separations

MDPI

Article

Recovery of Valuable Metals from Polymetallic Refractory Concentrate by a Sulfuric Acid Curing and Leaching Method

Wei Jiang [1,2], Jilai Xue [1], Kaixi Jiang [2], Xunxiong Jiang [2], Shengdong Wang [2], Jinping Hu [3], Derek O. Northwood [4], Kristian E. Waters [5] and Hao Ma [2,*]

[1] School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, China; jx@ustb.edu.cn (J.X.)
[2] BGRIMM Technology Group, Beijing 100070, China
[3] 224 Group Co., Ltd., SINO Shaanxi Nuclear Industry Group, Xi'an 710024, China
[4] Department of Mechanical, Automotive and Materials Engineering, University of Windsor, 401 Sunset Avenue, Windsor, ON N9B 3P4, Canada; dnorthwo@uwindsor.ca
[5] Department of Mining and Materials Engineering, McGill University, 3610 University Rd, Montreal, QC H3A 0C5, Canada
* Correspondence: haoma@bgrimm.com

Abstract: Sulfuric acid curing and leaching is a promising technology for treating refractory ores. In this work, a refractory concentrate containing 3191 ppm uranium (U), 2135 ppm niobium (Nb), and 0.7% rare earth minerals (REMs) went through two stages: curing by high-concentration H_2SO_4 and leaching by low-concentration H_2SO_4. We investigated the behavior of those valuable metals during the two stages. For both curing and leaching, the operating parameters include the acid-to-solid ratio, time, temperature, and H_2SO_4 concentration. The recovery for U, Nb, and REMs was as high as 95%, 86%, and 73.5% using a curing acid-to-solid ratio of 1:1, curing temperature of 200 °C, curing time of 1 h, H_2SO_4 concentration of 98%, leaching liquid-to-solid ratio of 4:1, leaching time of 2 h, leaching temperature of 60 °C, and leaching H_2SO_4 concentration of 5 g/L. A "sulfuric acid curing–leaching-U extraction by N235–Nb recovery by resin adsorption–REMs' recovery by resin adsorption" method was implemented, where the overall U, Nb, and REMs' recovery reached 93.1%, 84.5%, and 69.6%, respectively.

Keywords: curing; leaching; polymetallic ore; recovery; rare metals

check for updates

Citation: Jiang, W.; Xue, J.; Jiang, K.; Jiang, X.; Wang, S.; Hu, J.; Northwood, D.O.; Waters, K.E.; Ma, H. Recovery of Valuable Metals from Polymetallic Refractory Concentrate by a Sulfuric Acid Curing and Leaching Method. *Separations* **2024**, *11*, 7. https://doi.org/10.3390/separations11010007

Academic Editor: Mohamed Khayet

Received: 20 October 2023
Revised: 13 December 2023
Accepted: 19 December 2023
Published: 23 December 2023

Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/).

1. Introduction

Research on polymetallic ores mainly adopts a step-by-step recovery method, which has a relatively long process flow and high production costs [1]. Thus, further research is required to develop and utilize low-grade and complex refractory metallurgical resources [2]. A polymetallic refractory concentrate from a plant in Northwestern China contains uranium, niobium, and rare earth minerals, and an effective method needs to be developed to recover and separate these valuable metals.

Hydrometallurgical methods are primarily used in processing uranium [3]. Two steps are usually involved: leaching and recovering uranium from the leaching solution [4]. Uranium ore leaching methods can be divided into acid, alkali, salt, and bacterial. Methods for separating uranium from the leachate mainly include ion exchange and extraction [5–8]. Acid leaching is mainly used to extract the hexavalent uranium to form a uranyl sulfate (UO_2SO_4) solution. Oxidants, such as sodium chlorate ($NaClO_3$), are usually added to improve the uranium leaching rate [9]. In the case of pyrite, a pressurized oxidation leaching process may be implemented, where iron is oxidized to high-valent iron, thus leaching uranium in the form of UO_2 [10]. For alkaline leaching, carbonate is mainly used as a leaching agent to react with uranium-bearing oxidized ore so that uranium and carbonate can form $UO_2(CO_3)_3^{4-}$ in solution [11,12]. Salt leaching uses a high-valence iron salt as a

leaching agent (such as iron (III) sulfate) to leach uranium ore under acidic conditions, and the ferric iron will oxidize low-valence uranium into high-valence uranium, thus forming soluble uranium salts [13]. Bacterial leaching is utilizing bacteria to undergo biochemical reactions with the sulfides in the ores, thereby converting metals into soluble salts and entering the solution [14].

Niobium has a high melting and boiling point, paramagnetic properties, low-temperature superconductivity, and chemical stability [15,16]. Generally, niobium recovery must be carried out in steps: niobium concentrate decomposition during leaching and niobium separation from leaching solution [17]. Niobium concentrate recovery generally uses alkali, acid, chlorination, and fluorine leaching methods [18–21]. Due to its high acid resistance, niobium concentrates are difficult to decompose with a regular inorganic acid, and hydrofluoric acid, or a mixture of hydrofluoric acid and sulfuric acid, is usually used [22]. The alkaline leaching method mainly uses NaOH and KOH as reagents to react with niobium, converting niobium into sodium and potassium niobate [23]. Hydrofluoric acid is the most conventional metallurgical method for niobium concentrate treatment [24]. This method generally uses 60–70% hydrofluoric acid to react with niobium concentrate at 90–100 °C under atmospheric pressure, allowing niobium to enter the solution in the form of fluooxycolumbic acid complexes (H_2NbOF_5, H_2NbF_7, and $HNbF_6$) [25,26]. It should be noted that concentrated sulfuric acid can also be used to mix with the niobium concentrate and then calcine under high temperatures to form a soluble sulfate. The mixture can then be leached into the solution using water or acid additions [27]. The chloride process mainly uses the difference in chloride vapor pressure to separate low boiling point materials, such as niobium and titanium, from the rest of the materials [28].

To effectively extract the uranium, niobium, and rare earth minerals contained in this polymetallic refractory concentrate, preliminary tests, including enhanced acid leaching, pressure leaching, and alkali roasting leaching, were carried out [29]. It was found that under enhanced acid leaching, although uranium's leaching rate is satisfactory, niobium's leaching rate is less than 10% [30], while under pressure leaching, the process investment and operating costs are high [31]. Although uranium's leaching rate is reasonable, niobium's leaching rate is also less than 20%, and thus not conducive to the complete recovery of valuable resources [32]. The alkaline roasting leaching process does not leach uranium, and the leaching rate of niobium is only around 30% [33].

As an effective method, sulfuric acid curing and leaching have attracted researchers' attention for the past few decades [34,35]. It mainly involves using concentrated sulfuric acid to react with ore: uranium forms uranyl sulfate, calcium silicate decomposes into gypsum and SiO_2, and niobium oxide forms $Nb_2O_4SO_4$ [36]. At the same time, pyrite can be decomposed, exposing uranium and other valuable minerals wrapped in pyrite and improving the uranium leaching rate [37]. Then, a much-diluted acid, or water leaching, can be implemented. Since concentrated acid curing can improve the leaching rate, and concentrated acid has strong oxidation performance, it can directly oxidize Fe^{2+} to Fe^{3+}, thereby enabling rapid leaching of UO_2 [38]. The main chemical reactions that can occur during the curing and leaching processes for each mineral are as follows.

