Hydrometallurgical removal of uranium and thorium from Ethiopian tantalite ore

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Abstract: This study focused on the leaching of uranium and thorium from a high grade Ethiopian tantalite ore using sulfuric acid. The effects of variables such as acid concentration, temperature and leaching time were studied. In general, the leaching efficiency of uranium increases with increasing temperature from 100 to 300 °C whereas the opposite trend was observed for thorium. The amount of uranium leached increased from 69.1% at contact time of 1 hr to 88.6% when the tantalite ore was leached for 3 hrs. On the other hand, for the experiments conditions considered in this study, the leaching behavior of uranium and thorium did not change significantly with varying sulphuric acid concentration from 70 to 90 wt%. Overall, the highest dissolution of uranium and thorium were achieved at 100 °C, 70 wt% H_2SO_4 concentration and 1 hr contact time.

Keywords: Leaching, Tantalite, Uranium, Thorium

1. Introduction

In many modern electronics and nuclear industries, tantalum (Ta) and niobium (Nb) are key commodities with wide applications in the manufacture of capacitors, jet engines and turbine parts, spacecraft and missiles skins, rocket nozzles etc. (Bose and Gupta, 2001; El-Hazek et al., 2012). These various applications have steadily increased the demand for Ta and Nb in the past decades.

Pegmatites are the major sources of these rare metals where columbite-tantalite, with a chemical formula of $(Fe,Mn)(Nb,Ta)_2O_6$, host Ta and Nb. The largest producers of Ta and Nb are Brazil and Australia, followed by Canada, Mozambique and Ethiopia (Nete and Purcell, 2018). The Kenticha pegmatite deposit found in southern Ethiopian is an example of a current tantalum mining operations up on which the present study is based. The Kenticha pegmatites are characterized by quartz-feldspar-muscovite framework containing tourmaline, beryl, columbite, and mangano-tantalite (Kim et al., 2013; Kuster et al., 2009; Tadesse and Zerihun,1996). In columbite-tantalite ores, Ta and Nb often occur in association with thorium, uranium, titanium, and rare-earth elements (REEs) (Dampare et al., 2005). Thus, these ores can be regarded as Naturally Occurring Radioactive Materials (NORM). The titanium in these tantalum ores is often recovered in the form of ilmenite by using magnetic separation (Nete et al., 2014).

The beneficiation and extraction of Ta and Nb from columbite-tantalite ores becomes extremely challenging in the presence of radioactive elements such as thorium and uranium. In addition, these radioactive elements will complicate the transportation and handling of concentrates (Dampare et al.,

2005). In general, if minerals and their concentrates contain 1 Bq/g ThO_2 and U_3O_8, they are classified as of concern for the purpose of mining and handling, while those containing ten times as high are considered a concern for transportation (Nete et al., 2014). The regulations on the transport of radioactive materials and concentrates vary depending on the countries concerned (Simandl, 2001). Generally, a license is required to handle and transport materials classified as Class 7 with total radioactivity exceeding 0.5 Bq/g. Thus, the safe removal of Th and U from minerals and concentrates is necessary prior to transportation.

The leaching behaviour niobium, tantalum, thorium, uranium, titanium and rare earth elements has been investigated on tantalite minerals collected from Kab Amiri using H_2SO_4 at different temperatures and acid concentrations (El-Hussaini and Mahdy, 2002). It was reported that a mixture of sulfuric acid and nitric acid (weight ratio of 1:3) at 200 °C resulted in an almost complete recovery of both niobium and tantalum, whereas the leaching efficiency of thorium and uranium were 86% and 70%, respectively (El-Hussaini and Mahdy, 2002). The low recovery of uranium was attributed to the refractory nature of the host mineral. A study on pegmatite ore deposit obtained from Mozambique indicated that 64.14 wt% U_3O_8 and 60.77 wt% ThO_2 were leached into solution by using acid leaching at 50°C for 3 hrs using concentrated H_2SO_4 (Nete et al., 2014). It was also suggested that, under these experimental conditions, the leaching of Nb_2O_5 and Ta_2O_5 was minimal. In another study, washing tantalite concentrates using 1 vol% KOH led to negligible amounts of U_3O_8 and ThO_2 in the concentrate (Berhe et al., 2017). Sulphuric acid leaching is a common dissolution method not only for the removal of Th and U from columbite-tantalite ores but also for the extraction of U from primary ores such as uraninite (Bhargava et al., 2015; Madakkaruppan et al., 2016) and betafite (Nettleton et al., 2015). The presence of reactive gangue minerals such as chlorite, biotite and apatite generates detrimental ions like Si, Al, Fe, Mg and P during leaching which eventually result in low U recovery (Madakkaruppan et al., 2016).

