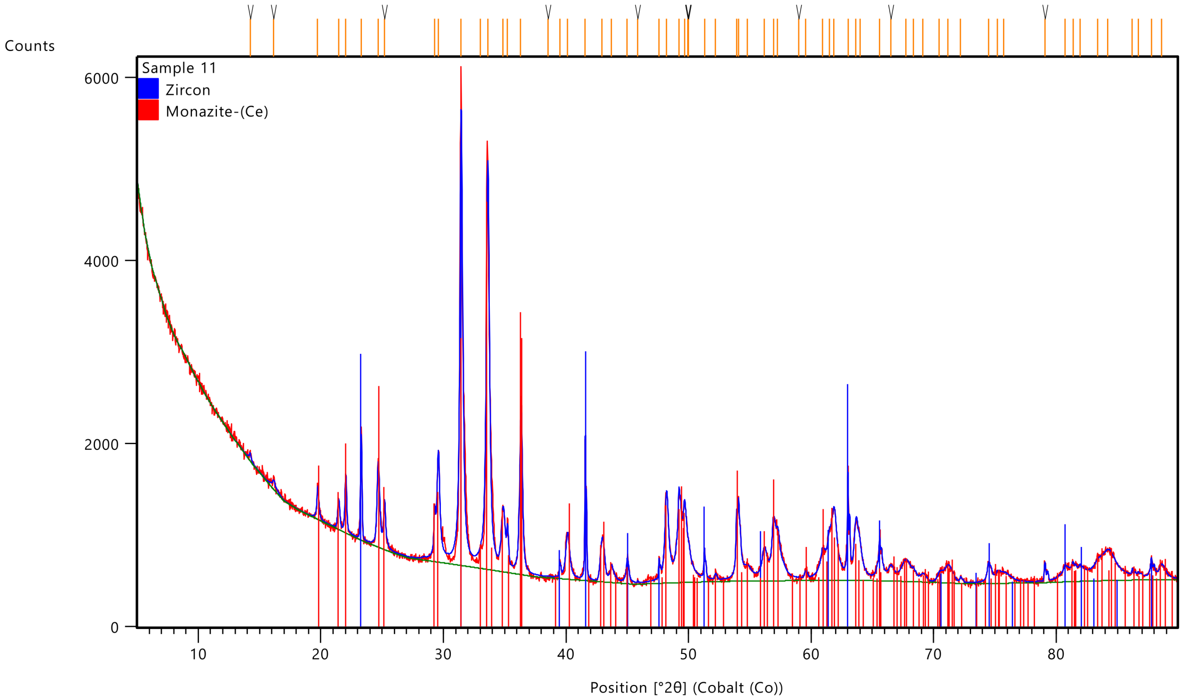

Figure 1. Perth monazite feed XRD output sample 1.

As depicted in Figure 1, the only minerals present in a detectable quantity were zircon and monazite. Note that the XRD results do not convey the relative fraction of each mineral. For this, further analysis via solid assay was required. Three samples of the Perth monazite were assayed for their elemental composition. The data is presented in Table 2.

Table 2. Elemental composition: Assay of the Perth monazite using 3 g samples.

	Sample 1 (wt.%)	Sample 2 (wt.%)	Sample 3 (wt.%)	Average (wt.%)	Absolute Error (wt.%)	Relative Error (%)
La	10.2	10.0	10.3	10.2	0.171	1.7
Ce	20.0	19.9	20.2	20.0	0.192	1.0
Pr	2.21	2.18	2.20	2.20	0.017	0.8
Nd	5.09	5.15	5.07	5.10	0.045	0.9
Sm	1.65	1.61	1.64	1.64	0.022	1.4
Eu	0.021	0.019	0.019	0.020	0.001	6.5
Gd	0.781	0.772	0.782	0.778	0.006	0.8
Tb	0.010	0.009	0.009	0.009	0.001	8.2
Dy	0.343	0.330	0.327	0.334	0.010	2.9
Ho	0.030	0.022	0.023	0.025	0.005	18.5
Er	0.112	0.106	0.104	0.107	0.005	4.3
Tm	0.006	0.004	0.004	0.004	0.001	31.8
Yb	0.018	0.032	0.040	0.030	0.012	40.4
Lu	0.005	0.003	0.003	0.003	0.001	41.1
Y	0.723	0.392	0.534	0.550	0.173	31.5