For U, Nb, and REMs, the main chemical reactions include [39–41]:

$$(Ca,U)_2(Ti,Nb)_2O_6(OH, F) + H_2SO_4 \rightarrow CaSO_4 + UOSO_4 + Nb_2O_4SO_4 + CaF_2 + H_2O \quad (1)$$

$$UO_2 + H_2SO_4 = UOSO_4 + H_2O \quad (2)$$

$$UO_3 + H_2SO_4 = UO_2SO_4 + H_2O \quad (3)$$

$$PbNb_2O_6 + 2H_2SO_4 = PbSO_4 + Nb_2O_4SO_4 + 2H_2O \quad (4)$$

$$2YNbO_4 + 4H_2SO_4 = Y_2(SO_4)_3 + Nb_2O_4SO_4 + 4H_2O \quad (5)$$

$$Nb_2O_5 + H_2SO_4 = Nb_2O_4SO_4 + H_2O \quad (6)$$

$$Nb_2O_5 + 2H_2SO_4 = Nb_2O_3(SO_4)_2 + 2H_2O \quad (7)$$

$$2REFCO_3 + 3H_2SO_4 = RE_2(SO_4)_3 + 2HF + 2CO_2 \quad (8)$$

$$2Ce_2Fe_3Si_2O_7SiO_4O(OH) + 13H_2SO_4 \rightarrow 2Ce_2(SO_4)_3 + Fe_2(SO_4)_3 + 4FeSO_4 + 6H_2SiO_3 + 8H_2O \quad (9)$$

For other minerals, including Ti, Fe, Al, Mg, and Pb, the reactions include:

$$TiO_2 + H_2SO_4 = TiOSO_4 + H_2O \quad (10)$$

$$TiO_2 + 2H_2SO_4 = Ti(SO_4)_2 + H_2O \quad (11)$$

$$TiO_2 + CaO + 3H_2SO_4 = CaTi(SO_4)_3 + 3H_2O \quad (12)$$

$$Ti_2O_3 + 3H_2SO_4 = Ti_2(SO_4)_3 + 3H_2O \quad (13)$$

$$CaTi(SO_4)_3 + H_2O = TiOSO_4 + CaSO_4 + H_2SO_4 \quad (14)$$

$$FeO + H_2SO_4 = FeSO_4 + H_2O \quad (15)$$

$$Fe_3O_4 + 4H_2SO_4 = FeSO_4 + Fe_2(SO_4)_3 + 4H_2O \quad (16)$$

$$Fe_2O_3 + 3H_2SO_4 = Fe_2(SO_4)_3 + 3H_2O \quad (17)$$

$$Al_2O_3 + 3H_2SO_4 = Al_2(SO_4)_3 + 3H_2O \quad (18)$$

$$MgO + H_2SO_4 = MgSO_4 + H_2O \quad (19)$$

$$CaO + H_2SO_4 = CaSO_4 + H_2O \quad (20)$$

$$PbO + H_2SO_4 = PbSO_4 + H_2O \quad (21)$$

In the presence of Fe^{3+}, U^{4+} and Ti^{3+} will be oxidized to uranyl ions and Ti^{4+}:

$$U^{4+} + 2H_2O + 2Fe^{3+} = {UO_2}^{2+} + 2Fe^{2+} + 4H^+ \quad (22)$$

$$Ti^{3+} + Fe^{3+} = Ti^{4+} + Fe^{2+} \quad (23)$$

Under low-acidity conditions, titanium and niobium sulfates precipitate:

$$TiOSO_4 + 3H_2O = Ti(OH)_4\downarrow + H_2SO_4 \quad (24)$$

$$Nb_2O_4SO_4 + 6H_2O = 2Nb(OH)_5\downarrow + H_2SO_4 \quad (25)$$

This study investigated the curing and leaching behavior of U, Nb, and REM concentrates, and the valuable metals' separation was also examined. A sulfuric acid curing and leaching method in the experimental stages has been proposed for U, Nb, and REM recovery that is HF-free and thereby attractive from both an industrial production and environmental protection perspective. Additionally, it realizes a comprehensive recovery of valuable metals in a short process from low-grade complex mine resources, and direct extraction of uranium under high-acidity conditions is also proposed. Meanwhile, the obtained results will provide crucial information on the leaching of valuable metals from polymetallic refractory concentrates, as well as the applicability of the process to commercial operations.

2. Materials and Methods

2.1. Materials and Analysis

The sulfuric acid (98% H_2SO_4) used in the study was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). The phase composition of the sample was investigated by X-ray diffractometry (XRD, D/max-Ultima IV, Rigaku, Japan). Metal content was analyzed using an inductively coupled plasma-optical emission spectrometer (ICP-OES, 700-ES, Varian, Palo Alto, CA, USA). The particle size was analyzed using a laser particle size analyzer (Malvern Panalytical Mastersizer 3000, Malvern, UK). The chemical states of metal elements were analyzed by X-ray energy-dispersive spectrometry (EDS,

EDAX-GENESIS JSM-6460LV, Pleasanton, CA, USA). The morphologies of the samples were examined using optical microscopy (Axioplan 2, Zeiss, Oberkochen, Germany). The concentrate was received from a polymetallic refractory ore mining site in China.

2.2. Curing and Leaching Process

The curing test was carried out in a 500 mL beaker. Here, 100 g of concentrate was added to the beaker, and concentrated sulfuric acid was added afterward. That is when the curing timing started. A thermometer was directly placed in the sample for temperature measurement. It can be seen that a large number of bubbles were immediately produced, releasing a large amount of heat, and the material temperature rapidly increased, resulting in the material being in a black solid state (Figure 1a). Once the curing process ended, the material had poor adhesion to the beaker, making it easy to process further (Figure 1b). If heating is needed after mixing the materials, they will be placed in a muffle furnace (NBD-M1200-20TI, Henan Nobody Materials Science and Technology Co., Ltd., Zhengzhou, China). We started heating them to the set temperature at a specific rate, and then the timing was started.



(**a**) (**b**) (**c**)

Figure 1. Photos showing the curing and leaching process: (**a**) when curing starts, (**b**) when curing ends, and (**c**) when leaching ends.

Once the curing materials had cooled to room temperature, the beaker was put into a water bath at a specific temperature. Diluted sulfuric acid was slowly added to the beaker for the materials to be leached. That is when the leaching time started. For all tests, a RW 20 digital mixer was used with a stirring paddle diameter of 35 mm and a rotational speed of 300 rpm. The slurry is usually grayish-white (Figure 1c). Once leaching ended, it was filtered, and the leaching solution had a yellow color, while the leaching residue was black. The solution and filtrate obtained after filtration were sent to analyze the concentration of U, Nb, and REMs.

Based on the technical evaluation of experimental parameters for the curing and leaching methods described above, and from the analysis of the advantages of hydrometallurgical methods, to efficiently separate and purify the valuable metals in the solution, a series of stages were required. They were as follows:

1. The leaching solution was treated with an extractant (type: N235) and synergic reagent 2-octanol diluted in kerosene. After two-stage uranium extraction, uranium was separated from other soluble sulfates, such as niobium, rare earth minerals, and iron. The loaded organic phase can form $Na_4UO_2(CO_3)_3$ through sodium carbonate stripping to enter the aqueous phase. The aqueous phase was then precipitated by caustic soda as a yellow cake (uranium content: 67.0%). At the same time, the carbonate ions in the complex were regenerated to form sodium carbonate, thereby enabling the recycling of sodium carbonate.
2. The residual uranium extraction solution was exchanged through resin (type: LSC-930B), allowing niobium to enter the resin phase selectively. The resin loaded with niobium was washed with diluted sulfuric acid to remove iron and desorbed

from the H_2SO_4 solution containing H_2O_2. The desorbed H_2NbO_4 solution was then hydrolyzed and precipitated at a high temperature (90–98 °C) to precipitate niobium hydrated oxide ($Nb_2O_5 \cdot nH_2O$). Then, the oxide was sent for drying with a Nb_2O_5 content of 90.2%, and titanium could be further extracted from the hydrolyzed solution.

3. After niobium adsorption, the solution went through resin (type: LSC-957) for REMs treatment, and the resin loaded with REMs went through a nitric acid solution for REMs desorption, oxalic acid precipitation, pH adjustment by NaOH, and roasting. The final REM oxide had a content of 82.5%.

U, Nb_2O_5, REMs, and other ions contained in the leach residue and leaching liquor were analyzed, and the metal leaching percentage was calculated using Equation (26) [42]:

$$x = \frac{W_S}{W_S + W_R} * 100\% \quad (26)$$

where x is the leaching percentage in %, W_S is the mass of the metal element in the solution in grams (g), and W_R is the mass of the metal element in the residue in grams (g).

