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Modelling and simulation of lanthanum (La) and neodymium (Nd) leaching from monazite ore using METSTM

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Modelling and simulation of lanthanum (La) and neodymium (Nd) leaching from monazite ore using METSIM

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Abstract. Digestion and leaching are known as part of the main processing pre-treatment method in the extraction of rare earth elements (REEs). This work aims to carry out the sensitivity analysis of the liquid–solid (L/S) ratio, the type of acids as well as the number of reactors for the recovery of REEs, namely lanthanum (La) and neodymium (Nd) from monazite concentrate through the leaching process. A model was developed and simulated by using METSIM, a software for modelling metallurgical processes. The process was modelled as a two-step process; the first is the digestion, followed by the leaching process to produce monazite leachate. The results show that the optimum L/S ratio was 8:1 to recover the highest amount of REEs, with HCl was found to perform better as a solvent for the recovery of REEs compared to H₂SO₄. In addition, the optimum recovery of REEs was achieved by using three reactors, which is in the range of 70-95%.

1. Introduction

Rare earth elements (REEs) have become essential ingredients for the development and application of high-technology products. The demand for the manufacturing of REEs has increased in many industries due to the sharp growth in information technology such as smartphones, tablet personal computers (PCs), and other electronic applications as well as medical applications. REEs are found in 34 countries in five continents and Brazil is one of the countries with a history of REEs production since 1884 [1], [2]. Many trading corporations have been made aware of the importance of REEs for the industrial economy especially China, the leader of world market for fast growing green technologies [3], [4].

The lanthanide (Ln) series is a group of elements that have similar chemical and physical properties [5]. The group of REEs consists of 15 Ln elements such as yttrium (Y), and scandium (Sc). REEs are often found together in natural soil at low concentration in various mineral ores or concentrate which are bastnasite (LnFCO₃), a fluorocarbonate of the light lanthanides; monazite (Ln, Th)PO₄; and xenotime (Y, Ln)PO₄ as phosphate of light and heavy lanthanides [5]–[8]. REEs may be categorized as heavy rare earth elements (HREEs) and light rare earth elements (LREEs), however, monazite concentrate consists mainly of the latter, which are Lanthanum (La), Cerium (Ce), Praseodymium (Pr), and Neodymium (Nd) [5].

Leaching is a separation method for substances from solid by dissolving it in a liquid acid solution. However, it is a complicated process due to the similarity of the REEs' chemical and physical properties, as the chemical bonding between the REE's and other elements cannot be broken down easily [6], whereby in aqueous solutions, REE's are present as trivalent cations [9]. The chemical processing of



REEs monazite concentrate occurs in two steps [9]–[11]; the first step is acid digestion which typically includes a chemical separation reaction between REEs ore and the liquid acid solution. This separation process effectively increases the percentage of REEs recovery [12] in the form of precipitate concentrate. Deionised water is added and dissolved with REEs sulphate in the leaching stage. However, some of impurities, unreacted monazite which consists of REEs phosphates and insoluble compounds which were produced in the digestion process remains as precipitate.

The liquid to solid (L/S) ratio affects the viscosity of the solution with the solvent agent. Higher L/S ratio provides a sufficiently low viscosity for the leachate solution to circulate freely, hence better leaching. Several studies have been done on the effect of L/S ratio for the extraction of rare earth elements from monazite concentrate [13]. The study was carried out using 93-95% of H_2SO_4 and the L/S ratio tested varied at 1, 1.75 and 2.5 for digestion process, while for leaching process were 5, 7.5 and 10 [13]. The results show that the recovery of REEs increased with the increase of L/S ratio. This is because a higher L/S ratio has the possibility of more effective contact between the solid and liquids present in the liquid phase in mixing digestion removes the solid products formed on the particles [13].