The literature review shows that the success in the removal of Th and U from parent ores and concentrates depends on both the mineralogical factors associated with the ores and the leaching conditions such as leaching temperature, acid concentration and leaching time. Thus, it is possible that acid leaching of radioactive oxides could be effective for some types of ores and ineffective for others based on certain mineralogical factors including degree of liberation, mineral association and degree of interlocking, and gangue mineral composition. The present study was conducted on a columbite-tantalite ore from the Kenticha area, Southern Ethiopia, which contains relatively high amounts of U and Th elements. The effect of concentration of sulfuric acid, temperature and leaching time on the rate of removal of radioactive oxides was studied.

2. Materials and methods

2.1. Materials and apparatus

Ore samples were collected from the Kenticha area, southern Ethiopia, from five different zones based on geological variation. Table 1 shows the composition of the ore collected from these zones, and each sample contains notable quantities of Ti, Mn and Fe in addition to the main economic metals (Ta and Nb). It can be seen that the wt% U_3O_8 in the ore can be as high as 1.23% while the ThO_2 can constitute up to 0.65% (Table 1, sample 4). Sample 4 was selected in this study as it contains the highest amount of uranium and thorium. At particle size of >100 μm, some coarse partcle size fraction of the sample remained undissolved during acid leaching. Thus, in this experiment the ore was crushed and milled (Thomas Wiley laboratory mill) to achieve particle size of <100 μm to ensure dissolution. The reagent used for leaching is analytical grade sulfuric acid (ATICO, India) while distilled water was used to prepare the required solutions.

Table 1. Composition of Ethiopian Kenticha tantalite ore collected from five sites (wt%).

Sample ID	Ta_2O_5	Nb_2O_5	U_3O_8	TiO_2	SnO_2	ThO_2	MnO_2	Fe_2O_3
1	45.24	19.58	1.07	0.31	0.08	0.37	6.93	16.23
2	59.23	17.38	0.80	0.15	0.08	0.17	5.55	14.89
3	53.71	16.30	0.92	0.58	0.10	0.21	7.00	14.98
4	48.69	15.96	1.23	0.88	0.10	0.65	7.60	17.95
5	44.23	16.65	1.21	0.83	0.10	0.07	7.66	17.47

2.2. Experimental procedure

The leaching experiments were carried out in an autoclave with 5 g of tantalite sample (<100 μm) in 250 ml silicate glass beakers with a maximum temperature of 300°C. The beakers were installed with magnetic stirrer with up to 250 rev/min stirring rate. Sulfuric acid with different concentration (70 – 90% wt/wt) was used for the leaching of uranium and thorium at an ore to acid ratio of 1:6 wt/wt. The leaching step was conducted in the presence of oxygen, which was introduced by blowing oxygen into the beaker, to facilitate the oxidation and dissolution of insoluble UO_2 that is naturally found in mineral deposits. Oxidation is expected to convert the UO_2 (U^{+4}) in to U^{+6} state. Following leaching, the leach liquor was separated from the residue by filtration. Finally, the dissolved species were sent for elemental analysis while the residue was washed with distilled water, dried and analysed. Scheme in Fig. 1 shows the steps followed while conducting the experiments. All experiments were repeated at least 5 times and standard deviation for each test has been given as appropriate.

