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ENRICHMENT ON BANGKA TIN SLAG'S TANTALUM AND NIOBIUM OXIDE CONTENTS THROUGH NON-FLUORIDE PROCESS

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В даному дослідженні вивчено вплив безфторидних розчинів, включаючи 8 М NaOH, 0,8, 1,6 і 2,4 М H_2SO_4 та 0,1, 0,4 і 0,8 М $HClO_4$, на збільшення вмісту оксиду танталу і ніобію в результаті вилуговування. Перед вилуговуванням олов'яний шлак Бангка (ОШБ) характеризувався за допомогою РФА. Потім шлак обжарювався при 900 °C, гартувався і зневоднювався. Далі ОШБ пройшов процес просіювання з розмірною класифікацією +100, –100 +150, –150 +200, –200 +250 і –250 меш. Після цього шлак –200 +250 меш витравлювали за допомогою 8 М NaOH. Потім вилужений продукт ділили на два, один з яких витравлювали з використанням 0,1, 0,4 і 0,8 М $HClO_4$, а решту – за допомогою 0,8 М $HClO_4$, а потім 0, 0,8, 1,6 та 2,4 М H_2SO_4 при 25 °C протягом двох годин. Для характеристики всіх залишків використовувався РФА, а для фільтратів – ААС, а також ІСП-ОЕС. Дане дослідження обумовлене дефіцитом танталу і його статусом як одного з найважливіших технологічних елементів. На додаток, більшість попередніх досліджень дозволили збільшити вміст оксиду танталу і ніобію завдяки використанню фторидної кислоти, в той час як в даному дослідженні розглянуті тільки безфторидні розчини. Результати показують, що перхлоратна кислота з подальшим вилуговуванням сірчаною кислотою злегка підвищує вміст танталу і ніобію. Однак цей метод є найбільш ефективним серед NaOH, $HCIO_4$ і $HCIO_4$ з подальшим вилуговуванням за допомогою H_2SO_4. Даний висновок є формою наукових зусиль з підтримки наявності танталу за рахунок використання непотрібних відходів олов'яної плавки

Ключові слова: вилуговування, оксид ніобію танталу (ОНТ), олов'яний шлак Бангка, NaOH, $HClO_4$

1. Introduction

The electronic, automotive, and aerospace new technology's high dependence on tantalum makes this metal one of the technology-critical elements [1]. Researches on the endurance of tantalum supply chain investigated the endurance improvement mechanism such as optimization of other sources (e. g. tin slag, scraps, etc.), recycling, material substitution, and hoarding [2]. Efforts to widen the knowledge of tantalum around environment were done especially that which merges with sea and freshwater [1]. Investigations on material and future sources revealed that the source of tantalum from tin slag was 13 % among all supplies [3] and a forecast in 2013 implied a deficit in 2015 and 2016 [4]. One of the measures to obtain the secondary sources of valuable elements is the metallurgical slag and several explorations presented the acquisition of valuable elements from metallurgical slag [5, 6]. Hydrometallurgy was used by prior researches to upgrade the concentration of elements [7, 8].

The aforementioned explanation of the tantalum scarcity and researchers' effort to keep the tantalum supply chain, to venture materials that, one never thinks before, contain this metal such as sea and freshwater, and to find the best technique to maximally pull this valuable element out of the unwanted matters such as hydrometallurgy draw researchers' attention to enrich the contents of tantalum.

2. Literature review and problem statement

Some information above becomes a challenge for the researchers to carry out the next research in order to raise the tantalum supply. In general, as is common in the case with tantalum, tantalum extracted from tin slag is followed by niobium [9]. Tin slag as a secondary source of tantalum and niobium was reported in prior researches [9, 10]. Tin slag primarily spreads across Nigeria [10], Zaire [20], Brasil [12], Thailand [13] and Indonesia [9]. Studies of tin slag leaching with acid and alkaline solution to upgrade the concentration of tantalum and niobium have been done in previous researches [14, 15].

This paragraph summarizes prior studies which exploited fluoride acid to enrich TNO contents from tin slag. The dissolution of HCl, HF, and NaOH in tin slag which involved either alkali-acid order reagents or acid-alkali order reagents increased tantalum recovery rate, from 60 % to 93 %, and niobium recovery rate, from 29 % to 78 % [20]. HF or ($HF+H_2SO_4$) dissolution in tin slag's $(Ta+Nb)_2O_5$ whose contents were over 25 % produced around 85 % recovery rate [21]. Tin slag's 3.4 % Nb_2O_5 and 3.05 % Ta_2O_5 underwent several processes of leaching, namely caustic solution leaching, alkali pugging, alkali fusion, acid leaching, and HF & H_2SO_4 leaching. The alkali processes and acid leaching optimally produced Ta_2O_5 and Nb_2O_5, 10.4 % and 10.6 % [14]. Among 4 %, 8 %, 16 %, and 32 % concentrations, dissolving 8 % HF in tin slag produced an optimal yield ratio of tantalum and niobium, 2.01 and 2.09 [15].

Chlorination was also used to enrich tin slag's TNO concentration. The summary of related investigations is provided in this paragraph. Tin slag's 7.5 % tantalum and 5.2 % niobium underwent HCl leaching (called low-grade composition (LGC)). Meanwhile, the other was HCl-leached followed by NaOH leaching (called high-grade composition (HGC)). Next, both LGC and HGC were chlorinated and added with Cl_2+N_2 or Cl_2+CO+N_2 in 200–1,000 °C. On the one hand, LGC in 1,000°C within 24 hours extracted more than 95 % of tantalum and niobium. On the other hand, at the same temperature and time, HGC chlorination produced 65 % tantalum and 84 % niobium [22]. Recovery refractory metal applied to tin slag which engaged HCl leaching and chlorination roasting optimally produced 82.1 % Nb_2O_5 and 84.7 % Ta_2O_5 [19]. HCl and NaOH dissolution in tin slag containing 0.33 % Ta_2O_5 and 0.64 % Nb_2O_5 optimally upgraded tantalum and niobium concentration to 0.52 % and 1.18 % [15].