The feasibility of various acid types for the chemical processing of REEs from monazite concentrate has also been studied. Sulphuric acid (H_2SO_4), hydrochloric acid (HCl), and nitric acid (HNO_3) are examples of strong acids, which dissociate completely in water at moderate concentrations. All acids are corrosive to living tissues and attract water molecules from the surrounding. HCl is the strongest acid, followed by H_2SO_4 and HNO_3 [14]. Different authors have studied the chemical processing of monazite using acidic solutions of H_2SO_4 , HCl, and HNO_3 under different processing conditions to dissolve REEs to get leachate concentrate [12], [15], [16]. The results show that increasing acid concentration slightly increased the REEs recovery. HCl was found to be the most effective and most commonly used solvent agent because of its capability to recover more than 90% apatite-based ore [12]. Apatite and monazite ore or concentrates phosphate-based and are rich in LREEs [11]. REEs mainly exist as trivalent compounds that can be dissolved in HCl and form soluble chlorides [10]. However, using a highly concentrated HCl poses corrosion problems to the equipment and also increases the operating cost [17]. Compared to HCl, H_2SO_4 is a weaker solvent agent for apatite-based ore, which is rich in calcium [12], as the side reaction from Ca(REEs) sulphate precipitation limits the REEs recovery. The HCl leaching does not exceed 80% even when excess acid was used [12]. HNO_3 is chemically compatible with various mineral ores and more economical. However, it is not strong enough as an oxidising agent by itself to convert organic molecules and does not form any insoluble compounds with the metals and non-metals compared to HCl and H_2SO_4 [14]. Another important factor in the REEs recovery is the number of reactors dedicated to the acid digestion step. During digestion, the bonds between REEs and oxygen are broken down to form REEs sulphates which are soluble in water, also known as leachate [13]. To increase the amount of material dissolved, the leaching reactor must be increased and for a conversion of 70%, the reactor should be at least five, which implies that the residence time is also increased [18].

There are many simulators available commercially for process modelling and simulation, such as Aspen Plus, Aspen HYSYS, HSC Sim, CFD, SysCAD, and METSIM. They are powerful tools to improve process plant performance in terms of design, plant start-up, and operation, and can be utilised for continuous improvement. METSIM is one of the available process simulators that specialises in metallurgical processes. This software is capable of performing steady-state and dynamic modelling for most mineral processing operating units and reducing the number of experiments required in the laboratory [19], and has with extensive rare earth elements database [20]. METSIM has been successfully applied in the modelling and simulation of the leaching of gold and copper using cyanide [21] and extraction of arsenic from solution through scorodite crystallisation [19], with excellent agreement with the experimental results.

Modelling and simulation work on LREEs are very limited in the open literature. Zhao et al. [22] simulated the leaching of ionic rare earth carbonate with HCl using METSIM. Kumari et al. [8] also simulated the steady-state leaching with HCl as a solvent to optimise the extraction of bastnasite concentrate. On the other hand, Yaqi et al. [23] studied the radiation damage resistance of the LaPO₄,

monazite-type ceramics using atomistic modelling techniques. The simulator used was LAMMPS, a molecular modelling simulator software, to improve understanding of the radiation-induced amorphisation process in monazites.

The increasing demand of REEs and their limited supply have attracted researchers to perform further investigation. However, due to the high cost of experimental works in REEs processing, simulation studies is an alternative method in process improvement. Hence, the objective of this work is to develop a process model and simulate the leaching of monazite concentrate to recover two types of REEs; La and Nd using METSIM. Subsequently, sensitivity analysis was carried out which includes the effect of the number of reactor, acid types, and liquid to solid (L/S) ratio.

2. Methodology

2.1 Simulation Framework

The digestion and leaching processes are the main processes for extraction of metal species from monazite concentrate. Figure 1 illustrates the block diagram that represents the leaching process that is being considered in this study. The details of equipment are described in the following subsections.

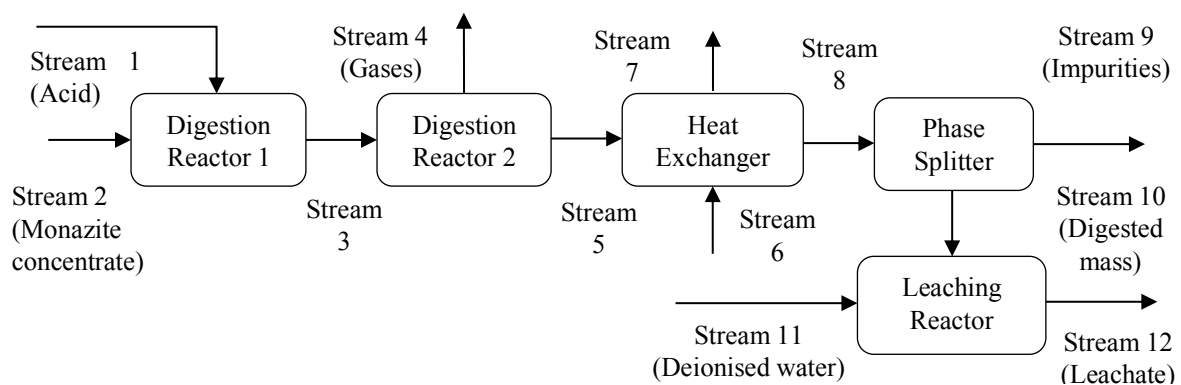


Figure 1. Leaching process [20, 27].