3. Results and Discussion

3.1. Concentrate Phase and Composition

The ore received contained 0.04% betafite, 0.22% allanite, and a small amount of other U-, Nb-, and REM-bearing minerals. The concentrate was obtained through gravity separation and flotation. The main elemental compositions of the concentrate are provided in Table 1. The XRD pattern of the concentrate is shown in Figure 2. The main phases contained in the sample included quartz (SiO_2), barite ($BaSO_4$), K-Feldspar ($KAlSi_3O_8$), betafite ($(Ca,U)_2(Ti,Nb)_2O_6(OH, F)$), and orthite ($(Ce,Ca)_2(Fe,Al)_3[Si_2O_7][SiO_4]O(OH)$). The particle size of the concentrate was analyzed, and the particle size of approximately −75 μm accounted for 96.0% of the concentrate.

Table 1. Chemical composition of the concentrate.

Element	CaO	MgO	TFe	TiO_2	Al_2O_3	REMs	SiO_2	U	Nb
Content (wt.%)	10.3	1.2	8.0	1.8	1.9	0.7	42.7	3191 ppm	2135 ppm

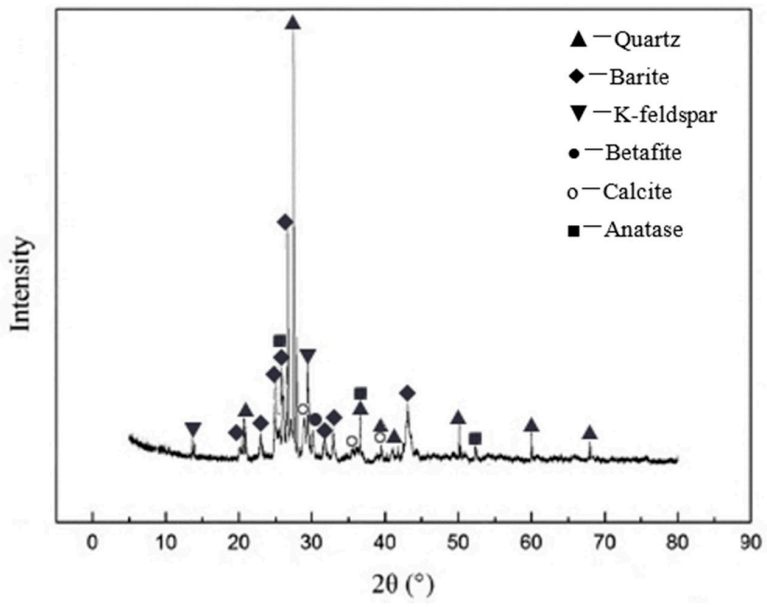


Figure 2. The XRD pattern of the received concentrate sample.

The phases of the main U- and Nb-bearing minerals (betafite, uraninite, fergusonite, feldspar, thorite, and ilmenorutile) and REM-bearing minerals (allanite, bastnaesite, gadoli-

nite, and monazite) were also identified with optical microscopy (Figure 3a–c), SEM (Figure 3d), and EDS (Figure 3e–h). It should be noted that EDS can only provide qualitative and semi-quantitative results.

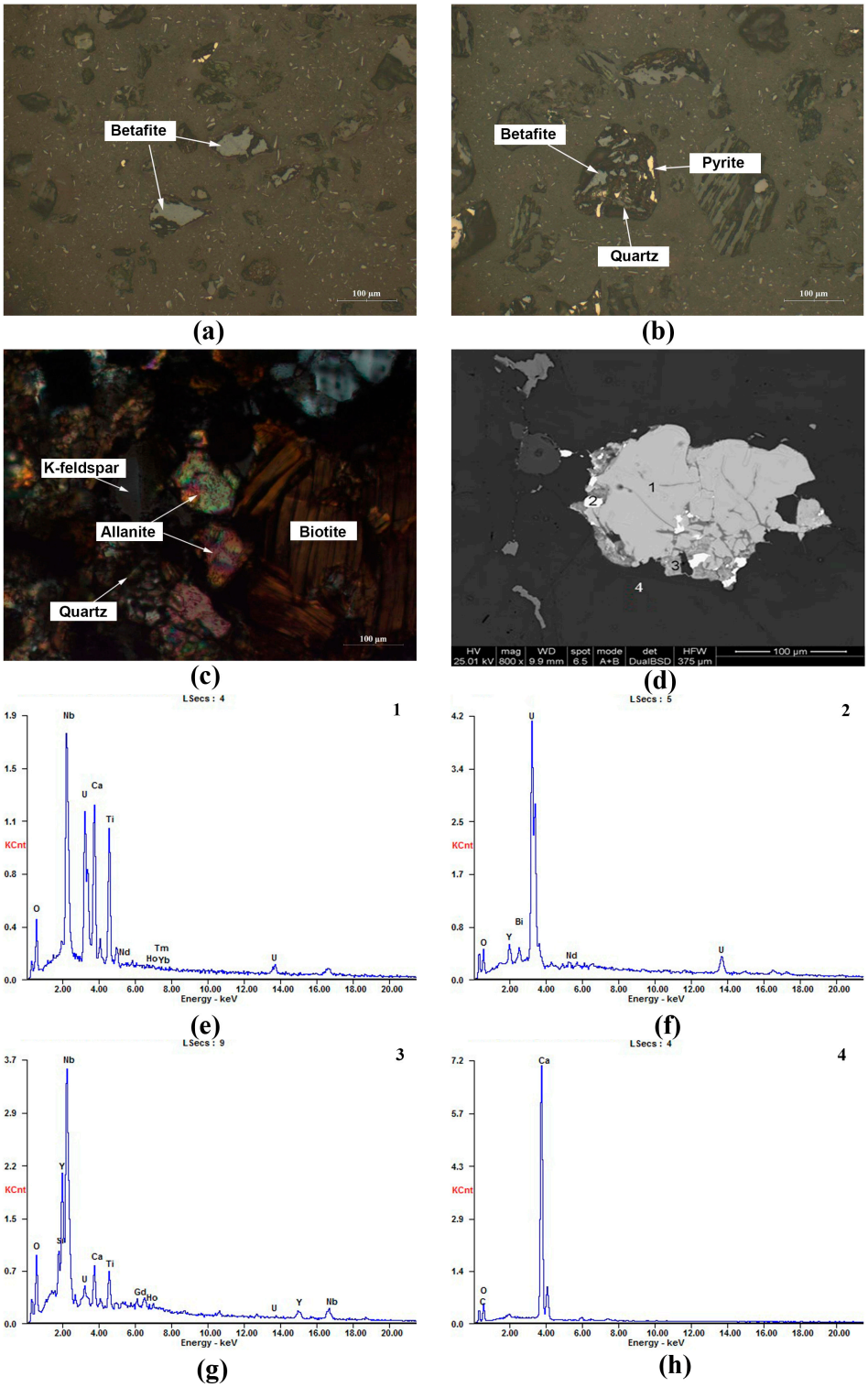


Figure 3. Phase characterization of the main minerals: (**a**) betafite, (**b**) betafite coexisting with quartz and pyrite, (**c**) orthite coexisting with other minerals, and (**d**) SEM of betafite, where the EDS graphs show a typical composition of betafite in 3(d): (**e**) uraninite, (**f**) fergusonite, (**g**) changbaiite, and (**h**) calcite, in the concentrate.

Figure 3 shows that betafite is the most critical uranium- and niobium-bearing mineral, which are usually automorphic and semi-automorphic octahedron crystalline grains (Figure 3a). Uranium minerals are often closely embedded to form uranium mineral aggregates and exist as a monomer. A small amount of uranium minerals is associated with other minerals (Figure 3b), and their particle size is mainly 30–80 μm. Rare earth minerals are mainly orthite (Figure 3c) and a small amount of bastnasite, gadolinite, and monazite. Orthite is mainly distributed in quartz and calcite in subhedral and allomorphic granular form. A small amount of orthite is embedded with K-feldspar, biotite, barite, and celestite. Sometimes, it can be seen that rare earth minerals, such as fine monazite, gadolinite, and thorite, are wrapped in orthite, mainly in the form of monomer and rich intergrowth. The particle size of rare earth minerals is between 30 and 60 μm. Betafite is the primary carrier of uranium and niobium. An EDS scan was performed on a selected betafite mineral (see Figure 3d), and it showed that the interior of betafite often develops cracks and holes. Its edge or interior often contains uraninite (Figure 3e), fergusonite (Figure 3f), and changbaiite (Figure 3g), while they are embedded in calcite (Figure 3h).