2.3. Characterization techniques

The thermal stability of the tantalite ore sample was analysed using thermo-gravimetric analysis (TGA, model TA 50, Shimadzu America) with a platinum cell in nitrogen at flow rate of 30 ml/min. The stability was determined by recording the rate of weight loss of the sample with increase in temperature. The TGA data was also used to investigate the moisture content of the ore and the presence of impurities decomposed at higher temperature. Elemental analysis of feed samples and leached residues were conducted using EDXRF (PANalytical Model Epsilon 3[XLE], Netherlands). The changes in topography and composition before and after leaching of the sample were studied by using scanning electron microscope (SEM) equipped with energy-dispersive spectroscopy (EDS) (model JEOL JSM-848).
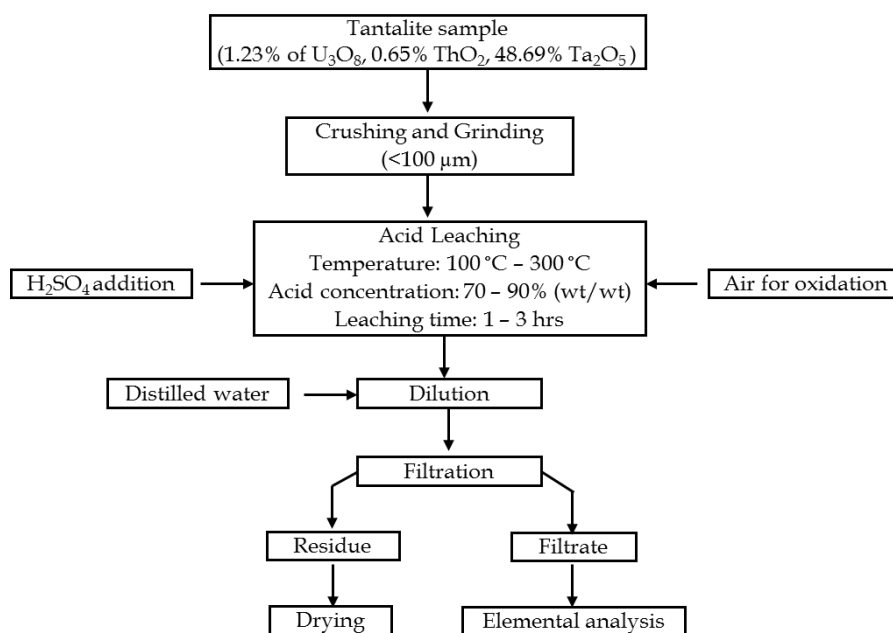

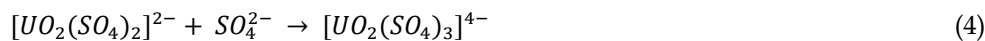Fig. 1. Experimental scheme for leaching of uranium and thorium from tantalite ore

3.   Results and discussion

3.1. Tantalite ore characterization

The stability of the tantalite ore sample was analysed using TGA-DTA. Fig. 2 shows that the weight loss in the sample up on heating to 1000 °C is insignificant (0.9%) and that the reaction is exothermic. The small weight loss suggests that the tantalite sample is thermally stable, and contains no significant moisture or gaseous decomposition products at the treatment temperature of 1000 °C. The grey-scale SEM image of the tantalite feed gives an indication that the mineralogical association of the ore is not highly intricate (Fig. 3). Although only few grain boundaries are seen in the specimen, in addition to

the mangano-tantalite main component, the ore is expected to contain ferro-columbite, ilmenite, iron oxides, and other gangue silicates (Berhe et al., 2018). The EDS of feed pegmatite ore shown in Fig. 4 shows the elemental composition of the ore with uranium, thorium and tantalum peaks detected as expected.