In this study, the tin slag which was used is from Bangka Islands. The tin slag will be further referred to as Bangka tin slag (BTS). Prior investigations that used BTS informed: BTS contained 2.7 % $(Ta,Nb)_2O_5$ [11], the study upgraded BTS' TNO contents [9, 19], the thermodynamic analysis was done in the study of upgrading BTS' REE [20].

3. The aim and objectives of the study

This research aims at enhancing the concentration of Bangka tin slag's tantalum and niobium through leaching processes using non-fluoride chemical substances.

To achieve this goal, the following objectives were set:

1. Characterizing BTS through XRF.
2. 900 °C-roasting, quenching, and dewatering BTS.
3. Leaching –200+250 mesh BTS by 8M NaOH and dividing the residues into two groups.
4. Leaching group one with 0.1, 0.4, and 0.8 M $HClO_4$ and leaching group two with 0.8 M $HClO_4$ followed by 0, 0.8, 1.6, and 2.4 M H_2SO_4 at 25 °C within 2 hours.

4. Materials and methods for enrichment on Bangka tin slag's tantalum and niobium oxide contents through the non-fluoride process

4. 1. Materials and apparatus used in the experiment

The tin slag was taken from a tin smelter in Bangka Belitung Islands in Indonesia. The investigation exploited sodium hydroxide (technical solution), perchloric acid (p.a), and sulfuric acid (p.a). These chemical elements are comprised of (a) 8M NaOH, (b) 0.8, 1.6 and 2.4 M H_2SO_4, and (c) 0.1, 0.4 and 0.8 M $HClO_4$.

The researchers used a ball mill (Toptek Topvert G1), a sieve, a Barnstead Thermolyne furnace, a ceramic container, a magnetic stirrer (Thermo Scientific CIMAREC), Scanning Electron Microscopy (SEM), X-Ray Fluorescence (Bruker handheld XRF analyzer), Atomic Absorption Spectroscopy (AAS – PerkinElmer Analyst 400), and Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES PerkinElmer Optima 8300).

4. 2. Procedure for conducting the experiments and determining the indicators samples' properties

Below is the layout of research stages which tantalum and niobium underwent during investigation. The details are systematically given under Fig. 1.

The first characterization of BTS was SEM and XRF. In the first process, BTS was roasted at 900 °C, quenched, and dewatered. Next, the results of these processes were characterized using XRF and SEM. After being dewatered, BTS was sieved with size distribution classifications of +100, –100+150, –150+200, –200+250, and –250 mesh. The sizes which were used in the next leaching were –200+250 mesh.

The first leaching exploited NaOH 8M. Then, the residues were divided into 2. The first one was $HClO_4$-leached with 0.1, 0.4, and 0.8 M concentrations while the other underwent 0.8 M $HClO_4$ and 0, 0.8, 1.6, and 2.4 M H_2SO_4 leaching. All the leaching processes were carried out at 25 °C within 2 hours. All residues were characterized using XRF. Meanwhile, the characterization of filtrates from iron and calcium elements involved AAS, and those from niobium and tantalum elements were characterized through ICP-OES. Fig. 1 illustrates all the research schemes.

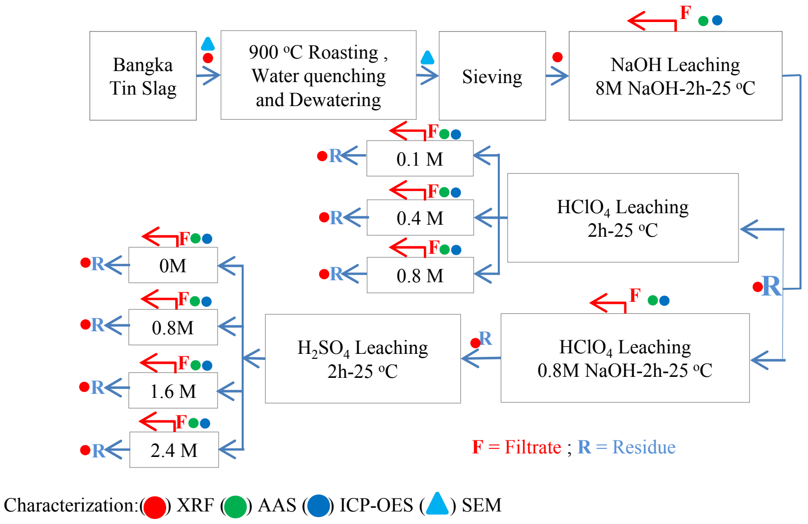


Fig. 1. Research scheme which tantalum and niobium underwent during investigation

5. Results of enrichment on Bangka tin slag's tantalum and niobium oxide contents through the non-fluoride process

The sub-chapters of the research results are as follows: (1) XRF and SEM characterization, (2) BTS 900 °C-roasting, quenching, dewatering, and sieving, (3) NaOH leaching, and (4) $HClO_4$ leaching and $HClO_4$ followed by H_2SO_4 leaching.