Uranium mainly exists in betafite (73.7%), followed by uraninite (12.8%). In addition, 13.5% of uranium is distributed in fergusonite (4.7%), feldspar (6.2%), and thorite (2.5%). Niobium also mainly occurs in betafite (71.9%), followed by fergusonite (12.7%), feldspar (12.0%), and ilmenorutile (3.4%).

3.2. Curing Process

The effects of the operating parameters, including the acid-to-solid ratio, curing temperature, curing time, and H_2SO_4 concentration, on the leaching efficiency were investigated during the curing process. It should be noted that once the test ended and cooled down, all samples were leached in a 10 g/L H_2SO_4 solution at a liquid-to-solid ratio of 6:1 under 60 °C for 3 h. The values of each parameter during the curing process are listed as follows: for the acid-to-solid ratio, it ranged from 0.7:1 to 1.5:1; for the curing temperature, it ranged from 160 °C to 250 °C; for the curing time, it ranged from 10 min to 4 h; for the H_2SO_4 concentration, it ranged from 40% to 98%. It should be noted that the REMs' recovery during tests is not shown in the following figures due to data privacy.

3.2.1. Effect of Acid-to-Solid Ratio

The acid-to-solid ratio is an essential parameter for the extraction of valuable metals from uranium polymetallic mines. It is a critical indicator for the commercial mining of uranium resources. In the solid–liquid reaction, the higher the acid-to-solid ratio, the higher the probability of the valuable metal coming into contact with the acid, thus improving the mineral leaching rate [43]. However, a very high acid-to-solid ratio (higher than 1.5) will increase the free acid concentration, and the subsequent processing process is usually complex, resulting in higher production costs. A small acid-to-solid ratio (less than 0.7) may reduce the leaching rate and hinder the recovery of valuable metals. At the same time, niobium sulfate is prone to hydrolysis and precipitation in low-acidity solutions.

The test conditions were set as: curing temperature 220 °C, curing time 2 h, and H_2SO_4 concentration 98%. The amount of concentrate tested was 100 g. The acid-to-solid ratios tested were 0.7:1, 0.8:1, 0.9:1, 1:1, 1.1:1, 1.2:1, and 1.5:1 (corresponding to 70 g, 80 g, 90 g, 100 g, 110 g, 120 g, and 150 g of 98% H_2SO_4 for each test). The effects of the acid-to-solid ratio on U and Nb leaching are shown in Figure 4a.

The test results showed that a higher acid-to-solid ratio would lead to a high uranium and niobium leaching rate. When the ratio reached 1:1, the uranium leaching percentage reached 94.9%. As the acid-to-solid ratio continued to increase, the uranium leaching percentage increased slowly and eventually reached 95.7%. For niobium, when the ratio was 0.8:1, the leaching percentage was 70.2%. When the ratio increased to 1:1, the leaching percentage of niobium reached 83.4%, and the leaching percentage of niobium stabilized at around 86%. Thus, an acid-to-solid ratio of 1:1 was selected, and the rare earth minerals' leaching percentage was 76.1% at this acid-to-solid ratio.

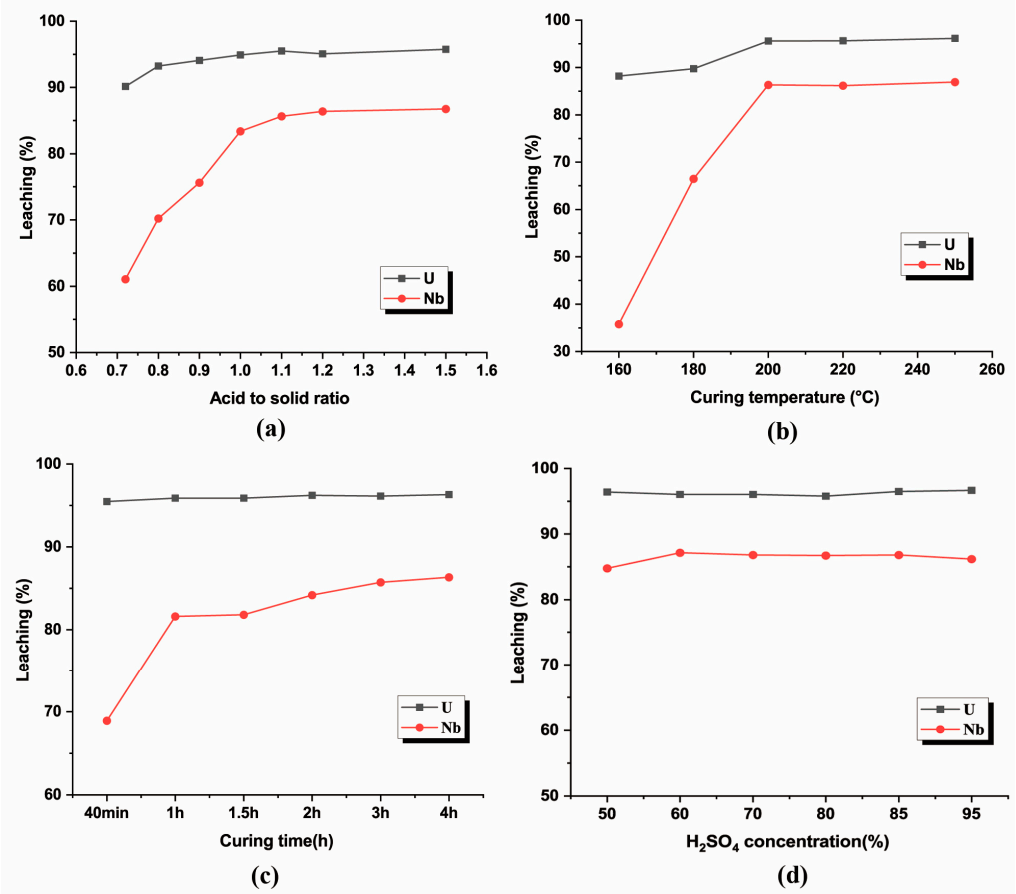


Figure 4. Effects of the: (**a**) acid-to-solid ratio, (**b**) curing temperature, (**c**) curing time, and (**d**) H_2SO_4 concentration on uranium and niobium recovery during the curing process.

3.2.2. Effect of Curing Temperature

In general, a high temperature is beneficial for increasing chemical activity and enhancing the driving force of the reaction, which helps promote the leaching of valuable minerals. However, as the temperature rises, it could lead to higher costs and operating risks to the equipment. Thus, the curing temperature should be as low as possible, ensuring an efficient recovery of valuable minerals. The temperature range tested was 160–250 °C, while the concentrate tested was 100 g. The remaining test conditions were set as: acid-to-solid ratio 1:1, curing time 2 h, and H_2SO_4 concentration 98%. The effect of temperature on the extraction of U and Nb was examined. The test results are shown in Figure 4b.

The test results showed that with an increased curing temperature, uranium and niobium leaching rates gradually increased. At 160 °C, the recovery rates of uranium and niobium were 88.2% and 35.8%, respectively. When the curing temperature increased to 200 °C, the recovery rates of uranium and niobium reached 95.6% and 86.3%, respectively. As the curing temperature continued to increase, there was an increase in the leaching percentage of both mineral plateaus. Thus, a curing temperature of 200 °C was selected, and the rare earth minerals' leaching percentage was 75.8% under this temperature.

3.2.3. Effect of Curing Time

Based on preliminary tests, leaching times between 40 min and 4 h were chosen to determine the effect of the curing time on the extraction behavior of U and Nb. The test conditions were as follows: acid-to-solid ratio 1:1, curing temperature 200 °C, and H_2SO_4 concentration 98%. The weight of the concentrate was 100 g. The results are shown in Figure 4c.

