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The decomposition possibility of vietnamese monazite concentrate by the pressure alkali method

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Abstract. The authors have studied the decomposition of monazite concentrate by alkali. They investigated the necessary decomposition conditions such as temperature, time, particle size and the ratio between alkali and monazite concentrate by mass (wt/wt). The decomposition is best performed at >140°C, within 8 hours, the required alkali and monazite concentrate ratio was 1,4/1, the ore particle size needs to be smaller than 48 μm. The decomposition efficiency was also only about 70%. If the desired recovery efficiency was higher than 90%, the alkali/concentrate ratio by mass needed to be at least 4/1, resulting in a large amount of residual alkali. To improve this process, the authors studied the decomposition of monazite concentrate by alkali under pressure. The monazite was taken from Ham Tan deposit. The effecting parameters such as temperature, time, ratio between NaOH/concentrate by mass and particle size of the concentrate were investigated. The results showed that, under the effect of pressure, decomposition occurs faster, more thoroughly and decomposition efficiency increases. The suitable temperature for concentrate decomposition was from 180 to 210°C, corresponding to a pressure of 4 to 7 at. The decomposition time was also reduced to 2 hours and the particle size of the concentrate was also larger to 55 μm with 70% alkali and ratio of alkali/concentrate was 1/1. The decomposition efficiency of rare earth elements reached 95%, while under the same conditions, U was 50% and Th was 77%. When the reaction time increased, the efficiency of rare earth elements decomposition did not change much, but the decomposition efficiency of radioactive elements tended to increase. For particle sizes >55 μm, the process efficiency decreased sharply, and changes in reaction conditions were needed to increase the decomposition efficiency.

Keywords: monazite, rare earth elements, thorium, uranium, phosphates, alkali leaching

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Возможность разложения вьетнамского монацитового концентрата щелочным методом под давлением

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Аннотация. Изучено разложение монацитового концентрата щелочью. Были исследованы условия разложения, такие как температура, время, размер частиц и массовое соотношение между щелочью и монацитовым концентратом по массе. Согласно литературным данным, процесс разложения лучше всего проводить при температуре >140 °C в течение 8 часов, требуемое соотношение щелочи и монацитового концентрата составляет 1,4/1, размер частиц руды должен быть меньше 48 мкм. Эффективность разложения составляет около 70 %. Если достигать степени разложения свыше 90 %, то соотношение щелочи/концентрата по массе должно быть не менее 4/1, что приводит к большому количеству остаточной щелочи, в последующем требующей дополнительных затрат на регенерирование. Для решения проблемы использования избыточного количества щелочи был изучен процесс разложения монацитового концентрата при повышенном давлении щелочью. Был использован монацитовый концентрат месторождения Хам Тан (Вьетнам, провинция Баньтхуан). Исследованы основные параметры, влияющие на полноту протекания процесса, такие как температура, время, соотношение NaOH/концентрат по массе и размер частиц концентрата. Результаты показали, что под воздействием давления разложение происходит быстрее и эффективность разложения увеличивается. Оптимальная температура для разложения концентрата составляет от 180 до 210 °C, что соответствует давлению от 4 до 7 атм. Время разложения также сократилось до 2 часов, а размер частиц концентрата также увеличился до 55 мкм при использовании 70 %-ного раствора щелочи и соотношения щелочь/концентрат 1/1. Эффективность вскрытия редкоземельных элементов достигла 95 %, в то время как при тех же условиях степень вскрытия для U составила 50 %, а для Th – 77 %. При увеличении времени реакции эффективность разложения редкоземельных элементов не сильно менялась, но для радиоактивных элементов имела тенденцию к росту. При размерах частиц >55 мкм эффективность процесса резко снижалась, и для увеличения эффективности разложения требовалось изменение условий реакции.

Ключевые слова: монацит, редкоземельные элементы, торий, уран, фосфаты, щелочное выщелачивание

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Introduction

Nowaday, along with the development of society, the demand for the market of rare earth elements (REE) is increasing, making the demand for rare earth processing higher. REE can be found in the minerals bastnazite, monazite, xenotime. Vietnam is one of the countries with significant REE reserves in the world. According to survey reports, the total reserves of REE are about 3.5 million tons [1]. Monazite is a light rare earth mineral containing Th in the form of phosphate salt ([REE,Th]PO_4). Monazite is mainly distributed in some countries such as India, Brazil, Australia. Vietnam also has a fairly large reserve of monazite. It is mainly concentrated in the original ores or accompanying titanium-zircon sands. The main components in monazite concentrate are shown in Table 1 [2, 3].

From Table 1, it can be seen that, for Vietnamese monazite concentrate, the content of TREO, radioactive (ThO_2) and P_2O_5 components is quite similar to that of Thailand, Korea and Malaysia.

The main monazite concentrate processes

Monazite concentrate is processed mostly with two main ways: using sulfuric acid and alkali [4, 5]. There were many independent studies on monazite processing conducted in Indonesia, Malaysia, Korea etc., in which, the alkali method had shown many advantages like recovery, processing and refining ability after decomposition [5–10].

The acid technology scheme is shown in Fig. 1 [6].

Table 1. *Main composition of monazite concentrate in some contries in the world [2, 3]*

Таблица 1. *Состав монацитовых концентратов в некоторых странах мира [2, 3]*

Countries/Страны	Composition/Состав (%)			
	Total REE (TREE) Общие РЗЭ	U_3O_8	ThO_2	P_2O_5
Australia/Австралия	61.33	0.34	6.55	26.28
India/Индия	60.00	0.35	9.65	26.23
Malaysia/Малайзия	59.65	0.24	5.90	25.70
Thailand/Таиланд	60.20	0.40	5.76	26.52
South Korea/Южная Корея	60.20	0.45	5.76	26.52
Italya/Италия	35.24	15.64	11.34	31.02
Brazil/Бразилия	58.13	N.D	10.05	31.82
Sri Lanka/Шри-Ланка	53.51	0.1	14.32	26.84
Vietnam/Вьетнам	58.85	0.31	5.03	26.13