5. 1. XRF and SEM Characterization

The first BTS characterization was conducted using XRF as shown in Table 1 and SEM as illustrated in Fig. 2, *a*. All the elements of BTS were split into 3 parts: the valuable oxides are tantalum-niobium; major other oxides (MOO); elements and minor other oxides (EMO). Tantalum and niobium are the two metals whose contents would be upgraded. Major other oxides (MOO) are the high-concentration of other oxides including SiO_2, CaO, TiO_2, Al_2O_3, Fe_2O_3, Sn, and Zr. Elements and minor other oxides (EMO) are the chemical elements not categorized as tantalum-niobium and MOO. Below are XRF characterization results. Table 1 shows the three highest MOO elements, namely SiO_2, TiO_2, Fe_2O_3, and EMO (83.15 %).

Table 1

The results of Bangka tin slag's XRF characterization

Nb (%)	Ta (%)	SiO_2 (%)	TiO_2 (%)	Fe_2O_3 (%)	Sn (%)	Zr (%)	CaO (%)	Al_2O_3 (%)	EMO (%)
0.47	0.23	6.56	2.38	2.36	1.78	1.33	1.22	0.52	83.15

5. 2. BTS 900 °C Roasting, Quenching, Dewatering, and Sieving

The results of BTS 900 °C roasting, quenching, dewatering, and sieving (RQDS) are revealed in Fig. 2. +100 and –100+150 mesh BTS samples represent the three dominant contents of oxides, namely SiO_2, TiO_2, and Fe_2O_3. On the other hand, in sizes –150+200, –200+250, and –250 mesh, SiO_2, Sn, and Fe_2O_3 become the three dominant contents of oxides.

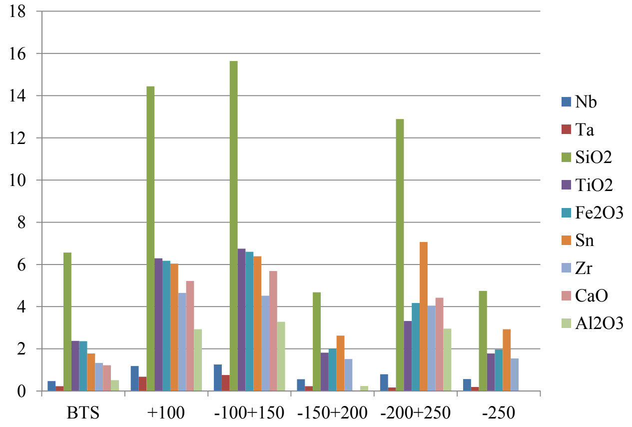


Fig. 2. The contents of oxides and elements in various grain sizes. *Note: in the X-axis, the unit of size is mesh*

Roasting, quenching, dewatering, and sieving on BTS left some physical distinctions. The differences in appearance between pure BTS and roasted, water-quenched, and dewatered BTS are shown below.

a *b*

Fig. 3. The SEM-EDS characterization at 100 times magnification: *a* – Bangka tin slag; *b* – Bangka tin slag after being roasted, water quenched, and dewatered

The SEM-EDS characterization which had been given a magnification of 100 times of BTS and BTS which had been 900 °C-roasted, quenched, and dewatered (called as BTS-RQD) are in Fig. 3. BTS-RQD has a bigger grain size and a more open touch surface area.

5. 3. NaOH Leaching

The roasted, quenched, dewatered, and –200+250 mesh-sieved sample was 8M NaOH-leached at 25 °C within 2 hours. The residues and filtrates characterization results are presented in Table 2. The results reveal an increase in niobium and MOO, and a decrease in tantalum and EMO. NaOH-leached filtrates have a small amount of niobium, 0.205 ppm, and tantalum, <0.1 ppm. In other words, the concentration of both metals is very low.

Table 2

After-NaOH leaching XRF characterization of Bangka tin slag's residues and filtrates

Nb	Ta	SiO_2	TiO_2	Fe_2O_3	Sn	Zr	CaO	Al_2O_3	EMO
Residue (in %)									
0.72	0.16	11.73	3.23	3.99	6.49	4.07	4.36	2.61	62.64
Filtrate (in ppm)									
0.205	<0.1	not. available	not. available	not. available	not. available	not. available	not. available	not. available	not. available

5. 4. $HClO_4$ Leaching and $HClO_4$ Followed by H_2SO_4 Leaching

The next step is the observation of NaOH leaching results. In this stage, the residues were split into two parts. The first part was 0.1 M, 0.4 M, and 0.8 M $HClO_4$-leached while the second one was leached two times. The first leaching engaged 0.8 M $HClO_4$ while the other leaching exploited 0, 8, 16, and 24 M H_2SO_4. Then, all the leaching results were XRF-characterized. Fig. 4 shows further information.

Both 0.8M $HClO_4$ leaching and 0.8M $HClO_4$ & 0 M H_2SO_4 leaching produced near results (see the red arrows). The 0.8M $HClO_4$ leaching reveals 1.28 % tantalum and 0.79 % niobium whereas 0.8M $HClO_4$ and 0 M H_2SO_4 leaching represents tantalum and niobium, with 1.27 % and 0.71 %, respectively.

On the one hand, Fig. 4, *a* provides the results of $HClO_4$ leaching which show an increase in tantalum and niobium contents higher than those not processed (as in pure BTS) and those undergoing RQDS and NaOH leaching. The maximum rise in the amount of niobium and tantalum contents through $HClO_4$ leaching is 1.28 % and 0.79 % respectively. On the other hand, Fig. 4, *b* provides the results of $HClO_4$ and H_2SO_4 leaching with their concentration variations. The results show a rise in niobium and tantalum contents higher than those undergoing only $HClO_4$ leaching. The highest rise in the amount reaches 1.57 % for niobium and 0.94 % for tantalum.