It can be seen that the behavior of U and Nb was very different. U recovery was relatively unaffected by the curing time. When the curing time was 40 min, the recovery of

uranium and niobium was 95.5% and 68.9%, respectively. When the curing time was extended to 1 h, the recovery of uranium and niobium reached 95.9% and 81.6%, respectively. The leaching rate of niobium was lower in the first 40 min but increased rapidly with the extension of the curing time between 40 min and 1 h. In addition, due to the high curing temperature, the curing time should be as low as possible, while ensuring a satisfying metal recovery. Thus, a curing time of 1 h was selected, and the rare earth minerals' leaching recovery rate was 72.2% at this temperature.

3.2.4. Effect of H_2SO_4 Concentration

Based on our experience, H_2SO_4 concentrations of 50%, 60%, 70%, 80%, 85%, and 98% were tested. The test conditions were set as: acid-to-solid ratio 1:1, curing time 1 h, and curing temperature 200 °C. Here, 100 g of concentrate was tested. The results are shown in Figure 4d.

It can be seen that the recovery of uranium and niobium was little affected by the sulfuric acid concentration during curing. When the concentration of H_2SO_4 was 50%, the recovery rates of uranium and niobium reached 96.4% and 84.8%. When the sulfuric acid concentration reached 60%, the acid concentration continued to increase, and the leaching percentage of niobium tended to stabilize. Under the same amount of acid, low-concentration sulfuric acid (50%) may experience a loss due to volatilization during the heating process, decreasing in the effective acid content. This could be explained by the fact that the lower the concentration of sulfuric acid (50%), the more free water needs to be evaporated during the activation process, increasing energy consumption. In the curing process, in order to minimize acid volatilization loss, resulting in deficient-concentration sulfuric acid volatilization, there is a low-temperature insulation process (the silicon carbon rod in the muffle furnace is electrically heated to maintain the insulation process) during the experiment. When the concentration reached 60%, the volatilization of sulfuric acid was low; that is, the acid-to-ore ratio was the same, and the fluctuation of uranium niobium leaching rate was negligible. However, the determining factor may be the amount of acid used to cure.

The H_2SO_4 concentration was the last parameter to be tested, and when the concentration was $\geq$60%, the impact on uranium and other minerals' recovery was relatively small. However, concentrated sulfuric acid does not need to be diluted—it can generate heat on its own, which saves time and heating costs. Further, the lower the concentration, the greater the amount of solution used. In addition, there was also moisture in the concentrate after flotation (20–40%). When it reached the targeted temperature, the energy consumption was higher than concentrated sulfuric acid. An H_2SO_4 concentration of 98% was selected for more straightforward operation, and the rare earth minerals' recovery rate was 76.1% at this concentration.

3.3. Leaching Process

It should be noted from Section 3.2 that the optimal parameters for the curing process were as follows: acid-to-solid ratio 1:1, curing time 1 h, curing temperature 200 °C, and H_2SO_4 concentration of 98%. The effects of the operating parameters, including the liquid-to-solid ratio, leaching time, temperature, and solution concentration, on the leaching efficiency were investigated for the leaching process. The values of each parameter during the leaching process investigation were as follows: liquid-to-solid ratio (L/S) ranging from 1.5:1 to 8:1, leaching time ranging from 1 h to 6 h, leaching temperature ranging from 18 °C to 90 °C, and leaching solution concentration ranging from 0 to 50 g/L. It should be noted that the REMs' recovery during tests is not shown in the following figures due to data privacy.

3.3.1. Effect of L/S Ratio

The L/S ratio refers to the weight ratio of the leaching solution to the concentrate. The test conditions were set as: leaching time of 3 h, leaching temperature of 60 °C, and

leaching solution concentration of 10 g/L. Here, 100 g of concentrate was tested. L/S ratios tested were: 1.5:1, 2:1, 2.5:1, 3:1, 4:1, 5:1, 6:1, and 8:1. The effects of the L/S ratio on U and Nb leaching are shown in Figure 5a.

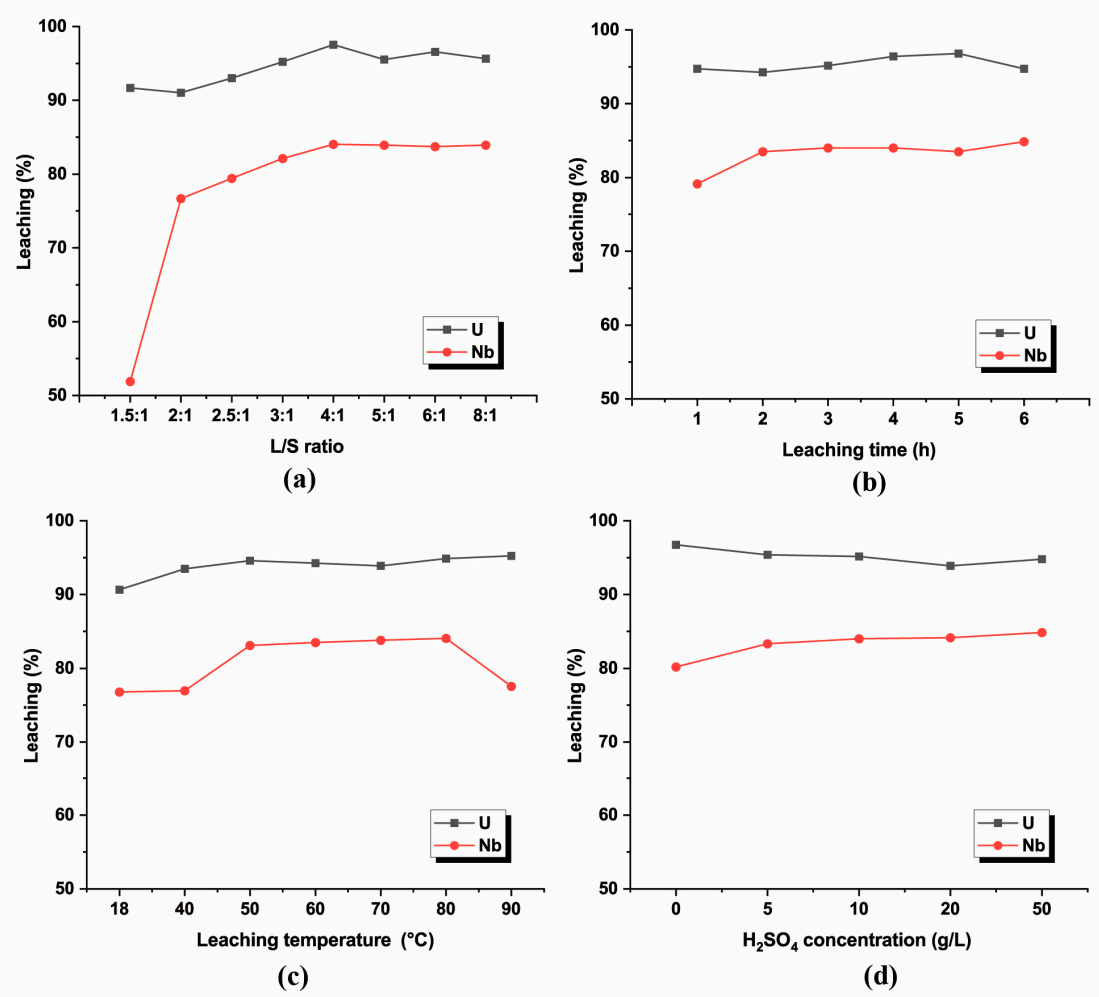


Figure 5. Effects of: (**a**) L/S ratio, (**b**) leaching time, (**c**) leaching temperature, and (**d**) H_2SO_4 concentration on uranium and niobium recovery during the leaching process.

As the L/S ratio increased, the recovery of uranium and niobium gradually increased and tended to flatten out beyond 4:1. When the L/S ratio was 4:1, the leaching percentage of uranium was 97.6%. The leaching percentage of niobium was 84.0%.

Thus, an L/S ratio of 4:1 was selected. The rare earth minerals' leaching percentage was 71.8% at this ratio.

3.3.2. Effect of Leaching Time

The test conditions were set as: L/S ratio 4:1, leaching temperature 60 °C, and leaching solution concentration 10 g/L. Here, 100 g of concentrate was tested. Leaching times tested were: 1 h, 2 h, 3 h, 4 h, 5 h, and 6 h. The effects of the leaching time on U and Nb leaching are shown in Figure 5b.