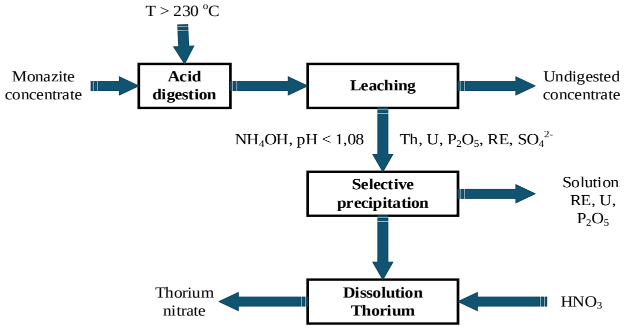

Fig. 1. *Technological diagram of monazite ore processing by acid method*

Рис. 1. *Технологическая схема переработки монацитовой руды кислотным способом*

Monazite ore processing includes the following stages:

- *Acid decomposition of ore concentrate*: requires high concentration of sulfuric acid, usually >93% and decomposition temperature is usually >230°C. This temperature depends on the acid concentration used. The higher the concentration, the lower the temperature required for the reaction and vice versa.
- *Water separation*: use water to perform this stage. The two main products obtained include: undecomposed ore concentrate and sulfate salt solution of Th, U and rare earth elements.
- *Selective precipitation*, using NH_4OH to convert Th from soluble salt into hydroxide. Filter to separate the precipitate (hydroxide) and the solution containing salt of rare earth elements.
- *Dissolve the precipitate* with dilute HNO_3 solution, to obtain thorium nitrate solution. The basis of the stage depends on the different pH dissolution of thorium nitrate and rare earth nitrate. Selective and fractional dissolution. For decomposition using sulfuric acid, the reaction temperature usually takes place from 180–240°C under the condition of concentrated sulfuric acid >93% and the ratio by mass of acid/concentrate 1,4/1 [11–13]. With this method, the actual decomposition efficiency is always >90%, however, the recovery of rare earth is not really large, only about 70%. In addition to the generation of radioactive waste, technologies are needed to handle it. Therefore, in fact, the acid method is a classic one, but less used. It is also suitable for rare earth ores containing low radioactive content, often applied on a large scale where the level of environmental impact assessment is less focused on because the waste is highly acidic and needs to be treated. Therefore, the cost of the obtained product is basically high. However, the great advantage is that it can be used for many different rare earth ores. The disadvantage of the method is that the amount of waste is large, in addition to excess acid, the ability to manage radioactive waste must also be taken into account.

Main reactions:

- Decomposition reaction (Digestion by sulfuric acid)

$$2REEPO_4+3H_2SO_4 \rightarrow REE_2(SO_4)_3+6H^++PO_4^{3-},$$

$$Th_3(PO_4)_4+6H_2SO_4 \rightarrow 3Th(SO_4)_2+12H^++4PO_4^{3-}.$$

- Selective precipitation:

$$Th(SO_4)_2+4NH_4OH \rightarrow Th(OH)_4+2(NH_4)_2SO_4.$$

With the method of decomposing monazite concentrate by alkali, concentrated NaOH (>50%) is the most widely used and most popular besides KOH and Na_2CO_3. The diagram of the method is shown in Fig. 2. Alkaline technology includes the following stages:

- magnetic separation to increase the monazite content to 99%;
- alkaline decomposition: forming rare earth and thorium hydroxides;
- removal of phosphate salts and excess alkali: using pure water to perform;
- neutralization to remove hydroxides of thorium and uranium. This is done by using HCl or HNO_3 acid solution;
- removal of Ra by co-precipitation with $BaSO_4$. Using a mixture of Na_2SO_4 and $BaCl_2$ solutions with appropriate concentrations to perform barium sulfate precipitation. The result is a rare earth salt solution.

With the decomposition using NaOH, the decomposition temperature was usually lower, about 140–160°C, with high concentration of NaOH ~70%, for a long time of about 8–10 hours. The mass ratio between NaOH/concentrate was 1,4–2/1, which could recover 70% of REE from the ore. To increase the efficiency, a much larger ratio of alkali/concentrate was needed [14, 15]. When using KOH, the results were almost similar to those of NaOH. The required temperature was from 150 to 250°C, the ratio of KOH/concentrate by weight (wt/wt) is from 1,4/1 to 2/1; the decomposition time was 4 hours, the recovery efficiency of rare earth elements is about 70%. To increase the recovery efficiency, the decomposition temperature was >200°C, the reaction time was 4 hours and the ratio of KOH/concentrate by weight was 4/1. Then, the recovery efficiency of rare earth elements reached 90% [16]. The alkaline technology scheme is shown in Fig. 2 [17, 18].

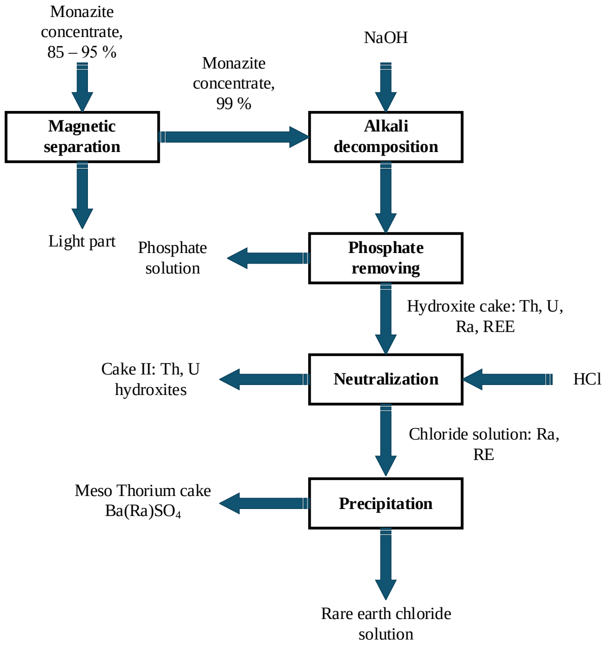

Fig. 2. *Flowchart of monazite ore refining technology*

Рис. 2. *Блок-схема технологии переработки монацитовой руды*

Main reactions:

- Decomposition reaction (Digestion by alkali)

$$REEPO_4+3NaOH\rightarrow REE(OH)_3+Na_3PO_4,$$

$$Th_3(PO_4)_4+12NaOH\rightarrow 3Th(OH)_4+4Na_3PO_4.$$

- Leaching reaction by HCl acid:

$$REE(OH)_3+3HCl\rightarrow REECl_3+3H_2O,$$

$$Th(OH)_4+4HCl\rightarrow ThCl_4+4H_2O.$$

Monazite concentrate processing in Vietnam

Since the 1990s, Vietnam has conducted many studies on processing rare earth concentrates, including the acid method using concentrated sulfuric acid and the alkaline method using NaOH to decompose rare earth concentrates. In addition, there are a number of cooperation and technology transfer programs for processing rare earth concentrates between Vietnam and some countries such as India (monazite), Japan (bastnaite). The earliest cooperation program with India was implemented in the late 1990s and early 2000s. The studies were conducted and implemented to process monazite concentrates by the concentrated alkaline decomposition method (NaOH, 70%) deployed on an industrial scale in India. The cooperation program with Japan was carried out for bastnasite concentrates with acid decomposition in the period from 2012 to 2015. The studies also showed the suitability of the alkaline method for Vietnamese monazite concentrates. In the cooperation with India, monazite concentrates obtained from placers were used to conduct survey and evaluation studies. Alkaline (NaOH) decomposition experiments with conditions of reaction time, decomposition temperature, alkali/concentrate ratio by mass, and monazite concentrate particle size were carried out.

The results showed a correlation with the decomposition conditions of previous studies. It is required reaction temperature higher than 140°C, decomposition time approximately 8 hours, alkali concentration >70% and the mass ratio between NaOH/concentrate was 1,4/1. The rare earth recovery efficiency reached nearly 70%. The particle size required to ensure efficient operation is d<48 μm. To increase the recovery of REE to 80%, the required alkali/concentrate ratio was 2/1 and for 90% this ratio was 4/1.

It can be seen that the recovery efficiency of REE is only about 70% by the alkaline decomposition method. To increase the efficiency of the process, it is necessary to increase the ratio between alkali and concentrate by mass or further grind the concentrate. Besides, according to reports on pressure impact on the decomposition of rare earth concentrates, pressure will make the efficiency of the process better [19, 20]. The decomposition reaction temperature is similar to that when working under normal conditions, however, the effective particle size is larger, the reaction time is shortened [21].

It should be mentioned that other methods of monazite decomposition can be used. For instance, processing some chemically inert concentrates with NH_4HF_2 is possible and may be realized in industry [22–25]. In case of monazite the application of ammonium bifluoride has some disadvantages. They are mostly related to formation of REE fluorides, which a more resistant in comparison with respective hydroxides.

With the requirements for improving the previous monazite concentrate processing technology, along with the ability to obtain rare earth products with higher recovery efficiency, which can be built and deployed on a large scale, the study of the ability to decompose monazite concentrate from Vietnamese mineral ore sources by alkali under pressure conditions needs to be carried out.

Experimental part

Materials and equipments

Materials

The experiments were performed with Vietnamese monazite concentrate, took from *Ham Tan deposit* (Binh Thuan province, Vietnam). Initial monazite concentration in the ore is about 80 %. After the ore enrichment with magnetic separation and froth flotation concentration of monazite in the product was increased to 99%.

The monazite concentrate was crushed to different sizes before taking the experiments. The grinding process was carried out by ball mill equipment (Mini Roll Laboratory Ball Mill QM-5 (TENCAN, China)).

The composition of monazite concentrate (Ham tan deposit)

X-ray diffraction (XRD) analysis (Rigaku Model Ultma+) was carried out on the monazite concentrate to determine the major mineral phases present (Fig. 3). The program Highscore plus V4.5 with a database PDF-4 was used for analysis. It was found that the major component of concentrate is a monazite ([Ce, La, Nd, Th](PO_4)).

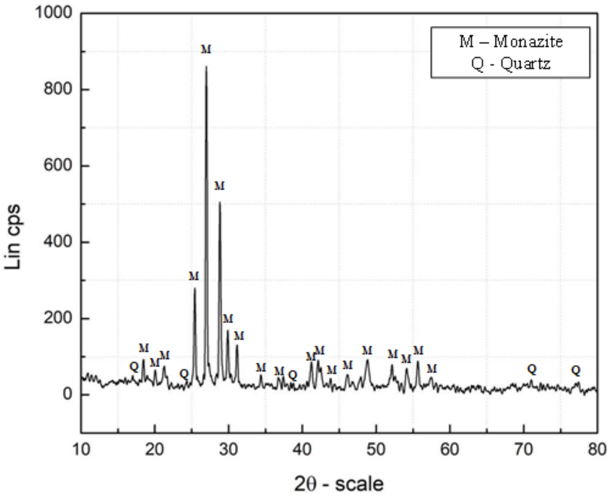

Fig. 3. *XRD imagine of Vietnamese monazite concentrate*

Рис. 3. *Рентгенограмма вьетнамского монацитового концентрата*

The chemical composition of the experimental monazite concentrate is shown in Table 2. The analysis was provided with optical emission spectroscopy with inductively coupled plasma (Horiba Ultima 2 ICP OES).

Table 2. *Composition of the experiment monazite concentrate*

Таблица 2. *Состав исследуемого монацитового концентрата*

Elements Элементы	Amount of elements in solid sample, mg/kg Содержание элемента в твердом образце, мг/кг	Mass concentration, % Массовая концентрация, %
Y	1067,27	1,07
La	11317,48	11,32
Nd	8017,93	8,02
Ce	21476,56	21,48
Pr	2166,9	2,17
U	329,33	0,33
Th	2946,88	2,95
Sm	1423,15	1,42
Gd	972,93	0,97
Dy	376,27	0,38

Thus, the main components in the experimental ore concentrate are Ce – 21,48%, La –11,32%, Nd>8%, Pr>2%, radioactive elements U>0,3% and Th approximately 3%.

