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Other Oxides Pre-removed from Bangka Tin Slag to Produce a High Grade Tantalum and Niobium Oxides Concentrate: An Advance Study

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ABSTRACT

Indonesia, as the second largest tin producer in the world, has a byproduct from the production of tin. This byproduct is in the forms of tin slag containing tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5). Tantalum and niobium are in the 14 critical materials, so the focus of their recovery from tin slag will be an alternative option. This study focuses on the recovery of tantalum pentoxide and niobium pentoxide from the tin slag. In the process, one part of the tin slag sample was sieved only (BTS), and the other was roasted at 900°C, water quenched and then sieved (BTS-RQS). Samples BTS and BTS-RQS were characterized by thermo gravimetric analysis (TGA) and X-ray Florence (XRF). One part of BTS-RQS sample was dissolved in hydrofluoric acid (HF) and the other was dissolved in hydrochloric acid (HCl), washed with distilled water, and then dissolved into sodium hydroxide (NaOH). Each sample was characterized by using XRF. The BTS sample produced the highest recovery of 0.3807 and 0.6978% for Ta_2O_5 and Nb_2O_5, respectively, from the particle size of -1.00+0.71 and a fraction of 47.29%, while BTS-RQS produced the highest recovery of 0.3931 and 0.8994% for Ta_2O_5 and Nb_2O_5, respectively, on the particle size of -0.71+0350 and a fraction of 21%. BTS-RQS, dissolved with 8% hydro fluoride acid, yields tantalum pentoxide and niobium pentoxide with a ratio of 2.01 and 2.09, respectively. For the sample BTS-RQS dissolve first with 6M hydrochloric acid, washed with distilled water, then dissolved with sodium hydroxide 10M, the yield ratios are 1.60 and 1.84 for tantalum pentoxide and niobium pentoxide, respectively. In this study, it is found that the dissolution by using hydrofluoric acid 8% yields the best ratio.

Keywords: Dissolution; Niobium pentoxide; Tantalum pentoxide; tin slag.

1. INTRODUCTION

Indonesia is the second largest tin producer in the world, with a production area located on the island of Bangka and Belitung. The number of tin smelters operating on Bangka and Belitung is 34 smelters, according to the organization for economic co-operation and development (OECD) in November 2012 [1].

In the tin production process, results from byproducts such as tin slag-1 and slag-2. Tin slag-1 contains 20-30% Tin, while tin slag-2 contains 2-3% Tin. Tin slag-2 is a remitted tin slag-1 into the reverberatory furnace and containing transition metal oxides such as tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5) [2]. Tantalum pentoxide and niobium pentoxide can be extracted from this slag. Other than from tin slag, tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5) can be recovered by mining.

Tantalum pentoxide (Ta_2O_5) can be recovered from tantalite ore (oxides) [3] that can be found in the earth's crust of about 2 ppm [4]. While niobium oxide can be economically extracted from the deposit

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of pyrochlore and columbite, which can generally be divided into two types, namely: primary carbonatite deposits with containing 0.5-0.7% Nb_2O_5 and enriched pegmatite deposits (carbonite mineral leaching products of primary carbonatite) that contains 1.0-7.0% Nb_2O_5 [5].

Tantalum and niobium are in the 14 critical materials [6] so the focus of their recovery from tin slag will be an alternative option. For tantalum itself, it is now considered as midterm critical metal where the level of its availability is only until 2020 [3,7–9]. Efforts of obtaining tantalum and niobium from tin slags have been carried out by some previous researchers [2,10–16]. Tin slag from Indonesia has been informed to contain $(Ta, Nb)_2O_5$ at about 2.7% [2].

For comparison, the recovery of tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5) from the mine, we can refer to Anita project owned by Les Mineraux Crevier at the mine site in the Lac-Saint-Jean Quebec, Canada. Anita project has measured reserves of approximately 25.8 million ton with niobium oxide of 0.196% and tantalum oxide of 234 ppm. Other than that, the mineral inferred resources are 15.42 million ton. These reserves are assumed to be a cut-off grade of 1,000 g/t Nb [17].

Metal extraction can be done by hydrometallurgy, pyrometallurgy and electrometallurgy. On simple hydrometallurgy can be done in liquid-liquid extraction [18–20] or the method of dissolution. This study uses the method of dissolution to reduce other oxides content.