All light rare earths presented consistent data with low error and thus confidence in these numbers is high. Whilst other elements (Ho, Tm, Yb, Lu, and Y) had higher than ideal relative errors, this was simply due to the low grades of these components within the monazite feedstock, and no adjustments were made to the data.

3.1.2. Dissolution of Rare Earths

Three leach experiments with different residence times (15 min, 30 min, and 60 min) were completed on the Perth monazite. In all experiments an initial mass of 6.0 g of Perth monazite was agitated in about 28 mL of leach solution at 260 °C. The mass of 6.0 g was twice the sample mass used for the solid assays presented in Table 2, so the expected sampling error should be lower. The solution analysis by ICP-MS indicated a rare earth content of 91 g/L to 96 g/L across the three leaching tests.

Figure 2 depicts the dissolution of the light rare earths (La through Sm) in the leach solution for the 15, 30, and 60 min leach tests. The column represents the average dissolution from the two calculation methods described in the materials and methods section and the dots represent the raw outputs from each to provide a range. As the figure shows, there is negligible difference between the two calculation approaches and so there is a high level of confidence in the results. The results indicate the very high and rapid dissolution of light rare earths in the leach solution, with about 95% dissolution within 15 min and greater than 99.8% dissolution within 30 min.

Figure 3 depicts the dissolution of the heavy rare earths (Eu through Lu plus Y) in the leach solution for the 15, 30, and 60 min leach tests. There appears to be lower dissolution of the heavy rare earths than the light rare earths. Dissolution varies from greater than 95% for Eu, Gd, Tb, and Dy in 30 min, to about 40% for Lu in 30 min. The calculation of dissolution based on the feed and solid residue results in considerably higher recoveries for the heavy rare earths from Er to Lu.

Due to the low fractions of heavy rare earths in the feed there is a lot of uncertainty in the data and conclusions therein. However, it appears as though increasing the leach time increases the dissolution of the heavy rare earths. Thus, for monazite sands with substantial heavy rare earth contents, a longer residence time or recirculation of the residue may be warranted to maximise recoveries of the heavy rare earths.