5. 4. 1. MOO Leached with $HClO_4$ and $HClO_4$ Followed by H_2SO_4

The contents of MOO leached with only $HClO_4$ and both 0.8M $HClO_4$ and H_2SO_4 are provided in Fig. 5. In Fig. 5, *a*, $HClO_4$ leaching with its concentration variations shows a dominant contents decrease and the start of this decrease is 0.1M $HClO_4$. On the other hand, TiO_2 and Zr are the two oxides whose contents rise from 0.1M to 0.4M.

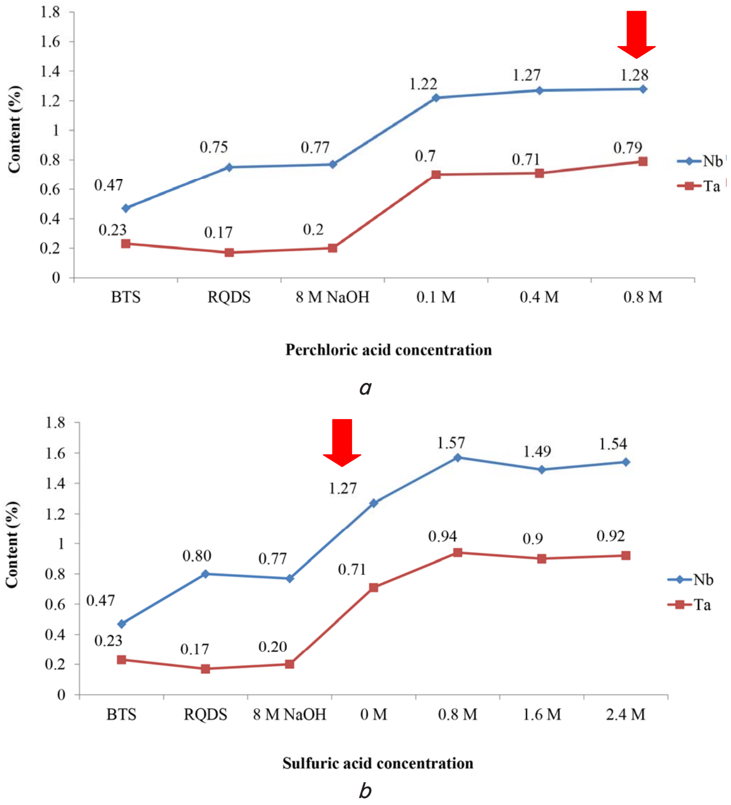
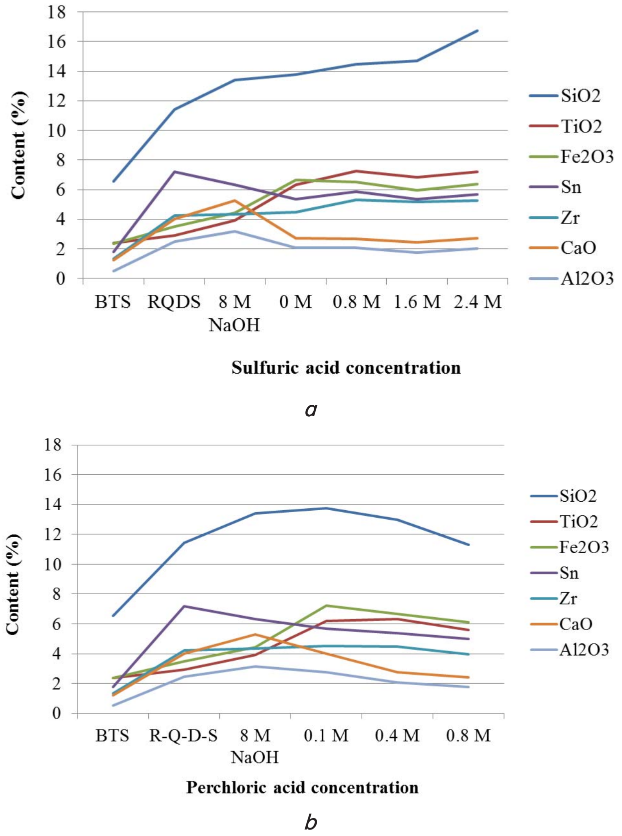


Fig. 4. The residue results of tantalum and niobium's leaching: *a* – BTS & BTS roasted, water quenched, dewatered, –200+250 mesh-sieved, and 8M NaOH as well as $HClO_4$-leached with its concentration variations; *b* – BTS & BTS roasted, quenched, dewatered, –200+250 mesh-sieved, and 8M NaOH, 0.8M $HClO_4$, and H_2SO_4-leached with its concentration variations

What 0.8M $HClO_4$ and H_2SO_4 leaching with its concentration variations produced can be seen in Fig. 5, *b*. SiO_2 shows a rise in all concentration variations while a fall is observed in the contents of TiO_2, Fe_2O_3, Sn, Zr, CaO, and Al_2O_3 which underwent from 0.8M to 1.6M H_2SO_4 leaching. All MOO elements went up from 1.6M to 2.4M H_2SO_4.



a

b

Fig. 5. The results of MOO's residues leaching: *a* — BTS & BTS roasted, water quenched, dewatered, −200+250 mesh-sieved, and 8M NaOH as well as $HClO_4$-leached with its concentration variations; *b* — BTS & BTS roasted, quenched, dewatered, −200+250 mesh-sieved, and 8M NaOH, 0.8M $HClO_4$, and H_2SO_4-leached

$HClO_4$ leaches and 0.8M $HClO_4$ and H_2SO_4 leaching with its concentration variations show that all concentration variations produce a small amount of niobium and tantalum. Table 3 will give further information.