This research's challenge is the dominant other oxides content with a total of 99.09% while the precious metal content is of less than 1%. Previous researches used tin slag with a total content of Ta_2O_5 and Nb_2O_5 over 2.7%, failed to describe the relationship of process to the decreasing content of other oxides [2,10–16]. The decreasing contents of other oxides on this study are expected to be the leading direction for the next processes in increasing Ta_2O_5 and Nb_2O_5 of tin slags.

We will observe in this study the effect of sieving, roasting at 900°C-water quenching-sieving to the weight and content fraction of Ta_2O_5 and Nb_2O_5, and the effect of BTS-RQS dissolution with hydrofluoric acid and its relationship with yield ratio of Ta_2O_5 and Nb_2O_5. Furthermore, we will see the effect of dissolution with hydrochloric acid and sodium hydroxide as well as its relationship with yield ratio of Ta_2O_5 and Nb_2O_5.

2. METHODOLOGY

2.1 Feasibility Study

To calculate the BTS potential reserved of tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5), we compared the BTS's of PT Timah Tbk and Anita project. The assumed minimum production of PT Timah Tbk to be of 35,000 MT/year [21], and slag output of about 10% [22] so slag amount was about 3,500 MT/year.

2.2 Materials

Tin slag used in this research was provided by PT Timah Tbk, Indonesia. Initial tin slag characterization was done by XRF (Spectro Xepos Ametek). The results can be seen in Table 1. This study used HF (technical solution), and HCl and NaOH (pa, Merck).

2.3 Procedure of pre-removed of other oxides

First sample, Bangka tin slag sieved with sieve size +1; -1+0.71; -0.71+0350; -0350+0063 and -0.063 mm, then each particle size was characterized by XRF.

Table 1. The initial characterization of Bangka tin slag

Ta_2O_5 (%)	Nb_2O_5 (%)	SiO_2 (%)	CaO (%)	TiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	ZrO_2 (%)	El & Min.OO (%)
0.33	0.64	34.26	15.44	11.92	11.7	8.84	4.78	12.06

**El & Min.OO = element & minor other oxides*

Second samples, Bangka tin slag was roasted at a temperature of 900°C for 2 hours, water quenched and heated in the oven to demoisturize. The samples, then, sieved to the size of +1; -1+0.71; -0.71+0350; -0350+0063 and -0.063 mm and each size particle were characterized by XRF. Both samples, BTS and BTS-RQS were analyzed with thermo gravimetric, Perkin Elmer STA 6000, to record the sample weight change as a function of temperature.

Dissolving with hydrofluoric acid has done by placing BTS-RQS into 4 pieces of plastic containers, each of which weighs 20 grams. Then, the plastic containers filled with 100 ml of hydrofluoric acid with each concentration of 4, 8, 16, and 32%. After 2 hours, each sample was rinsed with distilled water and characterized by XRF.

The other dissolutions were to put BTS-RQS into 16 beaker glasses, each of which weighs 20 grams, on every 4 beaker glasses filled with 100 ml of hydrochloric acid concentration of 6, 8, 10 and 12 M, then immersed for 2 hours. After the hydrochloric acid dissolutions completed, the samples were rinsed with distilled water. For every 4 beaker glasses from different concentrations of hydrochloric acid, sodium hydroxide with a concentration of 6, 8, 10, 12M, respectively, was added and immersed for 20 hours. The sample code, type, concentration and solvent dissolution time are showed in Table 2.

Table 2. Sample code, solvent type, concentration (%/M) and dissolution time (hours)