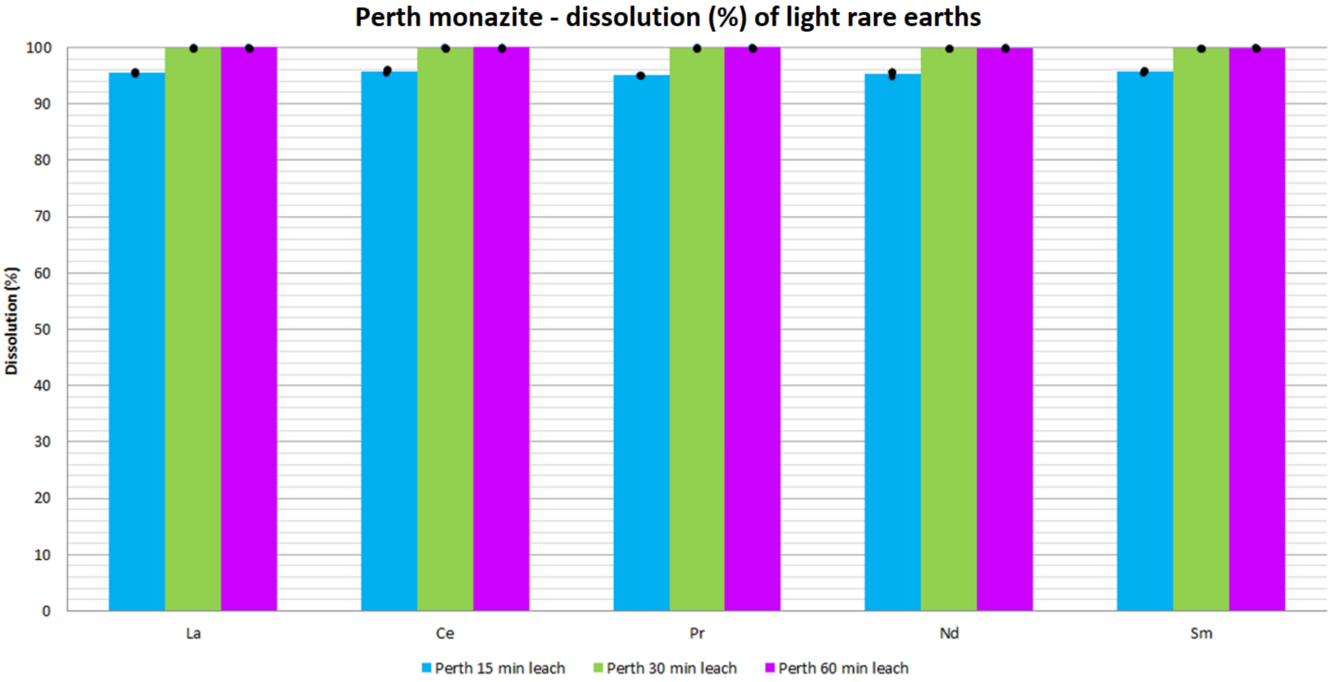

Figure 2. Light rare earth dissolution in leach: Perth monazite (260 °C leach, 215 g/L concentrate).