Figure 2 shows the flowchart for the process modelling in METSIM. The component database (DBAS) was added in METSIM and the REEs component database was imported from the HSC software, which includes H_3PO_4 , LaPO_4 , NdPO_4 , $\text{La}_2(\text{SO}_4)_3$, $\text{Nd}_2(\text{SO}_4)_3$, $\text{La}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$, and $\text{Nd}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$. The process involves several components at different phases as shown in table 1. The extraction rate of monazite concentrate was set as 90%, based on previous experimental results by Sadri et al. [13]. The addition of H_2SO_4 was controlled by mass balance [1, 24]. The simulation results were compared to the same set of data obtained from the literature according to the works by Sadri et al. [13] & [24].

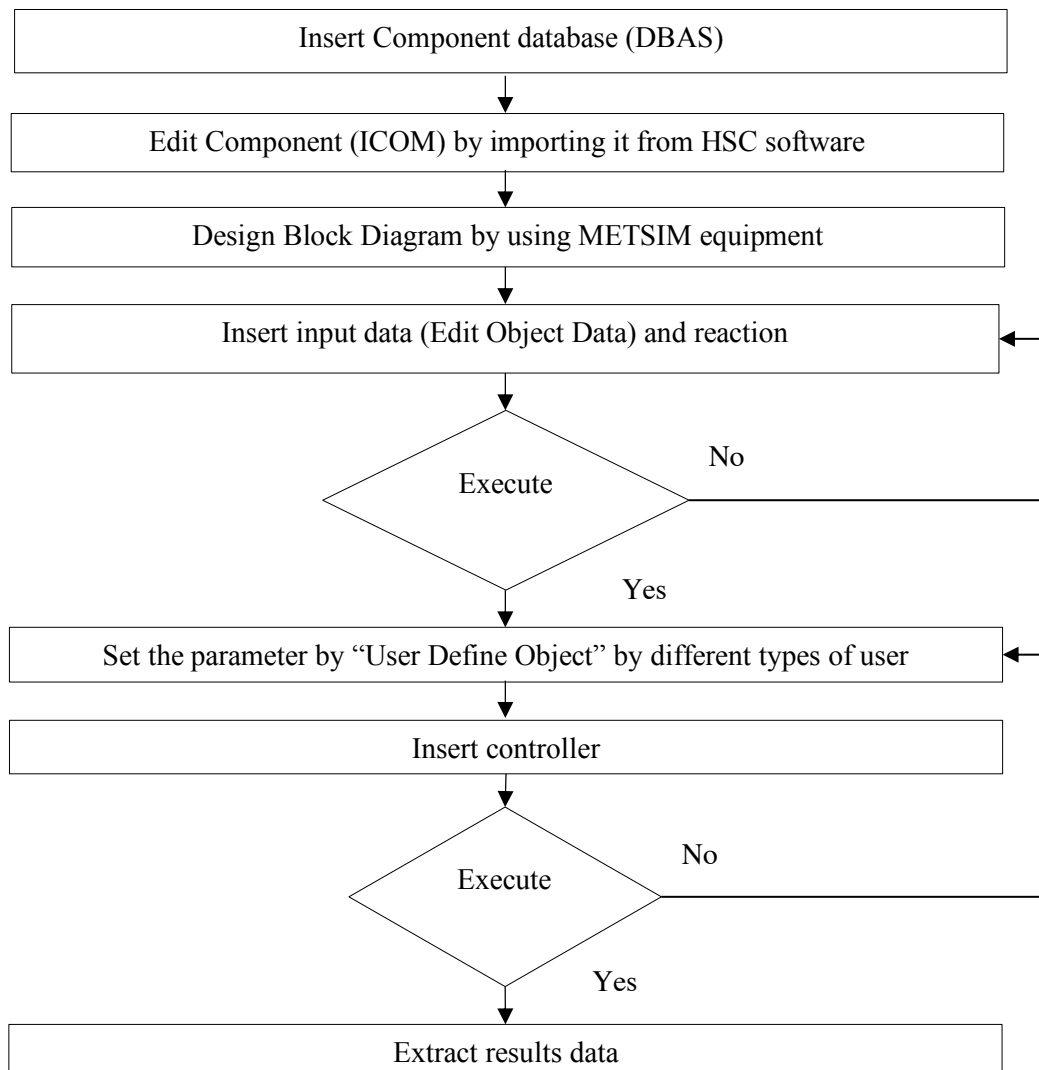


Figure 2. Flowchart of chemical processing using METSIM.

Table 1. Components added in METSIM at different phases.

Solid		Aqueous	Gas
sSiO ₂	sFe ₂ O ₃	aH ₂ O	SO ₃
sLa ₂ O ₃	sLaPO ₄	aH ₂ SO ₄	
sNd ₂ O ₃	sNdPO ₄	aLa ₂ (SO ₄) ₃	
sCeO ₂	sLa ₂ (SO ₄) ₃ ·9H ₂ O	aNd ₂ (SO ₄) ₃	
sP ₂ O ₅	sNd ₂ (SO ₄) ₃ ·8H ₂ O	aH ₃ PO ₄	

2.1.1 Digestion reactor 1

In METSIM, the first digestion reactor was represented by Tank Agitated with Coil (TAC) model block, denoted as Digestion Reactor 1. This model block is suitable to control temperature and can be represented as an aerated continuous stirred tank reactor (CSTR). Table 2 is the monazite concentrate compositions, while table 3 is the process conditions for the input to the Digestion Reactor 1. In this reactor, it was assumed that the reactions that occurred were as shown in reaction equations (1-2), to produce monazite concentrate leachate. Stoichiometric method was applied for the calculation of the mass balance. A 90% conversion was assumed to ensure high recovery of the REEs. The reactor was isothermal, the temperature was set as 225°C.

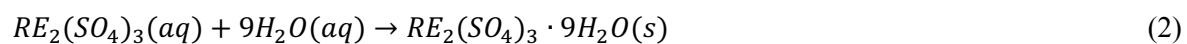
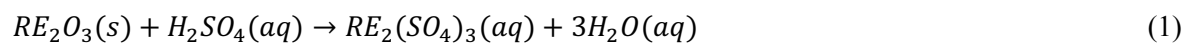
Table 2. The monazite concentrates compositions of monazite.

Monazite concentrate compositions	La ₂ O ₃	Nd ₂ O ₃	P ₂ O ₅	CeO ₂	SiO ₂	Fe ₂ O ₃
wt. (%)	6.3	6.8	12.2	10.1	37.4	27.2

Table 3. Process conditions of input.

Process Conditions	Unit	Feed stock (Monazite concentrate)	Acid (H ₂ SO ₄)
Mass flowrate	kg/h	30	50
Temperature	°C	25	150
Purity	%	-	93 [20]

Chemically, monazite concentrate is particularly rich in LREEs [11]. Two main types of LREEs considered in this study were La and Nd. The chemical processing started at the first digestion reactor where the monazite concentrates oxide (RE₂O₃) reacted with H₂SO₄ as shown in equation (1). Subsequently, deionised water was added to dissolve the monazite sulphate as shown in equation (2) [25], [26].



2.1.1.1 Digestion reactor 2

The second reactor, denoted as Digestion Reactor 2, was also represented by TAC model block. In this reactor, it was assumed that the reaction between monazite concentrate phosphate and H₂SO₄ occurred to produce monazite leach solution, La₂(SO₄)₃, Nd₂(SO₄)₃, and H₃PO₄ in stream 5. As shown in equation (3), a 90% conversion was also assumed, similar to the first reactor [6], [7], [13], [25]. The parameters were set as "User Defined Object", attached to this reactor are as shown in table 4.

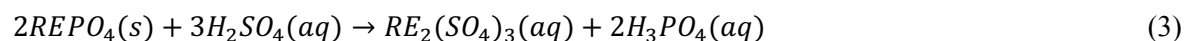


Table 4. "User Define Object" in METSIM.