Table 3

The results of BTS leaching with 8M NaOH, $HClO_4$ with its concentration variations, 0.8M $HClO_4$, and H_2SO_4 with its concentration variations

Element	8M NaOH	$HClO_4$ concentration variation			0.8M $HClO_4$+H_2SO_4 concentration variation			
		0.1M	0.4M	0.8M	0 M	0.8M	1.6M	2.4M
	(ppm)	(ppm)			(ppm)			
Nb*	0.205	0.208	0.207	0.206	0.206	0.364	0.450	0.222
Ta*	n.a	n.a	n.a	<0.1	<0.1	0.516	0.537	0.011
Fe**	2	82.33	626.9	1,281	1,278	1,821	2,356	2,942
Ca**	n.a	3,466	3,874	4,672	4,675	5,123	2,011	3,824

*Note: Characterized by * – ICP-OES;** – AAS*

The results of all leaching processes together with their chemicals' concentration variations in the above table imply that the non-fluoride solutions produce a small rise in the niobium and tantalum contents.

5. Discussion of results of enrichment on Bangka tin slag's tantalum and niobium oxide contents

The dominant oxide compounds produced ceramic structures in tin slag. The fragility of these structures was caused by the expansion of cracks in the materials before tin slag deformed. Roasting and water quenching on tin slag functioned to increase the number of pores. The rise in pores enlarged other oxides wetting areas. The enlargement of these zones helped accelerate the leaching of other oxides which would be dissolved.

The following is a description of the roasting and water quenching process: a – tin slag has a three-dimensional porous structure where a particle stone house, free other oxide particles, and both fully and partially-locked other oxides particles lie on its surface and in its inside. In other oxide particles, either fully or partially locked, there is a direct contact area next becoming the wetting zone when given a solvent; b – roasting made the particle stone house more porous and also expanded the wetting areas; c – the mismatch of heat expansion in the multiphase materials during water quenching process suddenly produced spontaneous micro-cracks; d – the thermal cracks eventually led to fractures, size reduction, and the increase in the porous and surface areas. Fig. 6 illustrates the occurrence of porous surface change, thermal cracking, fractures, and size reduction.

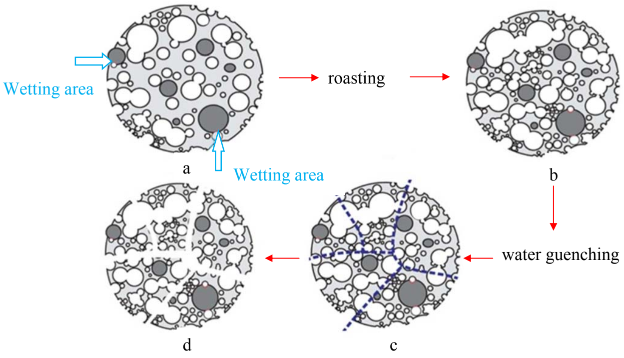


Fig. 6. Occurrence of porous surface change: a — Bangka tin slag where there is valuable oxide with a small wetting area; b — Porous surface change; c — thermal cracking and fracture condition; d — particle size reduction

By extending the porous areas, (1) the particle wetting surface widened, (2) the process of thermal cracking, fractures, and size reduction succeeded, and (3) so did the wetting process of fully and partially-locked other oxides surface areas. The results of SEM-EDS characterization at 100 times magnification of BTS and BTS RQD are in Fig. 3.

The roasting and water quenching effects on tin slag did not result in the oxide elements compounding (Fig. 7). In that figure, there is no intersection of the Gibbs free energy equation (ΔG) with the temperature variables. Meanwhile, the results of XRF characterization of several-sizes samples show that SiO_2 has the dominant contents (Fig. 2).

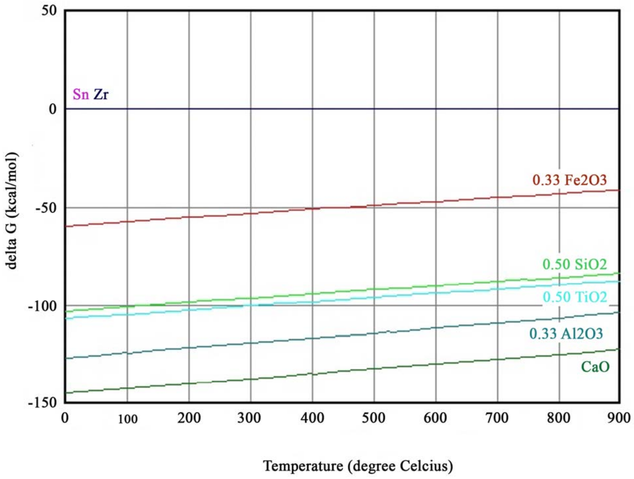


Fig. 7. The Ellingham diagram of MOO up to 900 °C

In Table 4, the value of $\Delta G_{25^o}<0$ implies that the NaOH solution can dissolve Nb_2O_5, Ta_2O_5, and MOO at 25 °C. On the other hand, the decrease in EMO contents from 83.15 % to 58.16 % (Fig. 12) shows that the percentage of EMO solubility in NaOH solution is much greater than that of MOO solubility. Meanwhile, the greater EMO solubility can raise the MOO contents from 16.15 % to 40.87 % (Table 1, 2).