Sample code	Type of solvent, concentration (%/M) and Dissolution Duration (hours)
F4	Fluoride Acid 4%, 2 h
F8	Fluoride Acid 8%, 2 h
F16	Fluoride Acid 16%, 2 h
F32	Fluoride Acid 32%, 2 h
A6B6	Chloride Acid 6M, 2h continue to Sodium Hydroxide 6M, 20 h
A6B8	Chloride Acid 6M, 2h continue to Sodium Hydroxide 8M, 20 h
A6B10	Chloride Acid 6M, 2h continue to Sodium Hydroxide 10M, 20 h
A6B12	Chloride Acid 6M, 2h continue to Sodium Hydroxide 12M, 20 h
A8B6	Chloride Acid 8M, 2h continue to Sodium Hydroxide 6M, 20 h
A8B8	Chloride Acid 8M, 2h continue to Sodium Hydroxide 8M, 20 h
A8B10	Chloride Acid 8M, 2h continue to Sodium Hydroxide 10M, 20 h
A8B12	Chloride Acid 8M, 2h continue to Sodium Hydroxide 12M, 20 h
A10B6	Chloride Acid 10M, 2h continue to Sodium Hydroxide 6M, 20 h
A10B8	Chloride Acid 10M, 2h continue to Sodium Hydroxide 8M, 20 h
A10B10	Chloride Acid 10M, 2h continue to Sodium Hydroxide 10M, 20 h
A10B12	Chloride Acid 10M, 2h continue to Sodium Hydroxide 12M, 20 h
A12B6	Chloride Acid 12M, 2h continue to Sodium Hydroxide 6M, 20 h
A12B8	Chloride Acid 12M, 2h continue to Sodium Hydroxide 8M, 20 h
A12B10	Chloride Acid 12M, 2h continue to Sodium Hydroxide 10M, 20 h
A12B12	Chloride Acid 12M, 2h continue to Sodium Hydroxide 12M, 20 h

3. RESULTS AND DISCUSSION

3.1 Feasibility Study Result

With Anita Project's economic parameter basic data, potential reserved calculation of tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5) from tin slag can be seen in Table 3.

Table 3. Comparative of potential reserves calculation project Anita and Bangka tin slag.

Description	Anita Project	Bangka Tin Slag
Ta_2O_5 content	234 ppm	0.33%
Nb_2O_5 content	0.196%	0.64%
Measured reserve	25.8 million ton	3,500 ton/year*
Inferred reserve	15.42 million ton	
Mine Life	18 year	

** Calculation in minimum production.*
Slag Production of PT Timah Tbk. 3,500-5,000 ton/year.

3.2 Initial Process of BTS

To simplify observation and discussion, other oxides were classified into two groups; one group was major other oxides (MOO) and another group that consisted of elements and minor oxides (EMO). Table 4 had the details data.

Table 4. Major others oxides and element & minor other oxides

Major Others Oxides (MOO)	Element & Minor Others Oxides (EMO)
SiO_2, CaO, TiO_2, Al_2O_3, Fe_2O_3, ZrO_2	Na_2O, MgO, P_2O_5, S, Cl, K_2O, Sc, V_2O_5, Cr_2O_3, MnO, CoO, NiO, CuO, ZnO, Ga, Ge, As_2O_3, Se, Br, Rb_2O, SrO, Y, MoO, Ru, Rh, Pd, Ag, Cd, In, SnO_2, Sb_2O_5, Te, I, Cs, BaO, La_2O_3, Ce_2O_3, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, WO_3, Au, Hg, Tl, Pb, Bi, Th and U

Characterization of BTS with XRF can be seen in Table 5. The particle size of -1.00+0.71 mm had the highest content of Ta_2O_5 and Nb_2O_5 that were 0.3807 and 0.6978%, each with a weight fraction of 47.29%.

The initial purpose of reduction roasting of 900°C and water quenched of tin slag was to observe the effects of reduction roasting and water quenched on particle size reduction. But the experiment showed another result that there was a decreased content of EMO. Particle size reduction due to roasted 900°C and water quenched affected only about 2% by weight in 1.00 mm particles size and a significantly affected particles size smaller than 1.00 mm. The percentage decreased EMO content was from 56.17%-16.44% to 20.84%-11:30%, and this can be seen in Table 5 and Table 6.

A similar process was also carried out in the study of Sulaksana Permana et al. [23], in this study it was explained that the 900°C reduction roasted processes followed by water quenched would expand the wetting area, thereby facilitating the leaching reaction of the other oxides. Explanation of this information with drawings is given the following details: BTS containing valuable oxides with a small wetting area; the occurrence of changes in the porous surface after the roasting process; there are thermal cracking and fracture condition after the water quenching process; finally there is a reduction in particle size to expand the wet surface.

3.3 Thermo Gravimetric Analysis of BTS

To observed thermal decomposition, the initial BTS and BTS-RQS were analyzed using TGA. The linear heating rate of TGA characterization was 10°C/min, from room temperature to 900°C. The result was showed in Fig. 1.