Name	Type
US_Digest_LS_Ratio	Scalar
US_Digest_Temperature_C	Scalar
US_Digest_Time_hrs	Scalar
DigestCase	Function
UV_Digest_Result_Headings	Vector
UM_Digest_Result	Matrix

2.1.1.2 Cooling reactor

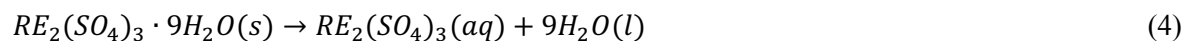
The cooling reactor denoted as Heat Exchanger model block is shown in figure 1. The outlet of the Reactor 2 was fed to the heat exchanger to reduce the temperature for the leaching process [20], [27] from 225 °C (Stream 5) to 75 °C (Stream 8) while the cold water supply at 30°C (Stream 6), increased to 65°C (Stream 7). The mass flowrate for cooling water was calculated by METSIM.

2.1.1.3 Phase splitter

The phase splitter was used to split one or more input streams into two or more output streams with different relative phases. The total input of all streams was added together and equilibrium calculations were performed prior to splitting the phase into various output streams. This was represented by “Phase Splitter (SPP)” model block in METSIM. Stream 9 from SPP was 100% liquid phase, released as the output upstream, while stream 10, which was 100% solid phase, was released as the output downstream to be diluted with deionised water to produce higher grade REEs.

2.1.1.4 Leaching reactor

The Leaching Reactor is represented by the TAC model block, where deionised water in Stream 11 was used in the reaction with monazite concentrate leachate to ensure higher recovery of leachate [27]. The temperature for both deionised water and leaching reactor was set at 75 °C and the reaction that took place is shown in equation (4).



2.1.1.5 Controller

In Digestion Reactor 2, the L/S ratio, time, and temperature were set by the Instrumental Controller (IC) to maintain the reactor conditions. In Stream 1, Feed Forward Controller (FFC) was applied to adjust L/S ratio for the sensitivity analysis. The FFC were set at value function (VF) and other data needed are as shown in equation (5-8):

For FFC:

$$\text{Unit operation (OP)} = 2 \quad (5)$$

$$\text{Stream number (SN)} = 1 \quad (6)$$

$$\text{Set point (SP)} = 0.93 \quad (7)$$

$$\text{Value function (VF)} = \frac{(\text{VKGH } s1)}{(\text{SC VKGH } s2)} \quad (8)$$

where,

VKGH = Volume Kilo Gram per Hour

SC = Solid Compound

s = Stream

2.1.1.6 Assumptions

Processes with multiple reactions with many input and output streams are more difficult to describe in analytical terms [26]. However, the models contain simplifications to ensure that the problems are easier to understand, handled, and solved, as well as allowing the user to gain a better perception of the process. In this study, the following assumptions were made:

- Process occurs in a well-mixed reactor [30–32].

- Reaction occurs at steady-state.
- There is isothermal temperature distribution in the reactor [18].

2.2 Sensitivity analysis

2.2.1 Liquid–solid ratio

The L/S ratio is an important parameter as it affect chemical processing, in particular the degree of solution viscosity in contact with solvent agent [31]. Here, L is the acid solution, while S is the monazite concentrate itself. In this study, the L/S ratio tested was 0.5:1 to 8:1, to evaluate the optimum recovery of REEs.

2.2.2 Acid types

There are various types of acid being used by various researchers in the chemical processing of REEs such as the common acid H_2SO_4 and HCl [5], [9], [32]. In this study, H_2SO_4 and HCl were tested to justify the type of acid and its maximum concentration that ensures high recovery of REEs. The mass flow of acid applied in this study were 50, 100, 150, and 200 kg/h. Table 5 shows the types of acid used with their respective reaction equations for La and Nd.

Table 5. Types of acid tested and reaction equations.