During the acid leaching of U containing ores, H_2SO_4 may ionize to form sulfate, bisulfate, and hydrogen ions. These species react with hexavalent uranium (UO_2^{2+}) to produce uranyl sulfate and the complex uranyl sulfate anions according to the following reactions (Merritt, 1971):

$$UO_3 + 2H^+ \rightarrow UO_2^{2+} + H_2O \tag{1}$$

$$UO_2^{2+} + SO_4^{2-} \rightarrow UO_2SO_4 \tag{2}$$

$$UO_2SO_4 + SO_4^{2-} \rightarrow [UO_2(SO_4)_2]^{2-} \tag{3}$$

$$[UO_2(SO_4)_2]^{2-} + SO_4^{2-} \rightarrow [UO_2(SO_4)_3]^{4-} \tag{4}$$

The dissolved uranium may occur in any of the above forms, depending upon acid and uranium concentration, temperature, and other variables in the system. In the next sections, the effects of leaching temperature, acid concentration and time on dissolution of U and Th were investigated.
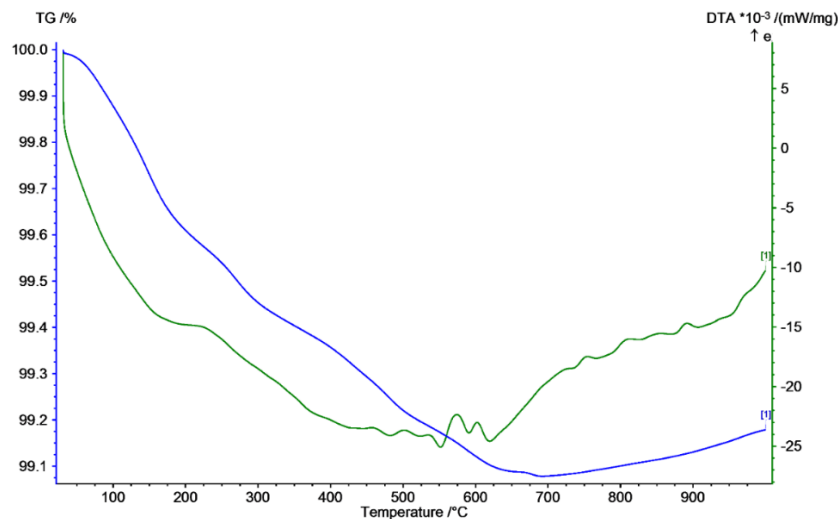

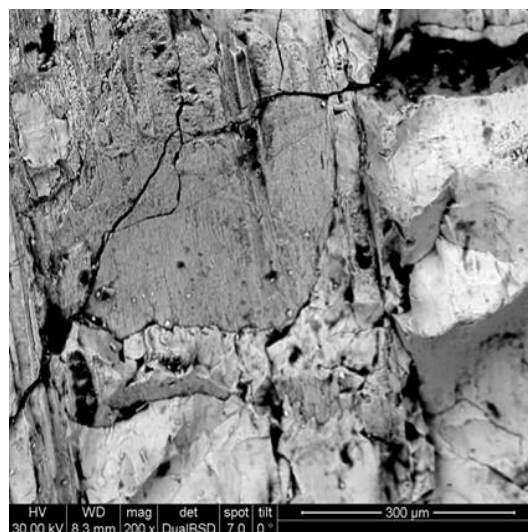Fig. 2. Thermogravimetric analysis of the tantalite ore showing wt% loss upon heating to 1000 °C

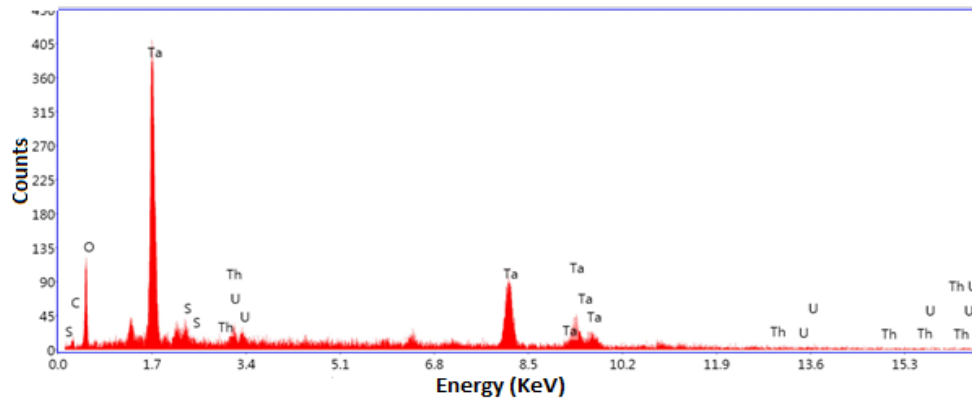Fig. 3. SEM micrograph showing the morphology of Kenticha tantalite ore