BTS endothermic peak can be seen at about 600°C, its weight loss started at temperature of 100°C until the endothermic peak, while the increased of its weight began at a temperature of 700°C. BTS-RQS weight loss occurred from the temperature of 100°C to about 900°C, refer to Fig. 1(b), Table 5 and Table 6, this weight loss occurred due to the oxidation of EMO.

Table 5. Size and oxides analysis of the initial tin slag

Particle size (mm)	Weight (gram)	Fraction (%)	Ta_2O_5 (%)	Nb_2O_5 (%)	ZrO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	TiO_2 (%)	CaO (%)	SiO_2 (%)	El & Min.OO (%)
+1.00	479	47.9	0.3239	0.6548	4.732	7.285	7.595	9.761	12.15	22.03	35.4683
-1.00+0.71	472.9	47.29	0.3807	0.6978	4.951	7.77	9.124	11.59	13.59	24.3	27.5965
-0.71+0.350	18.6	1.86	0.3393	0.6484	4.448	7.395	8.796	9.841	11.04	23.4	34.0923
-0.350+0.063	23.1	2.31	0.2758	0.6059	4.212	11.77	8.706	9.872	12.26	35.85	16.4483
-0.063	2.3	0.23	0.2937	0.3715	2.396	4.406	8.112	6.539	5.677	16.03	56.1748
Weight loss	4.1	0.41	-	-	-	-	-	-	-	-	-
Total	1,000	100	-	-	-	-	-	-	-	-	-

Table 6. Size and elemental analysis of BTS- RQS

Particle size (mm)	Weight (gram)	Fraction (%)	Ta_2O_5 (%)	Nb_2O_5 (%)	ZrO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	TiO_2 (%)	CaO (%)	SiO_2 (%)	El & Min.OO (%)
+1.00	441.2	45.8	0.3475	0.7439	5.354	11.25	9.063	12.03	16.4	33.51	11.3016
-1.00+0.71	221	22.9	0.3536	0.7981	5.641	10.55	9.28	11.53	15.48	34.1	12.2673
-0.71+0.350	202.5	21.0	0.3931	0.8994	6.358	9.672	10.53	11.96	15.88	30.12	14.1875
-0.350+0.063	71.2	7.4	0.3502	0.6343	4.412	10.27	9.84	11.51	14.85	32.19	15.9435
-0.063	15.9	1.7	0.3303	0.6867	4.754	9.558	11.63	10.74	13.46	28	20.841
Weight loss	11.8	1.2	-	-	-	-	-	-	-	-	-
Total	963.6	100	-	-	-	-	-	-	-	-	-

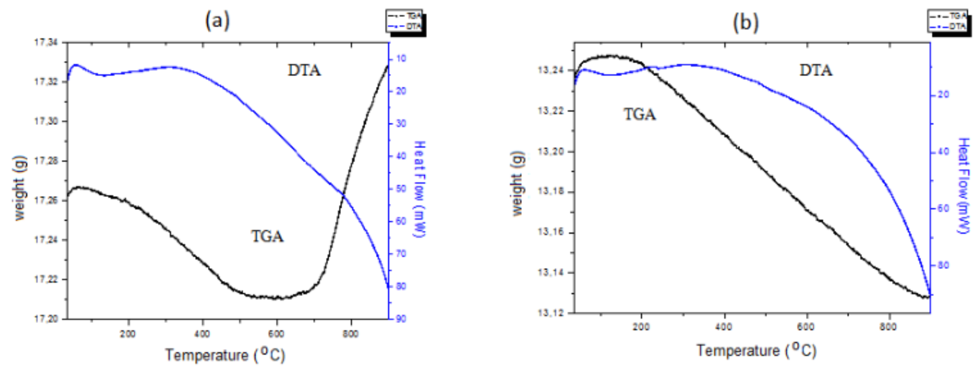

Fig. 1. (a) DTA and TGA chart from the initial Bangka tin slag. (b) DTA and TGA chart from the roasted 900°C and water quenched of Bangka tin slag

3.4 The Effect of HF and HCl+NaOH Dissolution

3.4.1 The effect of HF dissolution

BTS-RQS dissolution with hydrochloric acid 8% produced the highest Ta_2O_5 and Nb_2O_5 content of 0.664% and 1.339%, with its yield ratio were 2.01 and 2.09, respectively, this can be seen in Fig. 2 (a) and Table 7.