The leaching percentage of uranium and niobium gradually increased with the increasing leaching time for the first 4 h. This could be explained by the fact that a longer leaching time facilitates the breakdown of the solid mineral particles, resulting in a more significant release of its components into the solution. When the leaching time was 1 h, the uranium and niobium leaching percentages were 94.7% and 79.1%, respectively. When the leaching time was extended to 2 h, the percentages of leached uranium and niobium reached 94.3% and 83.5%, respectively. Extending the leaching time tended to stabilize

the percentage leached of both metals. This is probably due to the dissolution of other high-content impurities, such as iron, which might interfere with the permeation of acid on the surface of concentrated particles.

Thus, a leaching time of 2 h was selected. The rare earth minerals' leaching percentage was 75.6%.

3.3.3. Effect of Leaching Temperature

The test conditions were set as: L/S ratio 4:1, leaching time 2 h, and leaching solution concentration 10 g/L. Here, 100 g of concentrate was tested. Leaching temperatures tested were: 18 °C (room temperature), 40 °C, 50 °C, 60 °C, 70 °C, 80 °C, and 90 °C. The effects of the leaching temperature on U and Nb leaching are shown in Figure 5c.

The leaching percentage of valuable metals in the concentrate increased with the increase of the leaching temperature, and after reaching around 50 °C, the leaching percentage tended to stabilize. Increasing the temperature of the leaching process, in general, reduced the reaction time required. When the leaching temperature was room temperature, the leaching percentages of uranium and niobium were 90.7% and 76.8%, respectively. When the leaching temperature increased to 60 °C, the leaching percentages of uranium and niobium were 94.3% and 83.5%, respectively. When the temperature reached 90 °C, the leaching percentage of niobium tended to decrease, possibly due to the hydrolysis of some niobium under high-temperature conditions.

To avoid Nb hydrolysis and save energy costs, a leaching temperature of 60 °C was selected, and the rare earth minerals' leaching rate was 75.6% at this temperature.

3.3.4. Effect of Leaching Solution Concentration

The test conditions were set as: L/S ratio 4:1, leaching time 2 h, and leaching temperature 60 °C. Here, 100 g of concentrate was tested. Leaching solution (diluted H_2SO_4) concentrations tested were: 0 g/L, 5 g/L, 10 g/L, 20 g/L, and 50 g/L. The effects of the leaching solution concentration on U and Nb leaching are shown in Figure 5d.

Within the experimental conditions, the increase in the leaching solution concentration had little impact on the leaching percentages of uranium and niobium. When using direct water leaching (no H_2SO_4), the leaching percentage of uranium was 96.8%, and the leaching percentage of niobium was 80.2%. When the sulfuric acid concentration was 5 g/L, the leaching percentages of uranium and niobium were 95.4% and 83.3%, respectively.

A leaching solution concentration of 5 g/L was selected when making the maximum economic benefits a priority. The rare earth minerals' leaching percentage was 76.98% at this concentration.

3.3.5. Validation of Optimal Conditions

The optimal operating conditions were as follows: for the curing process—acid to solid ratio 1:1, curing time 1 h, curing temperature 200 °C, and H_2SO_4 concentration of 98%; for the leaching process—L/S ratio 4:1, leaching time 2 h, leaching temperature 200 °C, and leaching solution concentration 5 g/L. Three parallel tests were performed using 100 g of concentrate for every test. The test results showed that the residue rate was about 89.6% under optimal conditions. The chemical composition of the leaching residue is provided in Table 2.

Table 2. Chemical composition of the leaching residue.

Element	Fe	Al_2O_3	REMs	CaO	MgO	TiO_2	U	Nb
Content (wt.%)	3.0	1.8	0.2	12.8	1.5	1.2	185 ppm	479 ppm

The recovery percentage of U and Nb for all three tests averaged 94.8% (±1.6%) and 85.9% (±2.0%), respectively, confirming that the test has high reproducibility. The average recovery for REMs was 73.5%. The recrystallization of betafite by heat treatment appeared

to promote the formation of an enriched surface layer, which may be related to a lower extraction of Nb [36]. The leaching residue mainly comprises minerals, such as quartz, iron sulfate, and gypsum, with stable properties. However, it also contains certain low radioactive substances (U, Th), and suitable measures need to be taken to reduce their impact on the environment. The use of alkaline flotation tailings can neutralize the relatively small amount of acidic leaching residue.

3.4. Purification and Separation of Leaching Solution

In order to obtain an efficient method for separating the valuable minerals, as well as preparing for final U, Nb, and REM products for commercial purposes, approximately 35 L of leaching solution containing uranium, niobium, and REMs was prepared through sulfuric acid curing and leaching experiments with 10 kg of concentrate. The composition of the leaching solution is shown in Table 3. It should be noted that this was a laboratory-scale test, and the commercially sensitive results and details are not available to the public.

Table 3. Chemical composition of the leaching solution.

Element	U	Nb	REMs	TFe
Content (g/L)	0.51	0.23	0.62	7.1

Through the method described in Section 2.2, the final recovery rates of uranium, niobium, and REMs were 93.1%, 84.5%, and 69.6%, respectively. An overview of the complete process is shown in Figure 6.

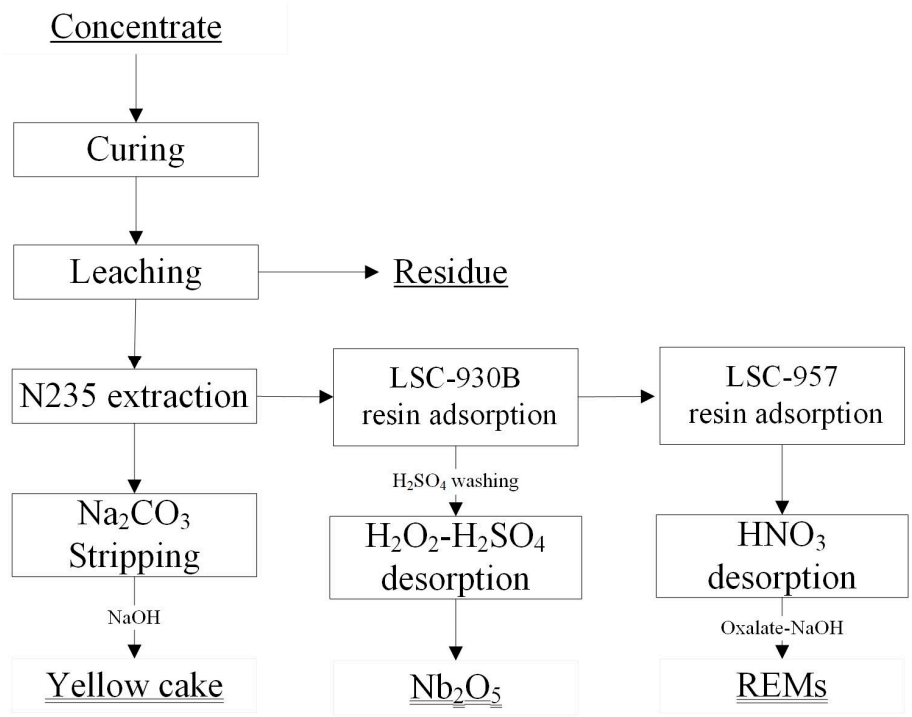


Figure 6. Schematic of the complete process for treatment of concentrates containing U, Nb, and REMs.