Fig. 4. EDS of Kenticha pegmatite ore before concentration and acid leaching (sample 4 in Table 1)

3.2. Effect of temperature on leaching of U, Th and Ta

The effect of temperature on the leaching of uranium and thorium from tantalite was studied in the range from 100 to 300 °C. Fig. 5 shows the change in leaching efficiency of U, Th and Ta in 80 wt% sulfuric acid concentration and 2 hrs contact time. The leaching behaviour of Ta was also shown to monitor to what extent Ta is affected by the leaching process. It can be seen that a total removal of up to 83.7 wt% of uranium was achieved at 300 °C. The maximum removal observed for Th was 67.7 wt% at 150 °C. In the case of Ta, the lowest dissolution observed was 10.1 wt% at 300 °C. In a related study on tantalite ore, Nete et al (2014) reported U dissolution of 86.51 wt%, Th dissolution of 80.05 wt% and Ta removal of 4.4 wt% at 18.0 M H_2SO_4 concentration, 230 °C and a leaching time of 1.5 hr. The results for U dissolution are comparable to our findings although the experimental conditions as slightly different. However, it is important to note that Nete et al. (2014) observed relatively low dissolution of Ta probably due to the lower grade of ore used in their study. El-Hussaini and Mahdy (2002) studied the effect of temperature during sulphuric acid leaching of U and Th bearing niobium–tantalum ore and found that leaching of Nb, Ta, Th and U elements does not necessarily increase with increasing temperature. They showed that leaching extents for these elements decreases significantly using concentrated sulfuric acid (18 M) when the temperature was increased beyond 200 °C on ore particle size of −74 µm at an ore to acid ratio of 1.0:2.5 and leaching time of 2 hr (El-Hussaini and Mahdy, 2002). It was suggested that at higher leaching temperatures hydrolysis may occur thus leading to decreased leaching efficiency.
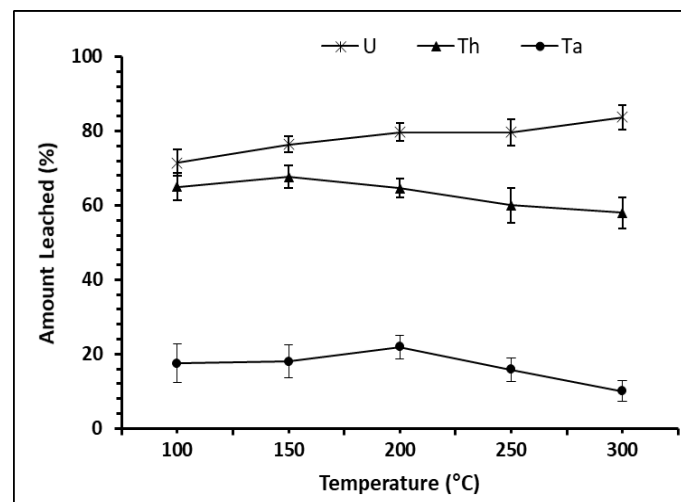

Fig. 5. The effect of temperature on leaching efficiency of U, Th and Ta in 80 wt% sulfuric acid concentration after 2 hrs contact time at ore to acid ratio of 1:6 wt/wt, particle size of <100 µm, and 250 rev/min stirring rate

3.3. Effect of sulfuric acid concentration on leaching of U, Th and Ta

The effect of concentration of sulfuric acid was studied to identify the maximum capacity of leaching. The concentration of sulfuric acid was varied, while other leaching conditions were kept constant.