Equipment

All decomposition experiments were carried out in an autoclave, a closed, heated and stirred reactor (Fig. 4).

Fig. 4. *Autoclave apparatus: 1 – furnace, 2 – control system, 3 – mixer, 4 – furnace lid with pressure meter*

Рис. 4. *Автоклав: 1 – печь, 2 – система управления, 3 – смесь, 4 – крышка печи с датчиком давления*

The apparatus was designed and manufactured at the Institute of Technology for Radioactive and Rare Materials (Vietnam). The apparatus includes four main parts:

- furnace: consists of a furnace shell containing heating wires, and a cylindrical tube made of chemically corrosive and heat-resistant material (stainless steel Inox310s). This is the space where the decomposition reaction took place;
- control system: sets the heating mode and stirring speed;
- mixer: includes motor and stirring blade whose rotation is controlled by the control system;
- furnace lid: has air release valve tubes, pressure meter and temperature sensor tube.

Materials used to manufacture reactor core and stirring blades: 310s stainless steel

Experiments

Investigations on reaction conditions were carried out and cross-selected. The main purpose was to improve the technological conditions currently being carried out at the Institute of Radioactive and Rare Earths Technology (Vietnam), where the decomposition with NaOH was carried out in the atmosphere. The main influencing parameters such as temperature, time, alkali/concentrate ratio by mass and particle size were tested and compared. Experiments were performed with 100 grams of monazite concentrate, mixed thoroughly with 70%-NaOH, and conducted in an autoclave (Fig. 3).

Investigation of decomposition temperature to decomposition efficiency

The decomposition temperature was investigated in the temperature range from 120 to 240°C with 100 grams of concentrate, mixed well with 70%-NaOH, A/M ratio (wt/wt)=1/1 and in a reaction time of 4 hours with a particle size d<48 μm (taken from the alkaline method under normal pressure conditions).

Investigation of time to decomposition efficiency

The process time was from 0,5 to 4 hours under the temperature conditions obtained from the investigation of the impact of decomposition temperature. The study was conducted with 100 grams of concentrate, mixed well with 70%-NaOH, A/M ratio (wt/wt)=1/1 with a particle size d<48 μm.

Investigation of the alkali/concentrate ratio (A/M) by mass (wt/wt) on the decomposition efficiency

Similarly, the A/M ratio (wt/wt) impact was investigated. The decomposition conditions such as temperature and time were taken from previous studies. The investigated ratios from 0,8/1 to 3/1 were carried out with 100 grams of concentrate with d<48 μm, the amount of NaOH was calculated according to the studied ratios.

Investigation of particle size on the decomposition efficiency

Particle size plays an important role for most chemical processes. Here, monazite concentrate was crushed to particle sizes d<48 μm, 48<d<55 μm, 55<d<63 μm, 63<d<75 μm, 75<d<106 μm. The researchs were conducted independently with 100 grams of concentrate, A/M ratio, time, temperature obtained from previous experiments. The research of parameters were performed multiple times to determine the best working conditions for the decomposition using monazite concentrate taken from Vietnamese placer.

Analysis methods

To determine the efficiency of the decomposition, the products obtained from the autoclave are washed with distilled water to remove Na_3PO_4 and NaOH. The washing was end when the pH of the washing water gained 7–8, then all Na_3PO_4 and NaOH were completely removed from the precipitate. The solution was filtered to collect the hydroxide precipitate. These hydroxides were leached with 3 M HCl, temperature 70°C, mixing time was 1 hour, stirring speed was set in 300 rpm. After leaching, filtering and washing to separate solid and liquid, the obtained solution was analyzed on ICP-MS to determine the decomposition efficiency according to:

$$\eta = \frac{m_i'}{m_i^o} \cdot 100\ \%,$$

where η is the decomposition efficiency, %; m'_i is the mass of element i after decomposition, gr; m^o_i is the mass of element i in concentrate, gr.

For calculating the degradation efficiency of the total REE and the total radioactive elements, the total efficiency is calculated as follows:

$$\eta_{TREE} = \frac{\sum m_i'}{\sum m_i^o} \cdot 100\ \%,$$

$$\eta_{TRaE} = \frac{\sum m_j'}{\sum m_j^o} \cdot 100\ \%,$$

where η_{TREE}, η_{TRaE} are the decomposition efficiency of TREE and radioactive elements, %; i is the REE; j is the radioactive element.

Results and disscusion

Reaction temperature effect on decomposition efficiency

Temperature plays an important role in most chemical processes. Normally, increasing temperature will increase the working speed, reduce the time required to carry out chemical changes, increase the driving force and working efficiency. Especially, with the working process in closed equipment such as autoclave, temperature will directly affect the working pressure. The particle size of concentrate was less than 48 µm. The pressure effect on temperature is shown in Fig. 5.

It could be seen that the temperature directly affects the pressure inside the autoclave used for decomposing monazite concentrate with alkali. Fig. 5 shows that the pressure increased slightly from 1,6 to 2 at when the temperature grew from 120 to 150°C. Then, the pressure inside the autoclave continued to increase more strongly to 7 at when the temperature increased to 210 °C and to 15 at when the temperature increased to 240°C, the pressure in the device reachesed over 15 at, and continued to increase more strongly when the temperature increased above 240°C. The cause of this phenomenon is the amount of water in the alkali used for decomposition. With 70% alkali, there will be 30% water leading to the phenomenon of water evaporation, forming superheated steam that increases the pressure in the device sharply. For the previous alkali method, monazite concentrate was decomposed with alkali at normal pressure, which means an open system. Therefore, there was no need to mention this pressure increase phenomenon.