Table 4

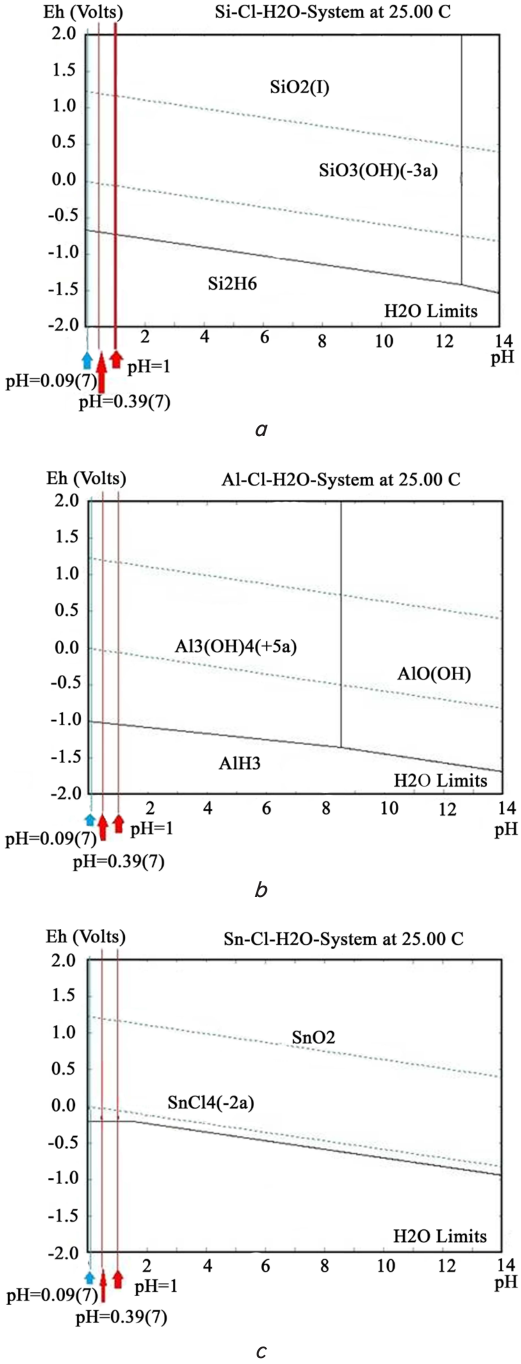
ΔG of Nb_2O_5, Ta_2O_5, and MOO solution in NaOH

No.	Reaction Equation	ΔG_{25^o} (kcal/mol)
1	Nb_2O_5+2NaOH=2$NaNbO_3$+H_2O	–30.400
2	Ta_2O_5+2NaOH=2$NaTaO_3$+H_2O	–45.235
3	SiO_2+2NaOH=Na_2SiO_3+H_2O	–21.228
4	6TiO_2+2NaOH=$Na_2Ti_6O_{13}$+H_2O	–23.582
5	Fe_2O_3+2NaOH=2$NaFeO_2$+H_2O	–4.262
6	SnO_2+2NaOH=Na_2SnO_3+H_2O	Not found in database
7	ZrO_2+2NaOH=Na_2ZrO_3+H_2O	–5.954
8	Al_2O_3+2NaOH=2$NaAlO_2$+H_2O	–8.165

Fig. 5, *a* shows the results of $HClO_4$ leaching. In this figure, the contents of SiO_2, Fe_2O_3, Sn, CaO, and Al_2O_3 decrease as the concentration of the leaching solution increases. The thermodynamic analysis of $HClO_4$ dissolution is in Fig. 8. In Fig. 8, the Pourbaix diagrams show silica, alumina, and tin oxides forming elemental ions at pH=1 ($HClO_4$=0.1M), pH=0.397 ($HClO_4$=0.4M), and pH=0.097 ($HClO_4$=0.8M). After the filtrate contents were $HClO_4$-leached, the contents of iron and calcium oxide reduced as shown in Table 3.

Compared to the MOO contents in BTS, those which were $HClO_4$-leached rise (Fig. 5, *a*, Table 1, 2). This increase occurs particularly in SiO_2 from 6.56 % to 11.73 % (Table 1). The rise in MOO contents occurs when EMO solubility is much greater than that of MOO.

Fig. 9 shows the thermodynamic analysis of $HClO_4$ leaching results in EMO. The Pourbaix diagrams illustrate the examples of cerium and yttrium oxides and the formation of cerium and yttrium ions at pH=1 ($HClO_4$=0.1M), pH=0.397 ($HClO_4$=0.4M), and pH=0.097 ($HClO_4$=0.8M). Both ions also exhibit cerium and yttrium oxides dissolved in HCO_4.



a

b

c

Fig. 8. The Poubaix diagrams of:
a — silica; *b* — aluminum; *c* — tin oxides

The 0.8 M $HClO_4$ leaching followed by H_2SO_4 and its concentration variations resulted in an optimum TNO content, 2.51 %. The calculation result of EMO contents after being AAS-characterized and both $HClO_4$ and H_2SO_4-leached shows a reduction (Fig. 12, *b*) and the iron and calcium solubility in filtrates (Table 3).

The cerium and yttrium ions on the Pourbaix diagrams (Fig. 11) show the solubility of cerium and yttrium oxides in H_2SO_4 solution between pH–1 and 0.

The 0.8 M $HClO_4$ leaching followed by H_2SO_4 and its concentration variations reduced the EMO contents (Fig. 12, *b*). In addition, the contents of leached EMO also have a smaller percentage than those in BTS.