Leaching temperature of 200 °C was selected because as can be seen from Fig. 5 no appreciable dissolution of the target elements occurs above this temperature. Fig. 6 indicates that at leaching time of 2 hrs and temperature of 200 °C, the leaching behaviour of U and Th did not change significantly with varying acid concentration from 70 to 90 wt%. In the leaching conditions of the present study, the average U dissolution was 80.8 wt%, while that of Th was 63.1 wt%. Relatively higher dissolution rates were also obtained for Ta. At sulfuric acid concentrations greater than 10.0 M, constant leaching of Th and U was also observed elsewhere (Nete et al., 2014). On a study conducted on a relatively similar ore with the current study, and at leaching temperature of 200 °C, it was found that sulphuric acid leaching of Th was independent of acid concentration and remains constant above acid concentration of 12.2 M (El-Hussaini and Mahdy, 2002). A slight decrease in leaching extent with increasing sulphuric acid concentration was noted for U at similar conditions. The observations are in close agreement with our findings.
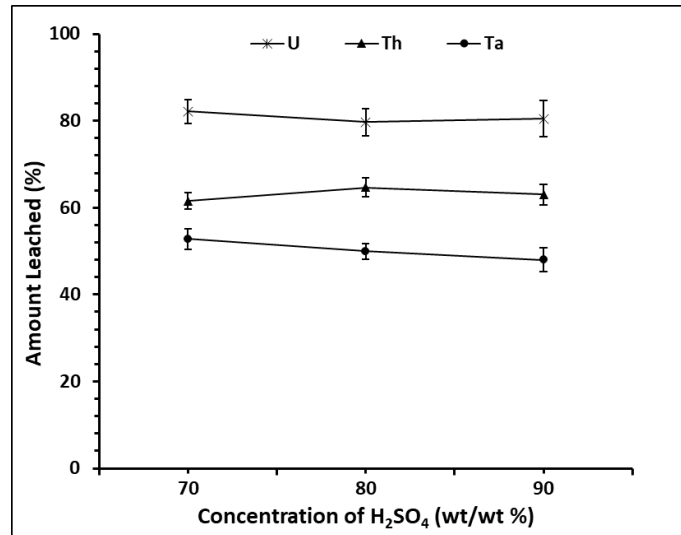

Fig. 6. The effect of sulfuric acid concentration (wt%) on leaching efficiency of U, Th and Ta at a leaching temperature of 200 °C, contact time of 2 hrs and at ore to acid ratio of 1:6 wt/wt
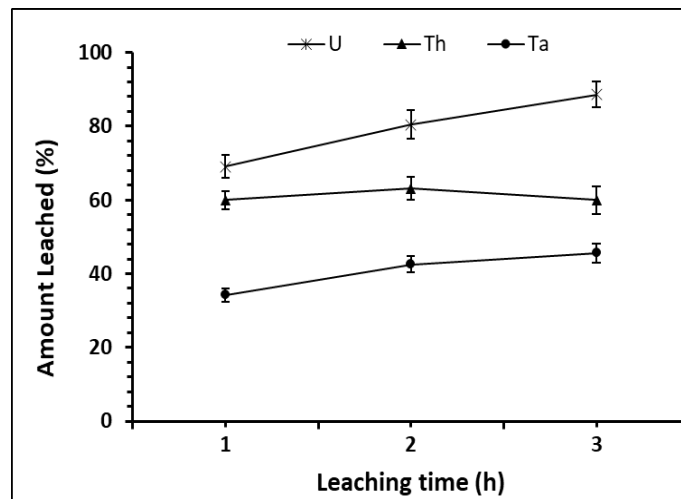

Fig. 7. The effect contact time on leaching efficiency of U, Th and Ta at a leaching temperature of 200 °C and 90 wt% sulphuric acid concentration

3.4. Effect of contact time on leaching of U, Th, Ta

Generally, longer leaching/digestion time may be considered as a favourable condition for the dissolution of U and Th from host ores because longer time provides better chance of reaction and hence, higher recovery (Habashi, 1997). However, some gangue minerals may also undergo dissolution at longer leaching times (Madakkaruppan et al., 2016). This may lead to competition for acid between the target and gangue minerals and consequently reduce dissolution of the economic elements. To ensure