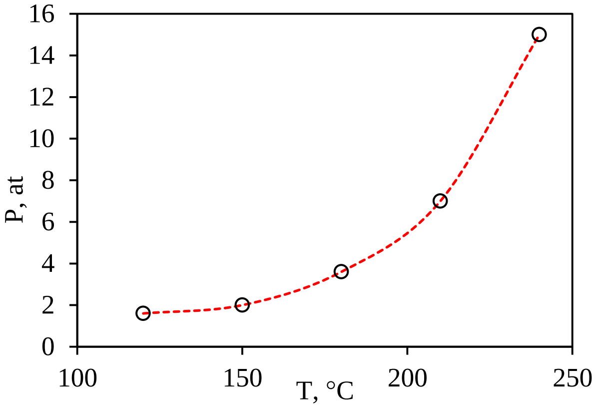

Fig. 5. *Pressure effect on reaction temperature in the autoclave*

Рис. 5. *Влияние давления на температуру реакции в автоклаве*

The results of the decomposition efficiency are shown in Table 3 and Fig. 6. Growth of the reaction temperature increased not only the reaction system pressure but also the decomposition efficiency. The yield of REE and U, Th was higher as well.

Table 3. *Dependence of decomposition efficiency of monazite concentrate on temperature*

Таблица 3. *Зависимость степени вскрытия монацитового концентрата от температуры*

T, °C	Decomposition efficiency/Степень вскрытия, η, %						
	Y	La	Nd	Ce	Pr	U	Th
120	91,02	84,45	72,99	88,99	84,30	43,59	65,97
150	93,69	95,34	87,69	91,18	96,86	45,57	80,23
180	94,79	99,15	89,72	93,91	97,55	49,63	79,74
210	88,32	98,86	88,24	94,18	93,70	53,44	69,74
240	86,17	96,82	86,46	92,46	90,46	54,34	51,32

It was found that, as the reaction temperature grew, the decomposition efficiency of REE increased. Especially from 120 to 150°C, then increased slightly to 180°C and then tended to decrease slightly.

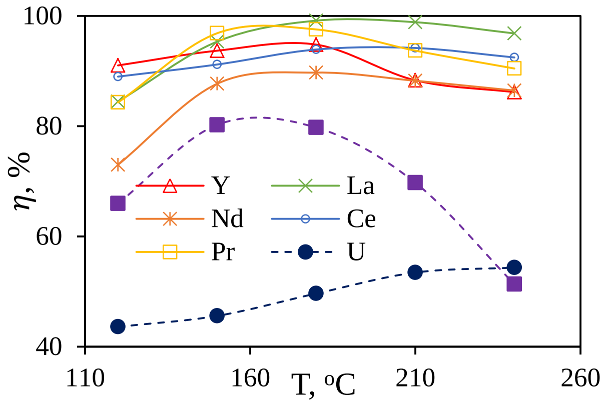

Fig. 6. *Temperature effect on the decomposition efficiency of REE, U and Th*

Рис. 6. *Влияние температуры на степень вскрытия РЗЭ, урана и тория*

The best efficiency for Y reached >93%, La>99%, Nd>89%, Ce>94% and Pr>97%. For U, the decomposition efficiency significantly increased at temperature growth till 210°C. After that, the decomposition efficiency of uranium did not change much. It should be mentioned that in case of uranium, hydroxides were formed from very limited decomposition reactions even at high temperatures. In the case of decomposition from phosphate ores uranium may form $UO_2(OH)_2$. However, in the case of concentrated alkali, there was a reciprocal transformation and the formation of $NaUO_2PO_4$ salt, which is a stable compound and almost insoluble in common acids. The results of analysis of residues or leaching with 3 M HCl containing a considerable amount of U elements have shown the suitability of this evaluation method. For Th, the efficiency increased at temperature growth from 120 to 150°C, then remained almost constant in the temperature range from 150 to 180°C, the highest efficiency reached is 80%. Continuing to increase the temperature, the process efficiency decreased sharply. The main reason here is that at temperature higher than 210°C, $Th(OH)_4$ decomposes partially into oxide form. In this case, the obtained thorium dioxide cannot be separated with HCl and stays in the solid part.

Fig. 7 shows the decomposition efficiency of the TREE and the total radioactive (U+Th) (TRaE). There was a clear separation in the decomposition efficiency as well as the recovery. The temperature increases, the decomposition efficiency of the TREE increases and does not change when the temperature came to 180°C and higher, with the total (U+Th) the decomposition efficiency decreased sharply at high temperature. The suitable temperature for decomposition will be in the range of 180–210°C. With the technologies put into actual production, the removal of radioactive impurities was important, the difference in the working path of TRRE and total radioactive (TRaE) could be seen, while TREE was less affected when T>180°C, TRaE tends to decreas gradually, especially at T=240 °C, the decomposition efficiency was only about 50%. This helps to choose the working conditions in the real activities. When compared with the alkaline method working under normal conditions, the decomposition efficiency of the TREE was much better. Under pressure effect the monazite particles were not only affected by stirring, but also had to endure the compressive forces of the generated steam. This makes the ability of the particles to contact with alkali better, thereby the reaction became better even though the time was shorter.

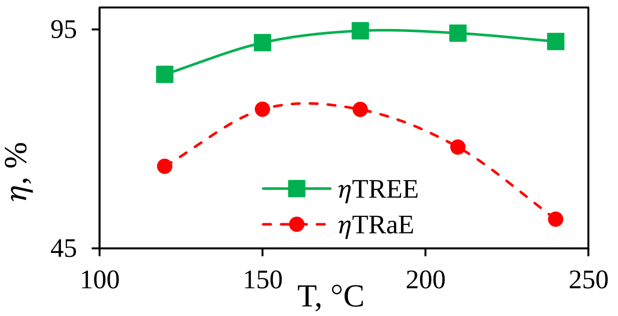

Fig. 7. *Temperature effect on the decomposition efficiency of TREE and radioactive elements*

Рис. 7. *Влияние температуры на степень вскрытия всех РЗЭ и радиоактивных элементов*

Reaction time effect on the decomposition efficiency

Besides the decomposition temperature, reaction time was an important parameter in the decomposition. The working time has many implications for the development of working regimes. To determine the appropriate working time, experiments were carried out with 100 grams of monazite concentrate, with a grain size of d<48 μm, with 100 grams of alkali (A/M ratio=1/1) at 180°C, selected from the temperature study. This was the best temperature for the total collection of rare earth and radioactive elements. The radioactive elements U and Th would be separated through selective leaching. The results are shown in Table 4 and Fig. 8.