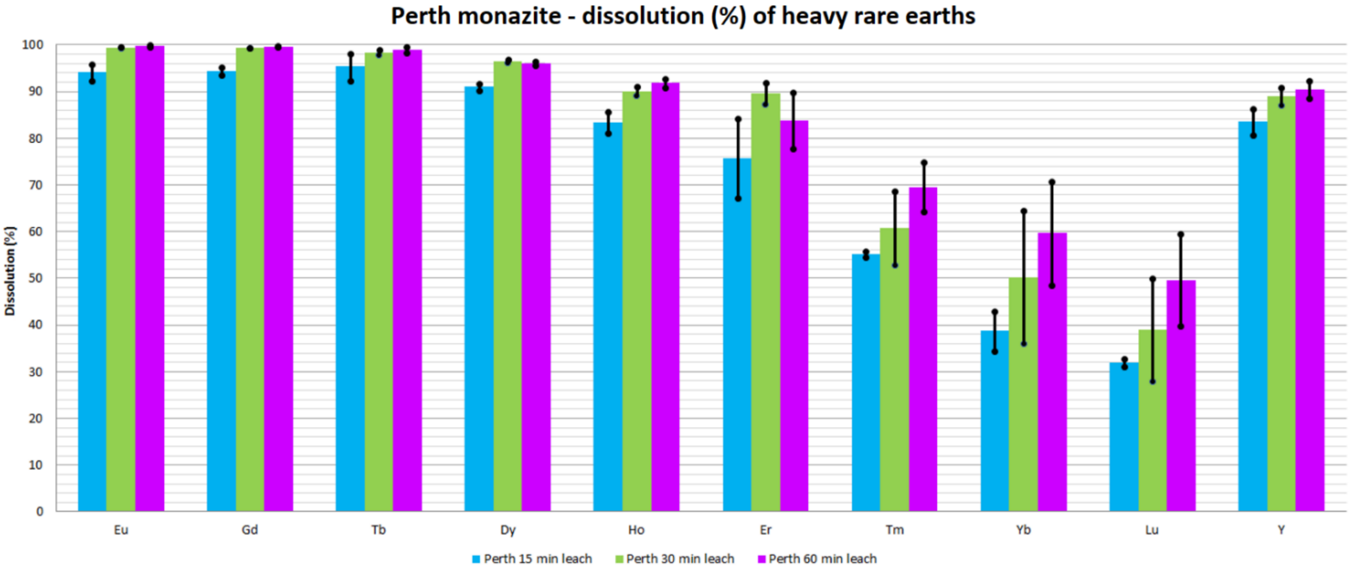

Figure 3. Heavy rare earth dissolution in leach: Perth monazite (260 °C leach, 215 g/L concentrate).

3.1.3. Residue Analysis

XRD diffractograms and solid assays from the residue have also been analysed to confirm conclusions from the ICP-MS analysis. Figure 4 (diffractogram for the 15 min leach) shows that both monazite and the heavy rare earth mineral xenotime are present. However, the diffractograms in Figures 5 and 6 (30 min and 60 min leaches, respectively), show no detectable monazite after 30 min. Xenotime continues to be detectable in the residue for both the 30 min and 60 min leaches, indicating the incomplete breakdown of xenotime. This provides further support for a longer residence time or recirculation for concentrates with substantial heavy rare earths.