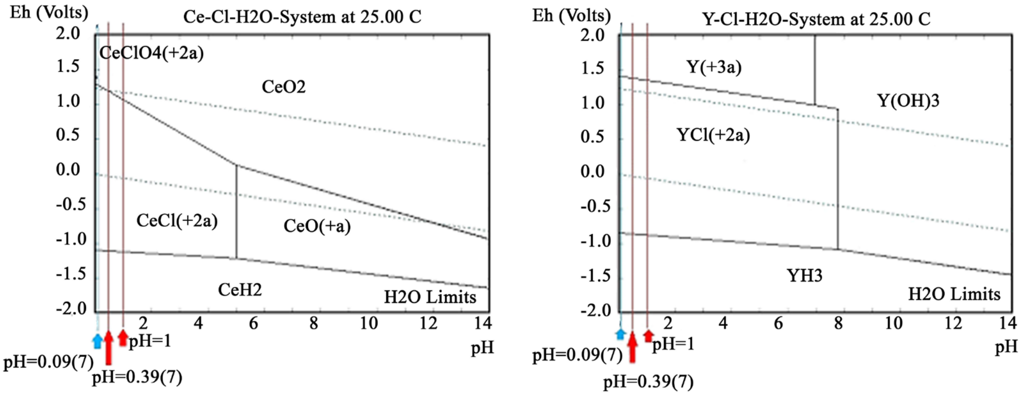
The upgrading of tantalum and niobium in BTS through double leachings and leaching duration variation, especially with HCl (20 minutes in the initial leaching and 50 minutes in the subsequent leaching), shows an increase in TNO (Ta_2O_5+Nb_2O_5) contents from 1.95 % (0.8 % Ta_2O_5+0.15 % Nb_2O_5) to 2.67 % (1.56 % Ta_2O_5+1.11 % Nb_2O_5) [15]. The leaching duration variation in the (second) leaching can be considered for further studies of enhancing Ta_2O_5 and Nb_2O_5 contents.

Table 5

ΔG of MOO solution in H_2SO_4

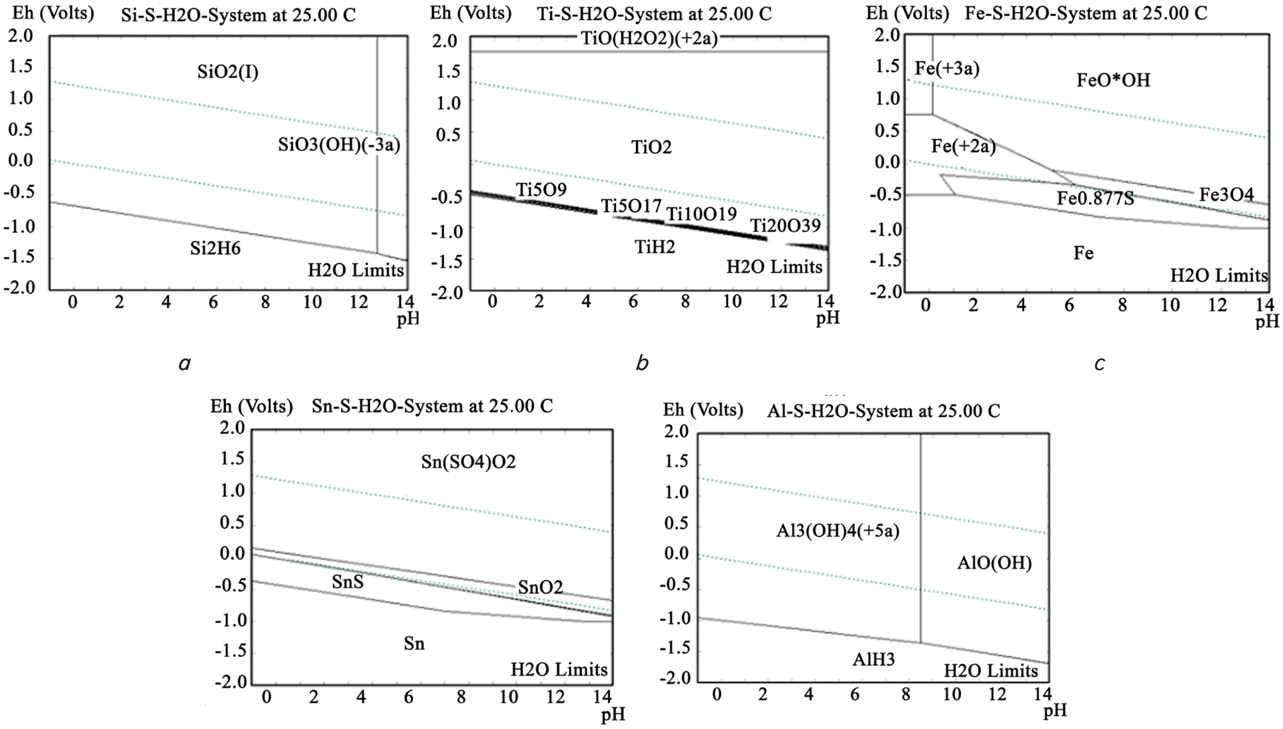
No.	Reaction Equation	ΔG_{25^o} (kcal/mol)
1	$SiO_2+H_2SO_4$	
2	$TiO_2+H_2SO_4=TiOSO_4+H_2O$	Not found in database
3	$Fe_2O_3+3H_2SO_4=Fe_2(SO_4)_3+3H_2O$	–39.437
4	$SnO_2+2H_2SO_4=Sn(SO4)_2+2H_2O$	2.726
5	$ZrO_2+2H_2SO_4=Zr(SO4)_2+2H_2O$	–14.254
6	$Al_2O_3+3H_2SO_4=Al_2(SO_4)_3+3H_2O$	–37.994

Even though the researchers here are successful to have slightly increased the contents of both tantalum and niobium, their study still has some drawbacks.