Types of acid	Equations
H_2SO_4	$La_2O_3(s) + 3H_2SO_4(aq) \rightarrow La_2(SO_4)_3(aq) + 3H_2O(aq)$ (9)
	$Nd_2O_3(s) + 3H_2SO_4(aq) \rightarrow Nd_2(SO_4)_3(aq) + 3H_2O(aq)$ (10)
	$La_2(SO_4)_3(aq) + 9H_2O(aq) \rightarrow La_2(SO_4)_3 \cdot 9H_2O(s)$ (11)
	$Nd_2(SO_4)_3(aq) + 8H_2O(aq) \rightarrow Nd_2(SO_4)_3 \cdot 8H_2O(s)$ (12)
	$2LaPO_4(s) + 3H_2SO_4(aq) \rightarrow La_2(SO_4)_3(aq) + 2H_3PO_4(aq)$ (13)
	$2NdPO_4(s) + 3H_2SO_4(aq) \rightarrow Nd_2(SO_4)_3(aq) + 2H_3PO_4(aq)$ (14)
HCl	$La_2O_3(s) + 6HCl(aq) \rightarrow 2LaCl_3(aq) + 3H_2O(aq)$ (15)
	$Nd_2O_3(s) + 6HCl(aq) \rightarrow 2NdCl_3(aq) + 3H_2O(aq)$ (16)
	$LaCl_3(aq) + 7H_2O(aq) \rightarrow LaCl_3 \cdot 7H_2O(s)$ (17)
	$NdCl_3(aq) + 6H_2O(aq) \rightarrow NdCl_3 \cdot 6H_2O(s)$ (18)
	$LaPO_4(s) + 3HCl(aq) \rightarrow LaCl_3(aq) + 2H_3PO_4(aq)$ (19)
	$NdPO_4(s) + 3HCl(aq) \rightarrow NdCl_3(aq) + H_3PO_4(aq)$ (20)

2.2.3 Number of reactor

To accelerate the monazite concentrate recovery with improved purity, the reaction process occurred continuously in a cascade of large agitated tanks [33]. In this study, the number of reactor was analysed to evaluate if increasing the continuous chemical processing reactor will increase the recovery of the REEs. The reaction equations involved in different reactors are shown in table 6. In this study, three reactors were used, where the first reactor was Digestion Reactor 1, which was explained earlier in Subsection 2.1.1.1. The second reactor was Digestion Reactor 2, which was the basis for this case study, and the simulation framework was explained in Subsection 2.1.1.2. The last reactor was Leaching Reactor in which its output was analysed. In this case study, additional reactors were added with similar process conditions as Digestion Reactor 2.

Table 6. Reactions set in different reactors.

Reactor Number	Equations involved
Digestion Reactor 1	Reaction Equation (15-18)
Digestion Reactor 2	Reaction Equation (19-20)
Digestion Reactor 3	Reaction Equation (19-20)
Digestion Reactor 4	Reaction Equation (19-20)

3 Results and discussions

3.1 Model validation

A validation case study based on an experimental work [13] was set up and run to compare with the results obtained from the developed model, where similar conditions were applied. Table 7 shows the results of the recovery of La and Nd in the simulation, which were 91.54% and 92.43%, respectively. They are in excellent agreement with the literature [13], where there was less than 1% error between the experimental and simulation work. The validation results confirm that the developed model is appropriate for the simulation and sensitivity analysis.

Table 7. Validation simulation results with experimental data [13].

REEs	REEs Recovery (%)		Percent error (%)
	Simulation (This study)	Experiment [13]	
La	91.54	92.44	0.98
Nd	92.43	92.86	0.46

3.2 Liquid–solid ratio

The effect of L/S ratio on the recovery of REEs is shown in figure 3 **Error! Reference source not found.** The L/S ratio was varied from 0.5:1 to 8:1. This was to ensure that there is enough liquid phase to react with the solid phase. The recovery of La and Nd showed an increasing trend, where the percent recovery was improved with increasing L/S ratio. At the lowest L/S ratio, of 0.5:1, the percent recovery of La and Nd were only 59.85% and 63.96%, respectively. This is consistent with a previous study which found that for lower L/S ratio, the recovery was also lower due to the formation of stable hydrolysis products and ions [27]. The recovery of Nd increased sharply at L/S ratio of 2, which was 90.99%, compared to La, which was 75.63%, which increased rather uniformly with increasing L/S ratio. The recovery of Nd increased in a more apparent manner compared to the recovery of La, as the maximum recovery Nd was 97.75% while the recovery La was 93.91% at the maximum 8:1 L/S ratio. As high L/S ratio may reduce the viscosity of the slurry from the presence of excess acid in the reacting medium, it can improve the mass transfer between the solid and liquid phases [31]. This allows for the leaching solution to circulate freely and to interact, which means a larger driving force for the diffusion of the dissolved ion [31][31][31][32]. In addition, the mixing of the acid and solid concentrate in the digestion reactor may remove the digested mass formed on the surface of the solid particles and increase the contacts between the phases [13], thus increasing the recovery of the REEs.