The decomposition efficiency of rare earth and radioactive elements both increased with reaction time and reached equilibrium. The results showed that the decomposition efficiency increased sharply up to 1 working hour, then increased very slowly from 1 to 2 hours of decomposition. And remained almost unchanged thereafter. Therefore, the best time to decompose monazite concentrate here was 2 hours. The decomposition efficiency of Y reached >92%, La>98%, Nd>85%, Ce>96% and Pr>95%. For U, the best decomposition efficiency was achieved after 3 working hours, reaching >48% and Th had the best decomposition efficiency after 3 working hours, reaching 80%.

Table 4. *Dependence of decomposition efficiency of monazite concentrate on decomposition time*

Таблица 4. *Зависимость степени вскрытия монацитового концентрата от времени процесса*

Time, h/Время, ч	Decomposition efficiency/Степень вскрытия, η, %								
	Y	La	Nd	Ce	Pr	U	Th	ηTREE	ηTRaE
0,5	60,11	88,00	56,00	82,11	44,00	5,00	47,00	76,46	42,77
1	84,57	96,18	79,93	93,80	86,20	13,68	60,32	91,29	55,63
2	92,17	98,75	85,75	96,24	95,58	41,81	79,03	94,85	75,23
3	93,59	98,76	87,04	96,57	96,68	48,11	80,59	95,33	77,33
4	94,79	99,15	88,62	94,41	97,55	48,63	77,74	94,73	74,82

As the time continued to increase, the decomposition efficiency of Th gradually decreased due to the phenomenon of partial decomposition into ThO_2. However, the efficiency also increased insignificantly compared to after 2 hours of work.

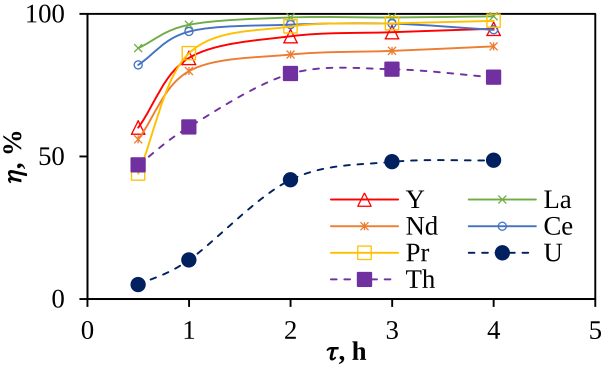

Fig. 8. *Reaction time effect on decomposition efficiency of REE, U and Th*

Рис. 8. *Влияние времени реакции на степень разложения РЗЭ, урана и тория*

The decomposition efficiency of TREE and TRaE (U+Th) over time is shown in Fig. 9. There was a clear separation in decomposition efficiency as well as recovery. After 2 hours of working, the recovery efficiency of REE and RaE is the best. However, when the working time was 1 hour, the decomposition efficiency of TREE reached >91 %, then increased slightly to 2 hours of working and reached >94%. For radioactive elements, after 1 hour of working, the decomposition efficiency was only about 55%, then increased sharply to >77% when increasing by 1 more hour of working. Therefore, to separate REE and RaE, the best working time was after 1 hour of working when still ensuring the decomposition efficiency, at the same time reducing a lot of Th and U in the product after decomposition. This was practical due to the need for the purity of the hydroxide after decomposition, but it caused many problems for waste management, due to the large content of Th and U in the waste after decomposition. Meanwhile, after decomposition, the hydroxides were selectively separated to obtain a rare earth solution and the radioactive elements were separated in the solid product (containing Th, U and a small part of REE). This solid waste can easily be processed through a solvent extraction scheme to separate Th, U out of rare earth products. Therefore, for the effect of decomposition time, 2 hours was the best selection.

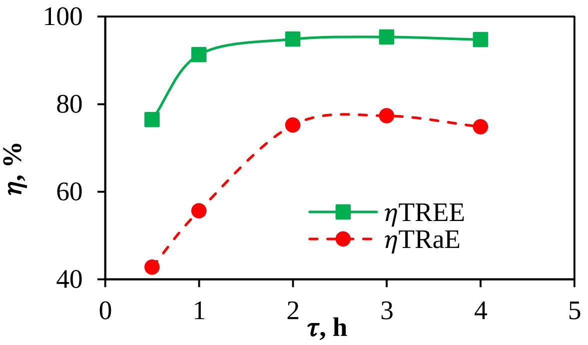

Fig. 9. *Reaction time effect on the decomposition efficiency of TREE and RaE*

Рис. 9. *Влияние времени реакции на степень вскрытия редкоземельных и радиоактивных элементов*

Effect of ratio of NaOH/concentrate by mass on the decomposition efficiency

Investigations on the ratio of alkali/concentrate by mass were carried out with different ratios from 0,8/1 to 3/1. The reactions were carried out with 100 g of concentrate with 70% alkali, according to the above ratios. The reaction temperature was 180°C with particle size d<48 µm, the investigation time was 2 hours. Decomposition experiments at longer times were of little significance as the degradation efficiencies obtained after 3 and 4 hours did not change much compared to 2 hours of operation. The results are shown in Table 5 and Fig. 10. The decomposition efficiency of REE and RaE slightly increased with the alkali/concentrate ratio. For the A/M ratio of 0,8/1 according to the theoretical calculation of molar equivalents, the alkali had an excess of about 40–50% of the amount of NaOH needed for complete decomposition compared to the theory if the decomposition efficiency was 100%, the decomposition efficiency of REE reached >90%, except Nd (>85%).