3.5. Discussion

For the curing process, it was found that the uranium leaching was relatively less affected by the acid-to-solid ratio compared to niobium. The main reason may be that uranium minerals have higher chemical activity during the curing process and are prone to react to form soluble salts [44]. When the acid-to-solid ratio was low, the final acid concentration in the leaching solution was low, resulting in the hydrolysis of niobium sulfate

being inhibited in the leaching residue, thereby reducing the leaching rate of niobium. Additionally, under 200 °C, the degree of structural damage to orthite and betafite minerals was low, resulting in an insufficient exposure of valuable metals and insufficient energy when reacting with sulfuric acid. When the temperature increased to 200 °C, the chemical reaction activity of the mineral increased, the reaction driving force increased, and the structure of orthite and betafite minerals was broken down. Overheating caused the reduction and dehydration of some rare earth elements, forming insoluble sulfate salts. Within 30 min, niobium minerals were in the initial activation stage, and the mineral structure was relatively less damaged. With the extension of the curing time, the structure of valuable metal minerals was rapidly destroyed, leading to the entry of sulfate into the solution during the process. When the curing time extended to 2 h, the leaching rate of niobium slowly increased, but the migration rate slowed down. In addition, calcium in minerals may form gypsum during the process, which may encapsulate the niobium. In addition, the lower the H_2SO_4 concentration, the higher the energy consumption, which is not conducive to energy conservation and emission reduction.

For the leaching process, the low efficiency at low L/S ratios was due to the high viscosity and plasticity of the slurry produced by the presence of large amounts of gypsum and bassanite. This increase in extraction, resulting from a decrease in viscosity, is due to increased contact between the leaching solution and the surface of the solids. Although a much higher L/S ratio may bring the desired viscosity and plasticity to the slurry, it may also dilute the free acid left from the curing process and cause a decrease in H+ activity for the leaching process, thus decreasing the amount of leaching [44]. As shown by other researchers, based on the shrinking core model, the surface chemical reaction is generally considered the rate-controlling step for the dissolution mechanism [35]. According to this model, the majority of the U and Nb reactions occur in the first hour since quite a number of the unreacted cores of the solid material already shrink toward the center of the solid material. The dissolution of the solid materials and release of their components occur during the curing process, which helps promote the leaching process. An increase in temperature generally increases the thermal movement of ions and the reaction rate. In the curing process, it is due to the facilitation of the migration of the leaching agent into the inner layers of the concentrate; in the leaching process, it is due to the transport of the dissolved metals from inside the particle to the leachate. In theory, the higher the initial sulfuric acid concentration during leaching, the higher the leaching percentage of valuable metals. After the curing process, there was a large amount of residual free acid (~1 mol/L). Here, free acid is the concentration of acid in the leaching solution, which was measured quantitatively using a pH meter on the H^+ concentration, and the corresponding soluble sulfate will not be precipitated through hydrolysis, even when leaching using water. Since there was an abundant amount of H^+ in the solution, the increase in the leaching solution concentration did not have a significant impact on the metal recovery.

4. Conclusions

The curing and leaching behavior of a refractory concentrate has been investigated. The recovery rate for uranium and niobium was 94.8% and 85.9% under the optimal processing conditions of acid-to-solid ratio 1:1, curing time 1 h, curing temperature 200 °C, and H_2SO_4 concentration of 98% in the curing process, and for a liquid-to-solid ratio of 4:1, leaching time of 2 h, leaching temperature of 200 °C, and leaching solution concentration of 5 g/L in the leaching process. It was found that the remaining metals were generally wrapped in delicate and dense gypsum for both uranium and niobium. To efficiently separate the U, Nb, and REMs in the leaching solution, a combination of extraction-stripping and resin absorption and desorption methods were conducted, resulting in a recovery percentage of U and Nb of 93.1% and 84.5%, respectively. The uranium content in the yellow cake reached 67.0%, the Nb_2O_5 content in the niobium oxide reached 90.2%, and the rare earth oxide content in the REMs reached 82.5%. These results contribute to the clean and efficient utilization of polymetallic refractory mineral resources.

Author Contributions: Conceptualization, J.H.; methodology, X.J., K.J. and J.X.; software, W.J.; formal analysis, S.W.; data curation, S.W. and J.H.; writing—original draft preparation, W.J. and X.J.; writing—review and editing, Northwood, D.O.N., K.E.W. and H.M.; supervision, X.J.; project administration, K.J.; funding acquisition, S.W. and J.H.; All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Data is contained within the article.

Acknowledgments: The anonymous reviewers are deeply acknowledged for improving the quality of the manuscript.

Conflicts of Interest: Author Wei Jiang, Kaixi Jiang, Xunxiong Jiang, Shengdong Wang and Hao Ma were employed by the company BGRIMM Technology Group. Author Jinping Hu was employed by the company SINO Shaanxi Nuclear Industry Group. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

References

1. Watling, H.R. Review of biohydrometallurgical metals extraction from polymetallic mineral resources. *Minerals* **2014**, *5*, 1–60. [CrossRef]
2. Aleksandrova, T.; Nikolaeva, N.; Afanasova, A.; Romashev, A.; Kuznetsov, V. Selective disintegration justification based on the mineralogical and technological features of the polymetallic ores. *Minerals* **2021**, *11*, 851. [CrossRef]
3. Chmielewski, A.G.; Wawszczak, D.; Brykała, M. Possibility of uranium and rare metal recovery in the Polish copper mining industry. *Hydrometallurgy* **2016**, *159*, 12–18. [CrossRef]
4. Wu, S.; Wang, L.; Zhao, L.; Zhang, P.; El-Shall, H.; Moudgil, B.; Huang, X.; Zhang, L. Recovery of rare earth elements from phosphate rock by hydrometallurgical processes—A critical review. *Chem. Eng. J.* **2018**, *335*, 774–800. [CrossRef]
5. Mudd, G.M. Critical review of acid in situ leach uranium mining: 1. USA and Australia. *Environ. Earth Sci.* **2001**, *41*, 390–403. [CrossRef]
6. Ghorbani, Y.; Montenegro, M.R. Leaching behaviour and the solution consumption of uranium–vanadium ore in alkali carbonate–bicarbonate column leaching. *Hydrometallurgy* **2016**, *161*, 127–137. [CrossRef]
7. Zhu, Z.; Pranolo, Y.; Cheng, C.Y. Separation of uranium and thorium from rare earths for rare earth production—A review. *Miner. Eng.* **2015**, *77*, 185–196. [CrossRef]
8. Muñoz, J.; González, F.; Blázquez, M.; Ballester, A. A study of the bioleaching of a Spanish uranium ore. Part I: A review of the bacterial leaching in the treatment of uranium ores. *Hydrometallurgy* **1995**, *38*, 39–57. [CrossRef]
9. Miki, H.; Nicol, M. The kinetics of the oxidation of iron(II) by chlorate in the leaching of uranium ores. *Hydrometallurgy* **2009**, *100*, 47–49. [CrossRef]
10. Edwards, C.R.; Oliver, A.J. Uranium processing: A review of current methods and technology. *JOM* **2000**, *52*, 12–20. [CrossRef]
11. Esmaeel, S.M.A. Sorption of uranium after carbonate leaching by low cost activated carbon–aluminum ferrisilicate composite. *Int. J. Environ. Anal. Chem.* **2022**, *102*, 2641–2660. [CrossRef]
12. Santos, E.A.; Ladeira, A.C.Q. Recovery of uranium from mine waste by leaching with carbonate-based reagents. *Environ. Sci. Technol.* **2011**, *45*, 3591–3597. [CrossRef] [PubMed]
13. Sapsford, D.; Bowell, R.; Geroni, J.; Penman, K.; Dey, M. Factors influencing the release rate of uranium, thorium, yttrium and rare earth elements from a low grade ore. *Miner. Eng.* **2012**, *39*, 165–172. [CrossRef]
14. Deng, Y.; Hou, W.; Wei, X.; Wang, Q.; Wang, H.; Hu, E.; Lei, Z.; Hu, F.; Zhang, Y. Mechanism analysis on synergistic leaching of uranium from pyrite oxidized by Acidithiobacillus ferrivorans. *J. Radioanal. Nucl. Chem.* **2021**, *330*, 951–961. [CrossRef]
15. Nico, C.; Monteiro, T.; Graça, M. Niobium oxides and niobates physical properties: Review and prospects. *Prog. Mater. Sci.* **2016**, *80*, 1–37. [CrossRef]
16. Mattheiss, L.F. Electronic structure of niobium and tantalum. *Phys. Rev. B* **1970**, *1*, 373–380. [CrossRef]
17. Shikika, A.; Sethurajan, M.; Muvundja, F.; Mugumaoderha, M.; Gaydardzhiev, S. A review on extractive metallurgy of tantalum and niobium. *Hydrometallurgy* **2020**, *198*, 105496. [CrossRef]
18. Wang, X.; Zheng, S.; Xu, H.; Zhang, Y. Leaching of niobium and tantalum from a low-grade ore using a KOH roast–water leach system. *Hydrometallurgy* **2009**, *98*, 219–223. [CrossRef]
19. Yang, X.; Zhang, J.; Fang, X.; Qiu, T. Kinetics of pressure leaching of niobium ore by sulfuric acid. *Int. J. Refract. Met. Hard Mater.* **2014**, *45*, 218–222. [CrossRef]
20. Makanyire, T.; Jha, A.; Sutcliffe, S. Kinetics of hydrochloric acid leaching of niobium from TiO_2 residues. *Int. J. Miner. Process.* **2016**, *157*, 1–6. [CrossRef]
21. Rodriguez, M.H.; Rosales, G.D.; Pinna, E.G.; Suarez, D.S. Extraction of niobium and tantalum from ferrocolumbite by hydrofluoric acid pressure leaching. *Hydrometallurgy* **2015**, *156*, 17–20. [CrossRef]