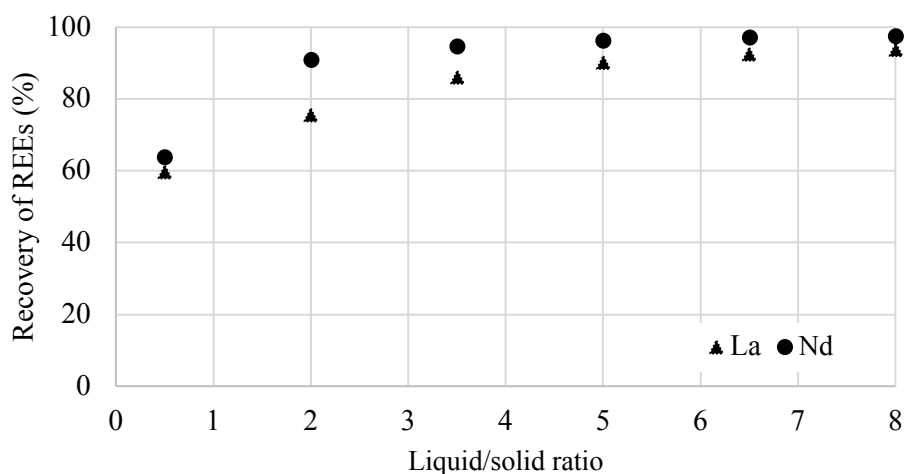


Figure 3. Recovery of REEs by different L/S ratios.

3.3 Acid types

The second case study was to evaluate the effect of using different types of acid for the chemical processing. Two types of acid were used in this study H_2SO_4 and HCl with concentration 93%. The mass flowrate of ore was maintained at 30 kg/h. In the digestion reactor, the solvent concentration is also important as it affects the dissolution of the soluble elements for different types of acid. Therefore, in addition to the type of acid for the chemical processing, the impact of various mass flowrates of acid was also tested at 50, 100, 150, and 200 kg/h, with 50 kg/h as the base case.

The recovery of La and Nd increased with the increase of acid mass flowrate as shown in figure 4 and figure 5, respectively. From figure 4, for the base case study of mass flowrate of acid feed at 50 kg/h, the recovery of La was 74.81% and 68.67% for HCl and H_2SO_4 , respectively. These values further increased as the mass flowrate of acid was increased. At the highest mass flowrate of 200 kg/h, the recovery of La using HCl was 91.29% while for H_2SO_4 was slightly lower at 89.21%. This shows that for La, HCl was more efficient compared to H_2SO_4 . For the effect of increasing mass flowrate of different acids used to recover Nd as shown in figure 5, a similar trend was observed. For the base case, the recovery of Nd was 90.69% and 70.44% for HCl and H_2SO_4 , respectively. Similar to the recovery of La, the recovery of Nd also increased with the increasing mass flowrate of the acids. At 200 kg/h, the recovery of Nd was 96.78% for HCl and 89.82% for H_2SO_4 .

These results show that the recovery of Nd was higher than La for both acids tested. This may be due to the higher solubility of Nd than that of La in acidic solvents. In addition, the trivalent REEs ions are stable in a solution with a pH value of lower than 6, which is the pH value of both for H_2SO_4 and HCl [34]. These results also show that HCl was able to achieve higher recovery for both REEs. In the industry, HCl is more preferable as it is better at extracting monazite concentrate and complex chloride concentrates, as well as having higher metal complex solubility [35].