Table 5. *Dependence of decomposition efficiency of monazite concentrate on ratio between alkali/monazite concentrate by mass*

Таблица 5. *Зависимость степени вскрытия монацитового концентрата от массового соотношения щелочь/ монацитовый концентрат*

Ratio, wt/wt Массовое соотношение щелочь/монацитовый концентрат	Decomposition efficiency/Степень вскрытия, η, %								
	Y	La	Nd	Ce	Pr	U	Th	ηTREE	ηTRaE
0.8	91.88	93	85.91	90.66	90.10	48.99	74.65	90.40	72.07
1	92.17	98.75	85.75	96.24	95.58	41.81	79.03	94.74	73.12
1.5	95.05	99.23	92.87	94.27	97.95	50.09	77.47	95.67	74.72
2	95.92	99.32	94.26	94.86	99.04	50.59	78.31	96.14	75.52
2.5	96.04	99.22	94.75	95.03	99.66	50.79	78.66	96.44	75.85
3	96.29	99.30	95.27	95.43	99.68	51.09	79.47	96.65	76.62

Increasing the ratio to 1/1, the decomposition efficiency of the elements grew slightly to >92% for Y, Ce>96%, La>98%, Pr>95%, Nd still only reached >85%. When the A/M ratio continued to increase to 1,5/1, the decomposition efficiency of the elements grew slightly and reached stability. The efficiency of REE is high enough (Y>95%, La>99%, Ce>94% and Pr about 98%). Only Nd continued to increase at this ratio growth, reaching >95% at ratio of 3/1. For radioactive elements, the decomposition efficiency also tended to increase at the A/M ratio growth and stabilized at a ratio of 1,5/1. And the decomposition efficiency of U reached about 50% and that of Th reached >77%.

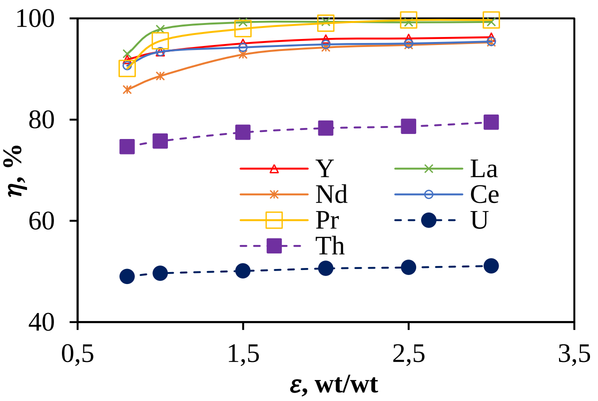

Fig. 10. *Effect of ratio between NaOH/concentrate by weight (wt/wt) on the decomposition efficiency of REE, U and Th*

Рис. 10. *Влияние соотношения NaOH/концентрат по массе на степень вскрытия РЗЭ, урана и тория*

The decomposition efficiency of TREE and TRaE (U+Th) according to the alkali/concentrate ratio (wt/wt) is shown in Fig. 11. According to the results obtained, it is found that the ratio between alkali and monazite concentrate by mass was best at 1,5/1. At that time, the decomposition efficiency of the TREE reached ~96%, decomposition efficiency of the RaE also reached ~75%, this efficiency was very high compared to other alkaline methods under normal conditions (without pressure). However, the biggest disadvantage of the alkaline method was the phenomenon of excess alkali. If the ratio is too large, combined with working under high temperature conditions, long reaction time, if SiO_2 from the ore could not be completely treated, it leads to the formation of silicate salts of Th and U. It is difficult to treat these two salts by conventional methods and to come to the sodium phosphate solution, contaminating this by-product. This is also the reason why the previous alkaline method was performed at 140°C, although the alkali/concentrate ratio by mass reached 4/1 with the desire to increase the decomposition efficiency to 90%. Comparing the results, it could be seen that although the A/M ratio was 1/1, the decomposition efficiency was only ~95%, 1% smaller than at the ratio of 1,5/1, but the amount of alkali used was much smaller. This had significance not only in production, but also in waste treatment and subsequent treatment of excess alkali. Therefore, the ratio between alkali and concentrate suitable for the fact meaning was 1/1.

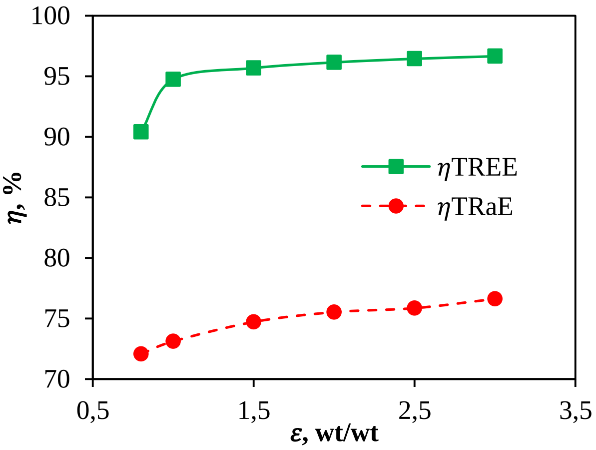

Fig. 11. *Effect of ratio between NaOH/concentrate by weight (wt/wt) on the decomposition efficiency of TREE and RaE*

Рис. 11. *Влияние соотношения NaOH/концентрат по массе на степень вскрытия РЗЭ и радиоактивных элементов*

Effect of concentrate particle size on the decomposition efficiency

Particle size plays a very important role in the monazite concentrate decomposition by alkali. There is a difference in the working mechanism of alkaline and acid methods. With the acid method, the working mechanism is adsorption, when the acid enters the ore particle and destroys ore structure from the inside. Therefore, large ore particles can still be decomposed, but it takes a long time. This is the advantage of the method. For the alkaline method, the decomposition mechanism comes from the surface of the ore particle, so if the ore particle is large, it causes a sharp decrease in efficiency. Previous studies shown that the particle size d<48 µm, in actual production, the particle size needs to be smaller than d<45 µm. The experiment conditions were selected and established according to previously conducted researches. The experiments were carried out at a decomposition temperature of 180°C, a decomposition time of 2 hours and a mass ratio of A/M of 1/1. The results of the investigation of the impact of particle sizes performed with d<48 µm, 48<d<55 µm, 55<d<63 µm, 63<d<75 µm, 75<d<106 µm are shown in Table 6 and Fig. 12. The decomposition efficiency of REE was guaranteed to particles with size d<55 µm, then the decomposition efficiency of elements at d<48 and 48<d<55 µm were quite similar. When the particle size continued to increase to d<63 µm, the decomposition efficiency of REE decreased, but basically reached 90%. For Ce and Pr elements, the decomposition efficiency still reaches >90% and the decomposition efficiency of Nd was almost similar when took with smaller particle sizes. For thorium the decomposition was effective up to the particle size d<63 µm with a decomposition efficiency of nearly 80%. Then it decreasesd sharply to about 65% when the particle size was 63<d<75 µm and to about 50% when the particle size reached d<106 µm.