22. Ghambi, S.; Sanchez-Segado, S.; Chipakwe, V.; Jha, A. An investigation on hydrofluoric (HF) acid-free extraction for niobium oxide (Nb_2O_5) and tantalum oxide (Ta_2O_5) from columbite/tantalite concentrates using alkali reductive roasting. *Miner. Eng.* **2021**, *173*, 107183. [CrossRef]
23. Wang, X.; Jia, Y.; Ma, S.; Zheng, S.; Sun, Q. Effect of mechanical activation on the leaching kinetics of niobium-bearing mineralisation in KOH hydrothermal system. *Hydrometallurgy* **2018**, *181*, 123–129. [CrossRef]
24. Lyu, J.; Liu, Y.; Ma, Z.; Lyu, X.; Zhou, J. Efficient recovery of niobium and tantalum from ferrocolumbium tantalum by a continuous leaching process using $HF-H_2SO_4-HNO_3$ synergistic system. *Min. Metall. Explor.* **2022**, *39*, 1763–1770. [CrossRef]
25. Rodríguez, O.; Alguacil, F.J.; Baquero, E.E.; García-Díaz, I.; Fernández, P.; Sotillo, B.; López, F.A. Recovery of niobium and tantalum by solvent extraction from Sn–Ta–Nb mining tailings. *RSC Adv.* **2020**, *10*, 21406–21412. [CrossRef] [PubMed]
26. Allain, E.; Kanari, N.; Diot, F.; Yvon, J. Development of a process for the concentration of the strategic tantalum and niobium oxides from tin slags. *Miner. Eng.* **2019**, *134*, 97–103. [CrossRef]
27. Gao, W.C.; Wen, J.K.; Wu, B.; Shang, H.; Liu, X. A novel approach to extract Nb, Y and Ce from a niobium-bearing ore of low grade by roasting $KHSO_4$–H_2SO_4 system. *Rare Met.* **2021**, *40*, 1979–1986. [CrossRef]
28. Yu, X.-L.; Bai, L.; Wang, Q.-C.; Liu, J.; Chi, M.-Y.; Wang, Z.-C. Recovery of rare earths, niobium, and thorium from the tailings of giant Bayan Obo ore in China. *Met. Mater. Trans. B* **2012**, *43*, 485–493. [CrossRef]
29. Sanchez-Segado, S.; Makanyire, T.; Escudero-Castejon, L.; Hara, Y.; Jha, A. Reclamation of reactive metal oxides from complex minerals using alkali roasting and leaching—An improved approach to process engineering. *Green Chem.* **2015**, *17*, 2059–2080. [CrossRef]
30. Ibrahim, M.E.; Orabi, A.H.; Falila, N.I.; Ismaiel, D.A.; Salem, H.M. Processing of the mineralized Black Mica for the recovery of uranium, rare earth elements, niobium, and tantalum. *Hydrometallurgy* **2020**, *197*, 105474. [CrossRef]
31. Redkin, A.F.; Velichkin, V.I. Research of the Uranium, Niobium, and Tantalum Behavior in the Granite Melt–Chloride Fluid System at 750 °C and 1000 Bar. *Geol. Ore Depos.* **2020**, *62*, 372–382. [CrossRef]
32. Lv, Z.; Zhao, D.; Sun, Q.; Liu, C.; Cheng, H.; Yang, F.; Zhang, B. Simultaneous extraction of uranium and niobium from a low-grade natural betafite ore. *High Temp. Mater. Process.* **2023**, *42*, 20220260. [CrossRef]
33. Wang, D.; Li, Q.; Guo, X.; Zou, X. Study on dissolution mechanism of mineral/liquid interface during HF acid leaching niobium-tantalum ore. *Miner. Eng.* **2022**, *188*, 107835. [CrossRef]
34. Jin, S. Extraction of uranium from coarse ore and acid-curing and ferric sulphate-trickle leaching process. *Uranium Min. Metall.* **1994**, *13*, 156–164.
35. Radwan, H.A.; Faheim, A.A.; El-Sheikh, E.M.; El-Wahab, Z.H.A.; Gado, M.A. Optimization of the leaching process for uranium recovery and some associated valuable elements from low-grade uranium ore. *Int. J. Environ. Anal. Chem.* **2021**, *103*, 5259–5281. [CrossRef]
36. Hu, E.; Lei, Z.; Wang, H.; Su, H.; Zhou, W.; Wan, Q.; Zhu, F.; Wang, Q. Extraction of uranium in caustic sludge from the production of nuclear fuel components by countercurrent dissolution and acid curing. *J. Radioanal. Nucl. Chem.* **2022**, *331*, 2445–2450. [CrossRef]
37. Musowoya, M.D.; Hara, Y.R.S.; Chileshe, F.; Mundundu, J.; Parirenyatwa, S. Extraction of Tungsten, Yttrium, and Uranium from Tantalum—Niobium Ore from Muchinga Province in Zambia. In *TMS Annual Meeting & Exhibition*; Springer Nature: Cham, Switzerland, 2023; pp. 95–103.
38. Van Lien, T.; Dinh, T.T.; Dung, N.T.K. Study on leaching systems and recovery for PALUA–PARONG low grade uranium sandstone ores. *Hydrometallurgy* **2020**, *191*, 105164. [CrossRef]
39. El-Hussaini, O.M.; Mahdy, M.A. Sulfuric acid leaching of Kab Amiri niobium–tantalum bearing minerals, Central Eastern Desert, Egypt. *Hydrometallurgy* **2002**, *64*, 219–229. [CrossRef]
40. El-Afandy, A.H.; Yousif, A.M.; Mubark, A.E. Subsequent Separation of Niobium (Nb), Thorium (Th), Rare Earth Elements (REEs), Zirconium (Zr), and Uranium (U) from Abu Rusheid Cataclastic Concentrate, South Eastern Desert, Egypt. *Radiochemistry* **2022**, *64*, 257–267. [CrossRef]
41. Nettleton, K.C.; Nikoloski, A.N.; Da Costa, M. The leaching of uranium from betafite. *Hydrometallurgy* **2015**, *157*, 270–279. [CrossRef]
42. Zheng, C.; Jiang, K.; Cao, Z.; Northwood, D.O.; Waters, K.E.; Wang, H.; Liu, S.; Zhu, K.; Ma, H. Agitation Leaching Behavior of Copper–Cobalt Oxide Ores from the Democratic Republic of the Congo. *Minerals* **2023**, *13*, 743. [CrossRef]
43. Burns, P.C.; Finch, R.J. (Eds.) *Uranium: Mineralogy, Geochemistry, and the Environment*; Walter de Gruyter GmbH & Co KG: Berlin, Germany, 2018; Volume 38.
44. Zheng, C.; Jiang, K.; Cao, Z.; Wang, H.; Liu, S.; Waters, K.E.; Ma, H. Pressure leaching behaviors of copper-cobalt sulfide concentrate from Congo. *Sep. Purif. Technol.* **2023**, *309*, 123010. [CrossRef]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.