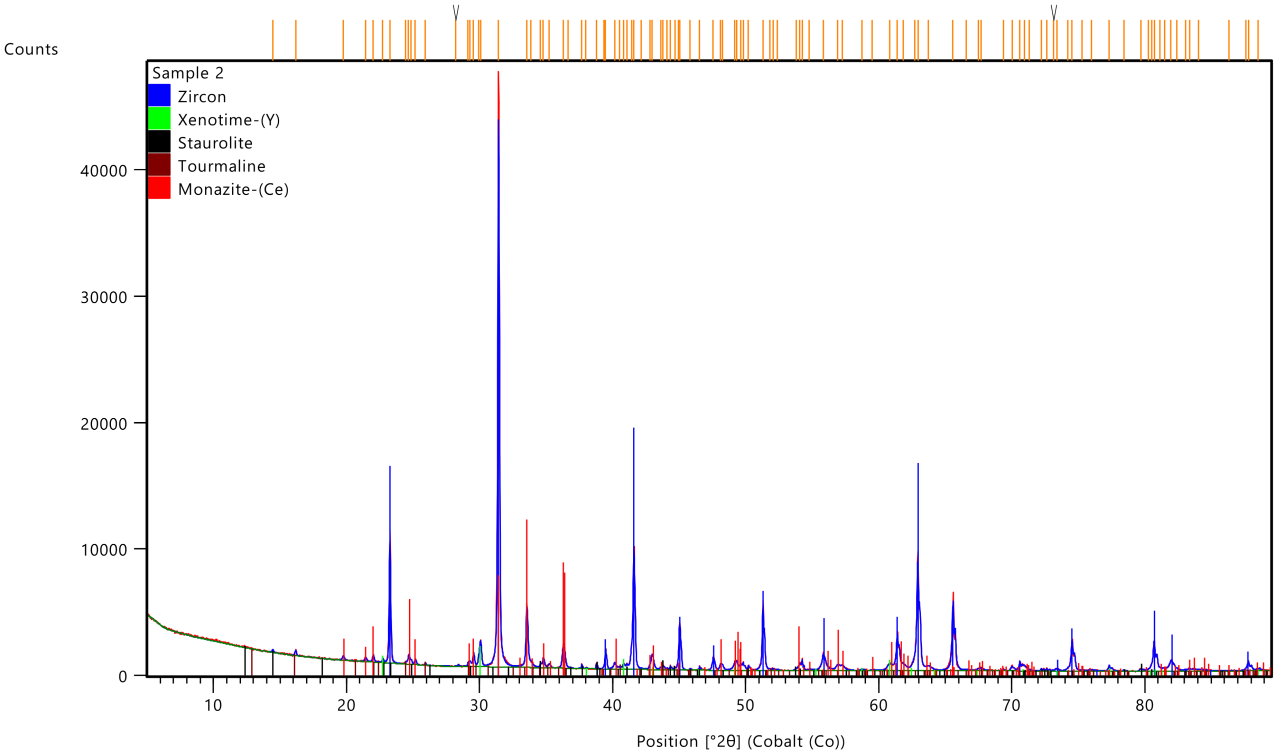

Figure 4. XRD diffractogram: Residue from 15 min leach, Perth monazite.

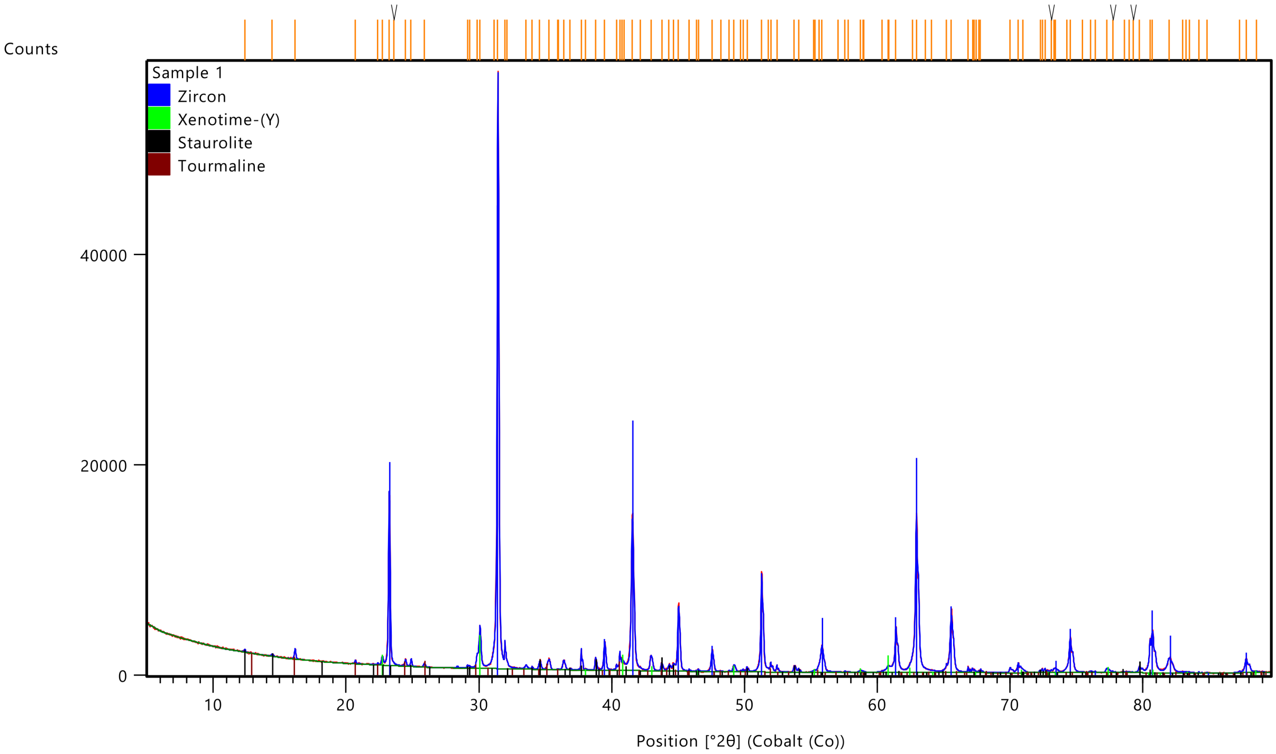

Figure 5. XRD diffractogram: Residue from 30 min leach, Perth monazite.