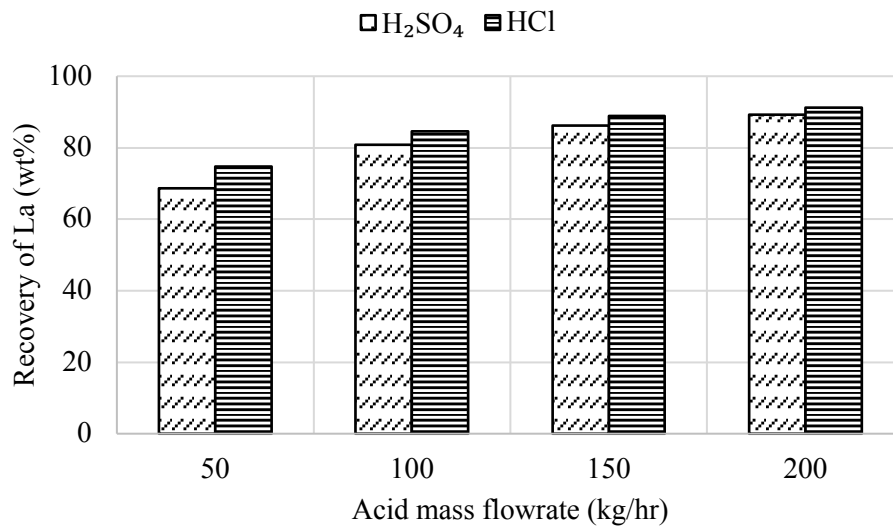


Figure 4. Recovery of La by using H₂SO₄ and HCl.

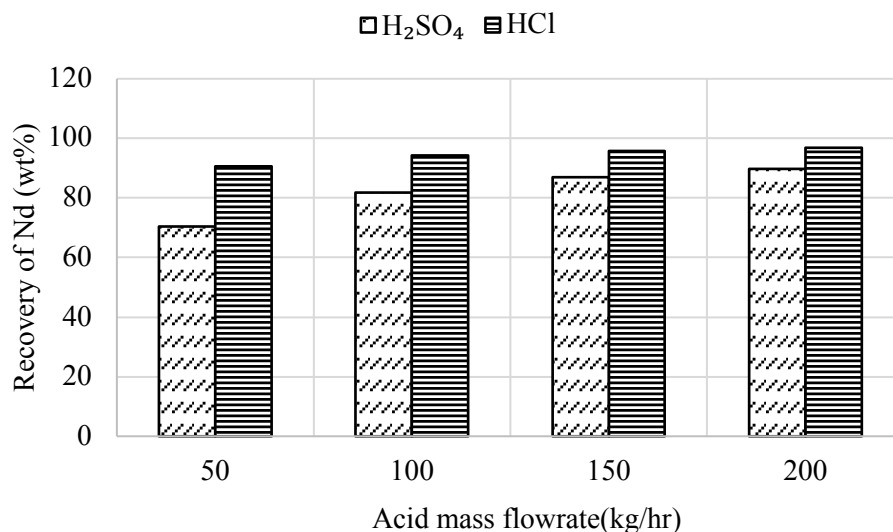


Figure 5. Recovery of Nd by using H₂SO₄ and HCl.

3.4 Number of reactor

A series of simulations was carried out in METSIM to study the effect of the number of reactors on the recovery of REEs. Digestion Reactors 1 and 2 are the main reactors which were used in the beginning of the process for the production of REEs leachate concentrate. In this case study, Digestion Reactor 2 was set as the base case for the additional reactors. The mass of acid flowrate was maintained at 50 kg/h and the conversion was fixed at 90%. Figure 6 shows the effect of increasing number of reactors on the recovery of REEs. The recovery of La and Nd were 88.82% and 83.30%, respectively, at Reactor 3, which were the maximum values obtained, only after one additional reactor was added to the system. The improvement of the recovery of La was higher compared to the recovery of Nd. This can be observed in between Reactors 2 and 3, whereby the percent recovery improvement for La was 7.91% while for Nd was 7.25%. However, there was no significant improvement of the recovery of REEs between Reactors 3 and 4. This may be related to the high rate of dissolution due to the additional

number of reactors. Furthermore, an increasing number of reactor increased the recovery of REEs due to the additional mixing, which also implies the increase of residence time [33].



Figure 6. Recovery of REEs by number of reactors.

4 Conclusion

This paper focuses on the development of a process model for the chemical processing of monazite concentrate which contained approximately 25% REEs. In a validation case study, a developed model in METSIM was compared with data from experimental work from the literature, and shows excellent agreement on the results of REE recovery. Sensitivity analysis was carried out for three factors, which were the L/S ratio, acid types, and number of reactors. The highest L/S ratio tested, 8:1, was able to recover the maximum amount of REEs. When comparing the efficiency of HCl and H₂SO₄, the former performed better in the recovery of the REEs. The highest number of reactors that achieved maximum recovery of REEs was three, whereby further increase did not improve the recovery. The developed model can be utilised for chemical processing designs as well as improving the efficiency of the current process, eliminating the need of tedious experimental work.

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