This dissolution showed that the higher HF concentration, the higher SiO_2 dissolution rate. This result was in line with SiO_2 dissolution on HF [24]. Dissolution with HF 32% will cause the increased SiO_2 content to 13.06%; this is due to the formation of solid SiO_2. This solid SiO_2 was why weight loss value becomes 106.5%, which can be seen in Table 7.

Hydrochloric acid dissolved Fe_2O_3 well and TiO_2 slightly. HF 4, 8 and 16% were less successfully dissolving EMO and could not dissolve CaO, Al_2O_3 and ZrO_2. This information can be seen in Fig. 2 (b).

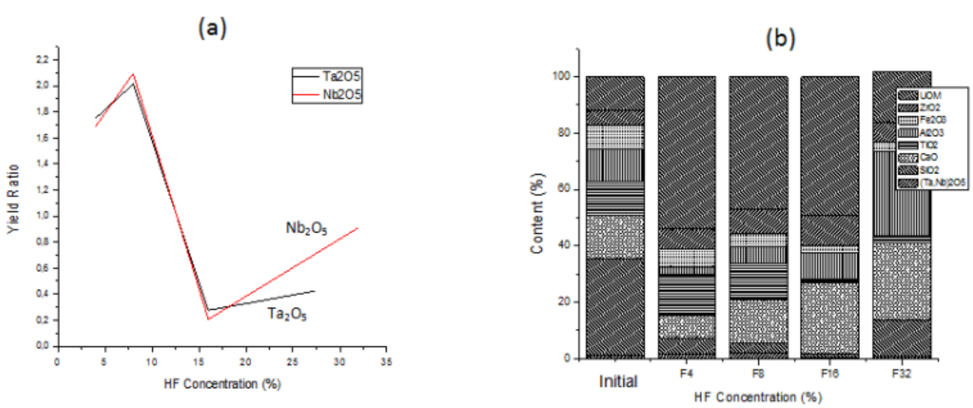

Fig. 2. (a) The effect of hydrofluoric acid concentration on Ta_2O_5 & Nb_2O_5 yield ratio. (b) The effect of hydrofluoric acid concentration to Ta_2O_5, Nb_2O_5 and other oxide content

Table 7. Chemical analysis of hydrofluoric acid dissolved residue

Sample Code	Total Pct. WL*	Pct. Ta_2O_5 (initial)	Pct. Ta_2O_5 (final)	Yield Ratio** Ta_2O_5	Pct. Nb_2O_5 (initial)	Pct. Nb_2O_5 (final)	Yield Ratio** Nb_2O_5	Loss of Ignition
F4	76.0	0.33	0.578	1.75	0.64	1.080	1.69	38.653
F8	56.5	0.33	0.664	2.01	0.64	1.339	2.09	30.589
F16	55.0	0.33	0.091	0.28	0.64	0.133	0.21	34.707
F32	106.5	0.33	0.160	0.48	0.64	0.584	0.91	-1.8748

**WL = Weight Loss. **Yield Ratio= content before process / content after process*

3.4.2 Effect dissolution with HCl+NaOH

The dissolution BTS-RQS with HCl 6M and NaOH 10M produced Ta_2O_5 and Nb_2O_5 content of 0.528 and 1.182%, respectively, with yield ratio for Ta_2O_5 and Nb_2O_5 were 1.60 and 1.85, respectively. HCl and NaOH dissolution graphics can be seen in Fig. 3 and Table 8. Contents of Ta_2O_5 and Nb_2O_5 would have about twice the yield ratio if HCl dissolution were followed by NaOH with a particle size smaller than 0.150 mm or the particle size of between 0.180 and 0.150 mm [16].