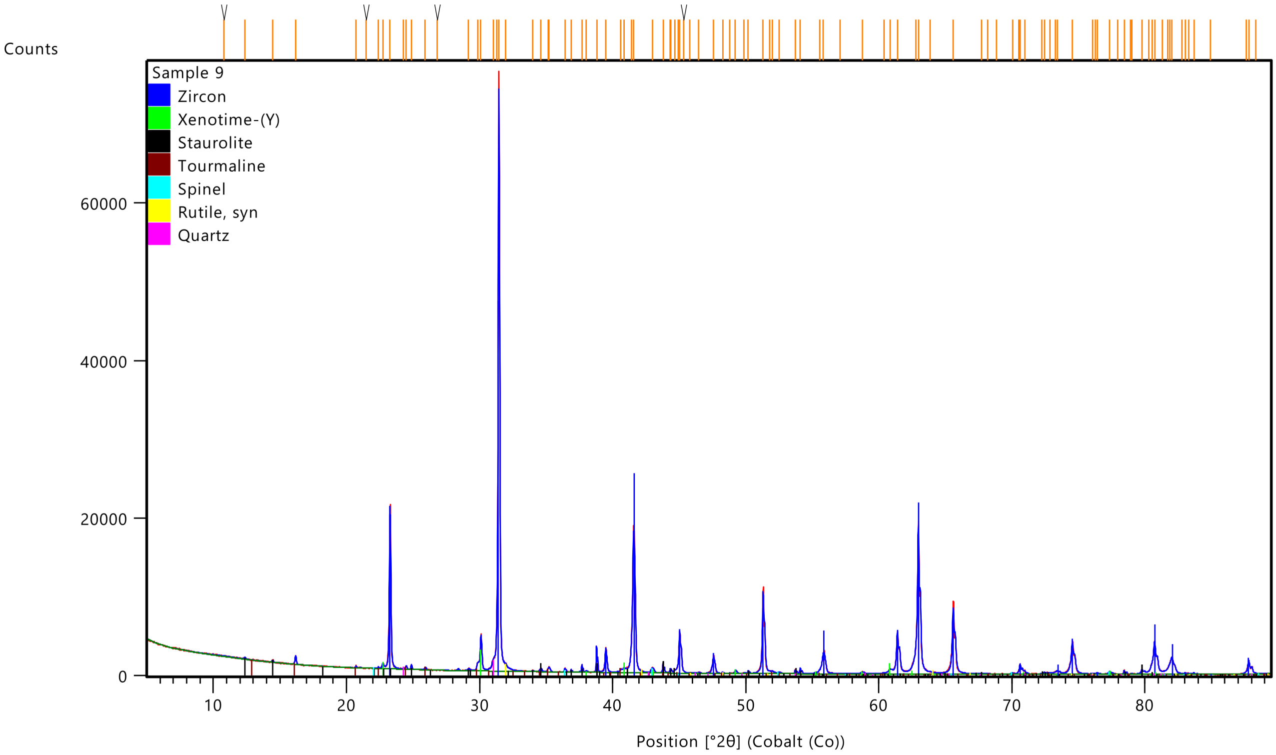

Figure 6. XRD diffractogram: Residue from 60 min leach, Perth monazite.

To confirm insights from the XRD and leach recovery analysis, the solid assay of the residues is provided in Table 3. The mass of residue remaining after 15 min is almost twice that remaining for the 30 and 60 min leaches, indicating the higher overall dissolution of the concentrate with increasing leach time.

Table 3. Rare earths: Mass and elemental composition of residues, Perth monazite (6.0 g feed).