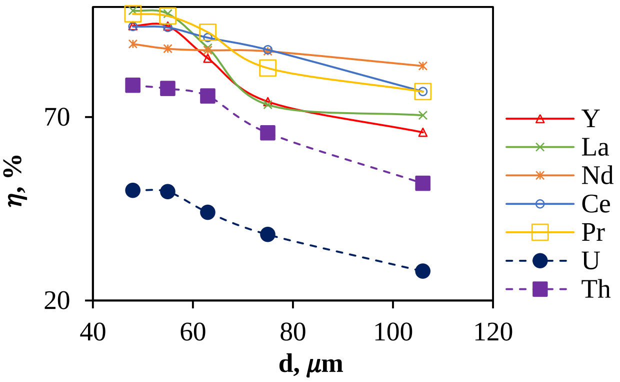

Fig. 12. *Particle size effect on the decomposition efficiency of REE, U and Th*

Рис. 12. *Влияние размера частиц на степень вскрытия РЗЭ, урана и тория*

For uranium the effective working size was at d<55 µm, then increasing the particle size caused the decomposition efficiency decrease sharply to 25% when the particle size was 5<d<106 µm.

Table 6. *Dependence of decomposition efficiency of monazite concentrate on particle size*

Таблица 6. *Зависимость степени вскрытия монацитового концентрата от размера частиц*

d, μm/мкм	Decomposition efficiency/Степень вскрытия, η, %								
	Y	La	Nd	Ce	Pr	U	Th	η_{TREE}	η_{TRaE}
48	94.88	99	89.91	94.66	98.1	49.99	78.65	95.09	75.77
55	94.79	98.15	88.62	94.41	97.55	49.63	77.74	94.48	74.92
63	86.00	88.86	88.20	91.63	93.11	44.01	75.70	90.23	72.51
75	74.18	73.34	87.93	88.34	83.33	37.95	65.67	83.82	62.88
106	65.82	70.47	83.91	76.95	76.94	27.95	51.92	76.28	49.51

To properly evaluate the process performance, it is necessary to determine the degradation efficiency f TREE, and TRaE (U and Th). The results are shown in Fig. 13 and Table 6.

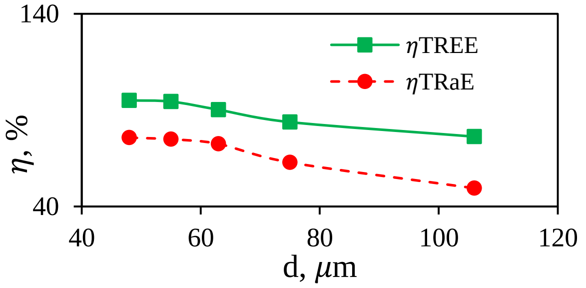

Fig. 13. *Particle size effect on the decomposition efficiency of TREE and RaE*

Рис. 13. *Влияние размера частиц на степень вскрытия РЗЭ и радиоактивных элементов*

The value of the decomposition efficiency of REE has a great effect on the selection of effective working particle size, as well as on the selection of grinding mode in actual production operation. For particles size of d<48 and d<55 µm, the decomposition efficiency of TREE is very high >94%. When increasing the particle size, the decomposition efficiency gradually decreases. Increasing the particle size to 63 µm, the efficiency was >90%, 83% when the particle size was 75 µm and 75% when the particle size increases to 106 µm. The total elements U and Th extraction also decrease from 75 to 49% when increasing the particle size. Therefore, it can be concluded that, for this method, particle size up to 55 µm can be decomposed very well under the given experimental conditions, working time 2 hours, alkali 70%, alkali/concentrate ratio by mass 1/1 and decomposition temperature 180°C. For particles with size of 63 μm, the decomposition efficiency can be increased by rising the alkali/concentrate ratio and working time. For larger particles, instead of changing the experimental conditions, it may be advisable to continue grinding the particles to a smaller size.

Therefore, when compared with the traditional alkaline technology performed under normal conditions, the decomposition under pressure conditions can be achieved with larger particles. Under the best working conditions of the previous method, which required 8 hours of decomposition at 140°C and an A/M ratio (wt/wt) of 1,4/1, the decomposition efficiency of REE was only about 70% when using particles with a size of d<48 μm. Meanwhile, with particles with a size of d<48 μm, this efficiency reached >94% under the following conditions: reaction temperature 180°C, A/M ratio 1/1 and decomposition time 2 hours.

Conclusion

Decomposition of Vietnamese monazite concentrate in autoclave obtained good results. The best decomposition time was 2 hours. The best alkali/concentrate ratio by mass was 1,5/1, however, the suitable ratio was 1/1 due to economic issues, waste associated with the technology. The suitable decomposition temperature was from 180 to 210°C. With decomposition temperature from 150 to 180°C, the decomposition efficiency was >90%, number of radioactive impurities was minimal but there was a problem with waste treatment when it contained about 10% of rare earth components and >25% of radioactive elements still in the undecomposed solid waste. For the particle size of the concentrate, with the conditions of time, temperature and alkali ratio obtained, the particle size works effectively with d<55 μm, when the total decomposition efficiency of rare earth elements reaches >94%. And it can be increased to 63 μm with the efficiency of >90%. The efficiency with particles of d<75 μm was >83%. It was possible to increase the ratio of alkali/concentrate (wt/wt) with particles of d>63 μm to increase the decomposition efficiency. However, it met problems with technology costs and waste treatment.

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