a *b*

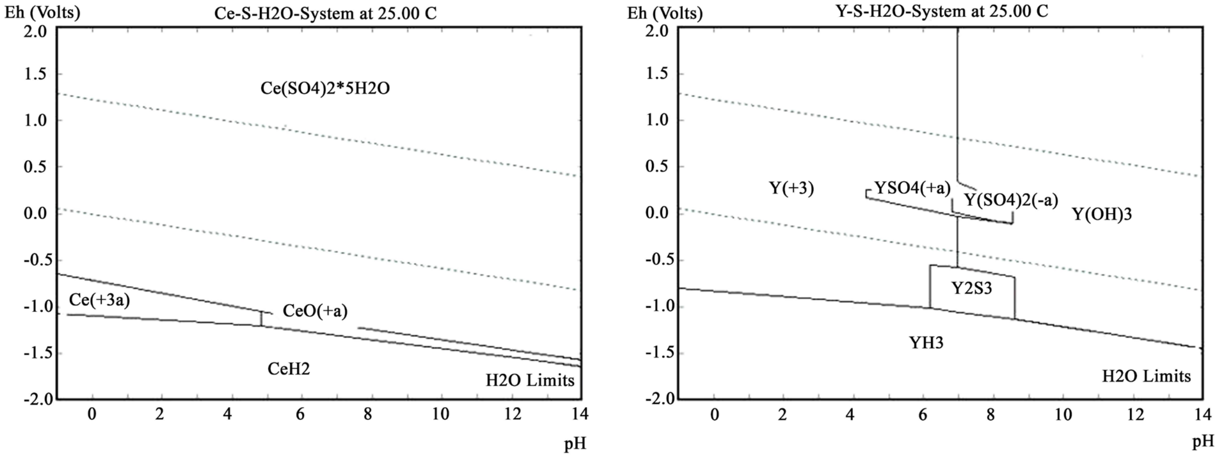
Fig. 9. The Poubaix diagrams of: *a* — cerium; *b* — yttrium oxides



a *b* *c*

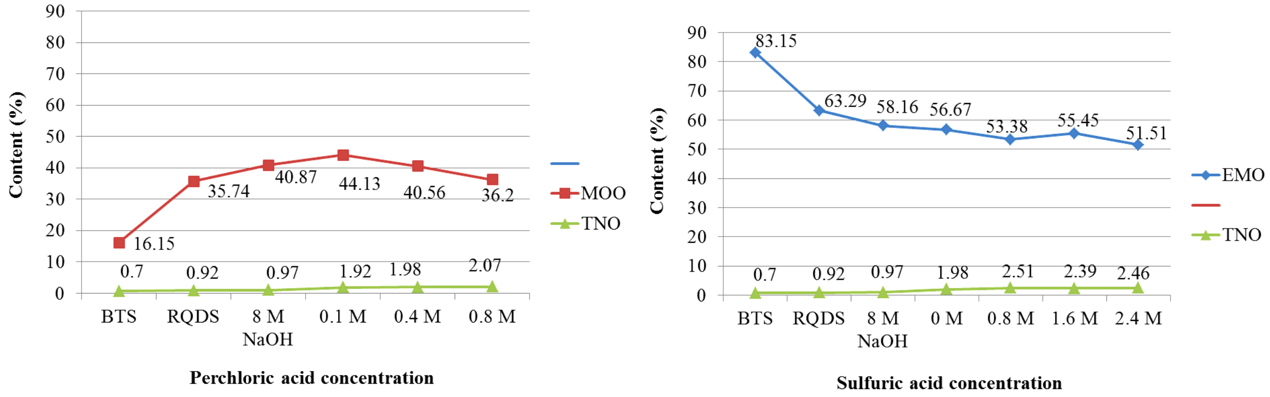
d *e*

Fig. 10. The Pourbaix diagrams of: *a* — silica; *b* — titanium; *c* — iron; *d* — tin; *e* —aluminum oxides



a　　*b*

Fig. 11. The Pourbaix diagrams of: *a* — cerium; *b* — yttrium oxides



a　　*b*

Fig. 12. The residues of leached TNO, MOO, and EMO:
a — The $HClO_4$ and its concentration variations leaching decreases the MOO contents,
b — The 0.8M $HClO_4$ leaching followed by H_2SO_4 and its concentration variations decreases the EMO contents

First, this study exploits high acid chemical solutions. Therefore, the filtrates as a leaching product are toxic. Secondly, this investigation involves tin slag only from Bangka Island, Indonesia. In fact, samples from various countries will produce varied and comparable results so that tantalum and niobium from the concerned regions' tin slag can be exploited as well as possible for human welfare.

For the development of further studies, other researchers are advised to use organic chemical solutions such as phytic acid so that the resulted filtrates are not toxic. On the other hand, increasing the tantalum and niobium contents through this method has a serious consequence as follows. The dissolution of elements and minor other oxides (EMO) raises the contents of Ta, Nb, and EMO itself. Increasing the contents of EMO will raise the contents of uranium (U) and thorium (Th) in EMO, leading to a rise in the effect of radiation on BTS.

6. Conclusions

1. XRF characterization reveals the three highest MOO elements that include SiO_2, TiO_2, Fe_2O_3, and EMO (83.15 %) while that of SEM shows that BTS-RQD has a bigger grain size and a more open touch surface area.

2. The 900 °C-roasting, quenching, dewatering, and sieving of BTS results in the three dominant contents of oxides, SiO_2, TiO_2, and Fe_2O_3.

3. 8M NaOH leaching shows a rise in niobium and MOO, and a reduction in tantalum and EMO.

4. $HClO_4$ leaching produces an increase in the contents of tantalum and niobium higher than those of pure BTS and those undergoing RQDS and NaOH leaching. On the other hand, $HClO_4$ followed by H_2SO_4 leaching produces an increase in niobium and tantalum contents higher than those leached with only $HClO_4$.

All the above investigations imply that the rise in both tantalum and niobium is relatively low.

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