	15 Min Leach	**30 Min Leach**	**60 Min Leach**
Residue mass (grams)	0.8	0.4	0.5
Sample Composition (wt.%)			
La	3.69	0.134	0.007
Ce	7.08	0.325	0.024
Pr	0.793	0.011	0.002
Nd	1.92	0.137	0.011
Sm	0.554	0.041	0.011
Total light rare earths	14.0	0.648	0.055
Eu	0.011	0.002	0.001
Gd	0.282	0.068	0.035
Tb	0.006	0.003	0.002
Dy	0.206	0.157	0.145
Ho	0.036	0.041	0.027
Er	0.127	0.129	0.130
Tm	0.015	0.021	0.014
Yb	0.129	0.161	0.106
Lu	0.018	0.026	0.017
Y	0.791	1.07	0.758
Total heavy rare earths + Y	1.62	1.68	1.24

The 15 min leach still has a considerable light rare earth (monazite) content, but within 30 min the total light rare earth content of the residue is below 0.7% and by the 60 min mark, below 0.1 wt.%, indicating essentially the complete dissolution of light rare earths. From 15 to 30 min the grade of heavy rare earths in the residue is similar, but the overall mass of the residue diminishes and thus there is less heavy rare earths remaining in the residue. By 60 min, the grade of heavy rare earths has decreased.

The overall recovery of the rare earths for each leach duration is provided in Table 4. The numbers presented provide the average of the two calculation methods with the range representing the variation between the methods.

Table 4. Overall light and heavy rare earth dissolution (%): Perth monazite.

	Recovery of Light Rare Earths (%)	**Recovery of Heavy Rare Earths + Yttrium (%)**	**Recovery of Total Rare Earths (%)**
15 min leach	95.5 ± 0.3	88.2 ± 0.2	95.2 ± 0.3
30 min leach	99.9 ± 0.0	94.1 ± 0.1	99.6 ± 0.0
60 min leach	100.0 ± 0.0	94.4 ± 0.1	99.7 ± 0.0

3.2. Bunbury, Mozambique, and Queensland Monazite Sands

The experiments were repeated on three other monazite sands in order to validate the process for monazite sand in general. The two samples from Bunbury and Mozambique comprised mainly the mineral monazite. The sample from Queensland was a mix of monazite sand and zircon. The compositional data for each of the monazite-containing sands and results for the deportment of the rare earths between the leach solution and the residue for each of the ores are presented below.

3.2.1. Feedstock Analysis

For the sake of brevity, Table 5 shows only the average assay of each rare earth element in the Bunbury, Mozambique, and Queensland monazite sands, along with the absolute error calculated from the three repeats.

Table 5. Elemental composition: Assay of various monazites, 3 g samples.

	Bunbury (wt.%)	**Absolute Error (wt. %)**	**Mozambique (wt.%)**	**Absolute Error (wt. %)**	**Queensland (wt.%)**	**Absolute Error (wt. %)**
La	9.30	0.45	9.25	0.153	2.77	0.228
Ce	19.1	0.912	19.2	0.369	5.67	0.301
Pr	2.15	0.104	2.16	0.019	0.658	0.038
Nd	5.09	0.237	5.04	0.139	1.65	0.135
Sm	1.66	0.080	1.78	0.021	0.535	0.042
Eu	0.017	0.004	0.025	0.001	0.007	0.000
Gd	0.766	0.057	0.883	0.012	0.255	0.025
Tb	0.009	0.002	0.014	0.000	0.004	0.000
Dy	0.324	0.028	0.434	0.023	0.130	0.012
Ho	0.023	0.005	0.037	0.000	0.015	0.001
Er	0.104	0.008	0.125	0.008	0.058	0.003
Tm	0.004	0.001	0.005	0.000	0.005	0.000
Yb	0.021	0.018	0.026	0.001	0.052	0.003
Lu	0.003	0.001	0.003	0.000	0.007	0.000
Y	0.458	0.015	0.686	0.012	0.343	0.003

3.2.2. Dissolution of Rare Earths

The resulting recoveries of the light and heavy rare earths for the Bunbury, Mozambique, and Queensland zircon mix are provided in Tables 6–8, respectively.

Table 6. Overall light and heavy rare earth dissolution (%): Bunbury monazite.