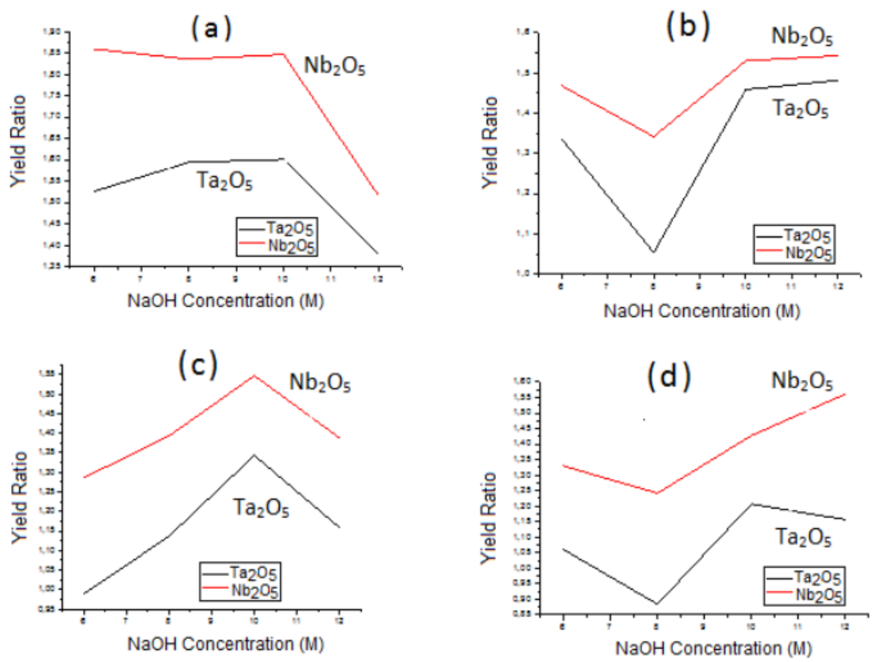

Fig. 3. The effect of chloride acid (a) 6M (b) 8M (c) 10 M (d) 12 M on concentrate variation of NaOH and Ta_2O_5 & Nb_2O_5 yield ratio

HCl and NaOH can be dissolved EMO well but was less successful dissolving SiO_2, Fe_2O_3 and TiO_2. HCl and NaOH were not successfully dissolving CaO, Al_2O_3 and ZrO_2, and this showed in Fig. 4. Leaching of Indonesia tin slag with acid (HCl 2N and HF 0.14 N) and alkaline (NaOH 2N), which used a Pyrex reactor with a double wall for hot water circulation, caused a weight loss of 46.3% with Ta_2O_5 and Nb_2O_5 final content of the leaching were 1.9 and 2.9%, respectively [2].

Tabel 8. Chemical analysis of HCl and NaOH dissolved residue

Sample Code	Total Pct. WL*	Pct. Ta_2O_5 (initial)	Pct. Ta_2O_5 (final)	Yield Ratio** Ta_2O_5	Pct. Nb_2O_5 (initial)	Pct. Nb_2O_5 (final)	Yield Ratio** Nb_2O_5	Loss of Ignition
A6B6	70.5	0.33	0.5037	1.53	0.64	1.19	1.86	-3
A6B8	74.5	0.33	0.5261	1.59	0.64	1.175	1.84	-3
A6B10	71.5	0.33	0.5283	1.60	0.64	1.182	1.85	-3
A6B12	72.5	0.33	0.4547	1.38	0.64	0.9705	1.52	36.304
A8B6	77.0	0.33	0.4412	1.34	0.64	0.94	1.47	30.496
A8B8	74.0	0.33	0.3475	1.05	0.64	0.8586	1.34	44.465
A8B10	76.5	0.33	0.4814	1.46	0.64	0.9795	1.53	26.217
A8B12	79.0	0.33	0.4889	1.48	0.64	0.9876	1.54	20.425
A10B6	80.0	0.33	0.3264	0.99	0.64	0.8234	1.29	42.218
A10B8	82.0	0.33	0.3753	1.14	0.64	0.8916	1.39	35.494
A10B10	83.0	0.33	0.4434	1.34	0.64	0.9901	1.55	22.12
A10B12	83.0	0.33	0.3823	1.16	0.64	0.8885	1.39	33.061
A12B6	91.0	0.33	0.35	1.06	0.64	0.8509	1.33	28.087
A12B8	91.0	0.33	0.2918	0.88	0.64	0.7947	1.24	39.887
A12B10	91.0	0.33	0.3982	1.21	0.64	0.9137	1.43	24.512
A12B12	84.5	0.33	0.3818	1.16	0.64	0.9993	1.56	-3