	Recovery of Light Rare Earths (%)	Recovery of Heavy Rare Earths + Yttrium (%)	Recovery of Total Rare Earths (%)
15 min leach	99.8 ± 0.0	82.9 ± 0.1	99.1 ± 0.0
30 min leach	99.8 ± 0.1	86.8 ± 1.4	99.2 ± 0.1
60 min leach	100.0 ± 0.0	89.4 ± 0.5	99.5 ± 0.0

Table 7. Overall light and heavy rare earth dissolution (%): Mozambique monazite.

	Recovery of Light Rare Earths (%)	Recovery of Heavy Rare Earths + Yttrium (%)	Recovery of Total Rare Earths (%)
15 min leach	99.0 ± 0.1	97.0 ± 0.1	98.9 ± 0.1
30 min leach	99.9 ± 0.0	98.3 ± 0.0	99.9 ± 0.0

Table 8. Overall light and heavy rare earth dissolution (%): Queensland zircon mix.

	Recovery of Light Rare Earths (%)	Recovery of Heavy Rare Earths + Yttrium (%)	Recovery of Total Rare Earths (%)
15 min leach	99.8 ± 0.0	73.7 ± 6.1	98.0 ± 0.5
30 min leach	99.8 ± 0.1	71.6 ± 2.1	97.7 ± 0.1

3.3. Further Discussion

In terms of monazite dissolution, the results for the other three monazite sands are very similar to those obtained for the Perth monazite, with almost all monazite dissolved within 30 min. The mineral xenotime appears to dissolve significantly more slowly. This was particularly evident in the results for the Queensland zircon mix, where heavy rare earth dissolution was less than 80%. Further investigation is required to determine the precise cause of the slower dissolution. A possible reason is that xenotime has a different crystal structure to monazite. It is also possible that the heavy rare earths in the xenotime have a lower solubility in the leaching solution. A study on a relatively pure concentrate of xenotime is recommended to test a variety of leaching conditions and durations.

It is difficult to discuss the possible nature of the rare earth species in the solution without undue speculation. The most that can be conjectured is that the extent of dissolution of the rare earths in this solution depends on the presence of pyrophosphate and some form of complexation. This is consistent with the fact that the solution remains stable for a period after dilution with water, and then deteriorates with the precipitation of rare earths. This deterioration may be a result of the hydrolysis of the pyrophosphate species back into orthophosphate in the presence of water, in a reversal of the condensation reaction. Since, as before, the acidity of the orthophosphoric and pyrophosphoric acids is likely to be too weak for the dissolution of free rare earth ions, complexation is likely.

The prospect of a strong rare earth complex in moderately acidic conditions is perhaps distinct from other rare earth complexes. Strong rare earth complexes are often derived from weak organic acids, such as ethylenediaminetetraacetic acid (EDTA), which precipitate in moderately acidic conditions.

4. Conclusions

The overall conclusion is that the condensed phosphoric acid provides a very rapid and effective means of dissolving rare earths from monazite sands. The breakdown of monazite to deliver the full dissolution of light rare earths is achieved within 30 min. The breakdown of xenotime is also evident but the full dissolution of the heavy rare earths appears to take longer than 60 min.

5. Patents

The work reported in this study is subject to patent application PCT/AU2020/051252 entitled "Dissolution Process". The applicant is Watts & Fisher Pty Ltd. The publication number is WO2021/097527 and the publication date is 27 May 2021.

Author Contributions: Conceptualization, H.W.; methodology, H.W. and T.F.; validation, H.W. and T.F.; formal analysis, H.W. and T.F.; investigation, H.W.; writing—original draft preparation, H.W. and T.F.; writing—review and editing, H.W. and T.F. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data in this study are available on request from the corresponding author.

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Conflicts of Interest: The authors declare that they are a private research and development firm seeking to commercialize this technology. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; or in the writing of the manuscript.

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