**WL = Weight Loss. **Yield Ratio = content before process / content after process*

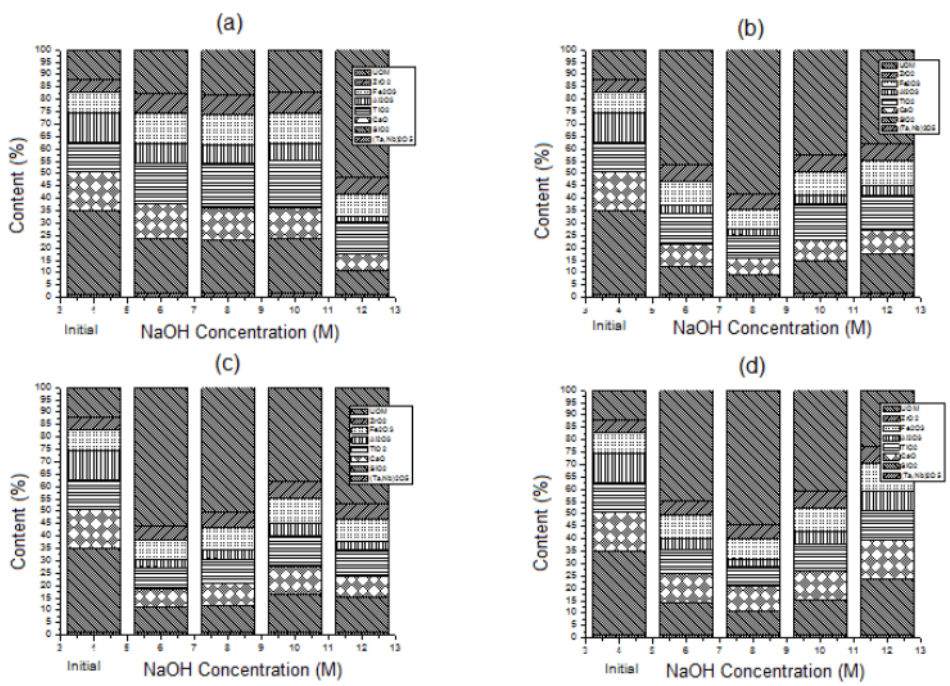

Fig. 4. The effect of chloride acid (a) 6M (b) 8M (c) 10 M (d) 12 M on concentrate variation of NaOH to Ta_2O_5, Nb_2O_5 and others oxides content

Other studies conducted by Hendri F. Akli et al. [9] and Shiva F. Vincia et al. [25] carried out in a sequence of 900°C roasting process followed by water quenching, the first leaching with 8M NaOH followed by variations in the concentration of H_3PO_4 [9] and $HClO_4$ [25]. The optimal condition of the study by Hendri F. Akli et al. occurred at 1M H_3PO_4 concentration. An increase in tantalum from 0.23 to 0.79% with a yield ratio of 3.43, and an increase in niobium from 0.47 to 1.45% with a yield ratio of 3.08 were achieved on the condition. The study's optimal condition by Shiva F. Vincia et al. occurred at 0.8M $HClO_4$ concentration, an increase in tantalum from 0.23 to 0.79% with a yield ratio of 3.43, an increase in niobium from 0.47 to 1.28% with a yield ratio of 2.72. This information shows that alkaline leaching, followed by an acid solution gives a better prospect of increasing tantalum and niobium content. This information contrasts with the results of the study by I. Gaballah et al. using samples of tin slag from Indonesia, where the first leaching with acid followed by alkaline had a higher yield ratio than the first leaching with alkaline followed by acid [2]. Gaballah et al., the 900^{o}C roasting process followed by water quenching was not carried out.

4. CONCLUSIONS AND SUGGESTIONS

Potential reserved of tantalum pentoxide (Ta_2O_5) and niobium pentoxide (Nb_2O_5) from BTS can be used as potential alternative revenues for Bangka and Belitung province.

The roasted at 900°C and water quenched can be reduced EMO content. BTS-RQS produced the highest contents of Ta_2O_5 and Nb_2O_5 with the dissolution of HF 8%; also, the dissolution with HCl 6M and NaOH 10M produced the highest contents of Ta_2O_5 and Nb_2O_5.

To dissolved SiO_2 and Fe_2O_3, HF was better than with HCl+NaOH. HF and HCl+NaOH did not dissolve CaO, Al_2O_3 and ZrO_2. BTS-RQS dissolution with HF diluted less EMO, while with HCl 6% and NaOH 6.8 and 10% showed lower contents of SiO_2, Fe_2O_3 and TiO_2.

For further research, the researcher will need to roast above 900°C and water quench to see its effect. The next, researcher will need to investigate that solid residue from HF 8% dissolution, redissolved in HCl 6M and NaOH 10M to reduce EMO content.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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