sufficient acid was available at longer contact times, leaching was performed at 90 wt% sulphuric acid concentration in this study. As can be seen from Fig. 7, the dissolution rate of U, Th and Ta was found to be directly proportional to the contact time. The amount of U leached increased from 69.1% at contact time of 1 hr to 88.6% when the tantalite ore was leached for 3 hrs. In case of Th, for the leaching times considered, longer leaching time did not lead to increase in dissolution of Th. The insignificant change in leaching of Th with increasing leaching time is in agreement with a study reported in the literature for sulphuric acid digestion of rare earth bearing mineral (Sadri et al., 2017). At leaching time of 3 hr, 45.6% of the Ta also dissolved at these leaching conditions. In a related study, it was shown that the leaching of U and Ta increased from about 15% to 35% and 60% to 86%, respectively, when the contact time was increased from 1 hr to 3 hr (El-Hussaini and Mahdy, 2002). The leaching extent of Th varies only slightly under the same conditions.

3.5. Comparison of leaching trends of U and Th

A number of leaching tests were conducted at various temperature, contact times and sulfuric acid concentrations to compare the leaching behaviour of U and Th from tantalite ore. The amounts (%) of U and Th left undissolved after leaching were determined and the results were presented in Fig. 8 (a), (b) and (c) for contact times of 1 hr, 2 hrs and 3 hrs, respectively. As shown in Fig. 8, in the leaching conditions considered in this study, a clear difference was observed in the trend of leachability of U and Th with temperature. In general, the leaching efficiency of U increases with increasing temperature from 100 to 300 °C for all the test conditions except at H_2SO_4 concentration of 70 wt% (i.e., Fig. 8 (a) and (c)). In contrast, the leaching efficiency of Th decreases with increasing temperature from 100 to 300 °C for all the test conditions as evidenced by a higher amount of Th remaining undissolved. Some reasons have been put forward for the negative effect of temperature on dissolution of Th. First, at temperatures above 200 °C, Th forms insoluble species thus reducing its solubility (Gupta, 1990; Panda et al., 2014). The formation of insoluble compounds is also possible at higher temperatures since the residue may be completely dried during digestion (Hu and Qi, 2014). Another explanation is that when the acid digestion is conducted in the presence of air (this study), increasing the temperature increases sulfuric acid evaporation rate, and consequently decreases contact time between the acid and the solid sample, leading to reduced recovery (Sadri et al., 2017).

Among all the variables compared, the lowest U content in the residues was obtained at 70 wt% H_2SO_4 concentration (Fig. 8). Remarkably, the dissolution of U at 70 wt% H_2SO_4 concentration and 1 hr contact time was found to be >92% for the temperature range considered in the study (Fig. 8(a)). Comparatively, 70 wt% H_2SO_4 concentration resulted in Th dissolution of 70.8% (i.e., 0.19% Th left undissolved), which is the highest leaching efficiency achieved in this study as can be seen from Fig. 8.

3.6. Characterization of H_2SO_4 leached ore

Further investigation on the H_2SO_4 treated sample was conducted by using SEM coupled with EDS, and a typical micrograph is shown in Fig. 9. In comparison with the SEM image of the parent ore shown in Fig. 3, the acid treated sample appears to be porous and crumbled. This appearance is most likely due to U, Th, Ta and other species dissolving out from the surface of mineral components in the ore. It is also possible that some side reactions may lead to the precipitation of various species on the mineral surface. The elemental composition of the acid treated sample was determined using EDS by analysing the selected area shown in red in Fig. 9. The resulting spectra was shown in Fig. 9 with each peak assigned with the corresponding element. As expected, Ta was found to be the main element while the amounts of U and Th were considerably low. Table 2 is a representation of the spectra shown in Fig. 10 which confirms the relative abundance of each element in the treated ore. In comparison with sample 4 (Table 1), it can be seen from Table 2 that the Ta dissolution was only small (reduced from 48.69% to 46.11%). In contrast, the dissolution of U and Th is significant as the leached residue contains 0.2 wt% U and 0.31 wt% Th, in comparison with 1.23% and 0.65% (sample 4 in Table 1), respectively. It is important to note that the Mn obtained in Fig. 10 and Table 2 originated from the parent sample. This is to be expected because the ore sample has been found to be a predominantly mangano-tantalite ore (Berhe et al., 2018). The results are in close agreement with the XRF results shown in Fig. 8(b) for the same leaching conditions.
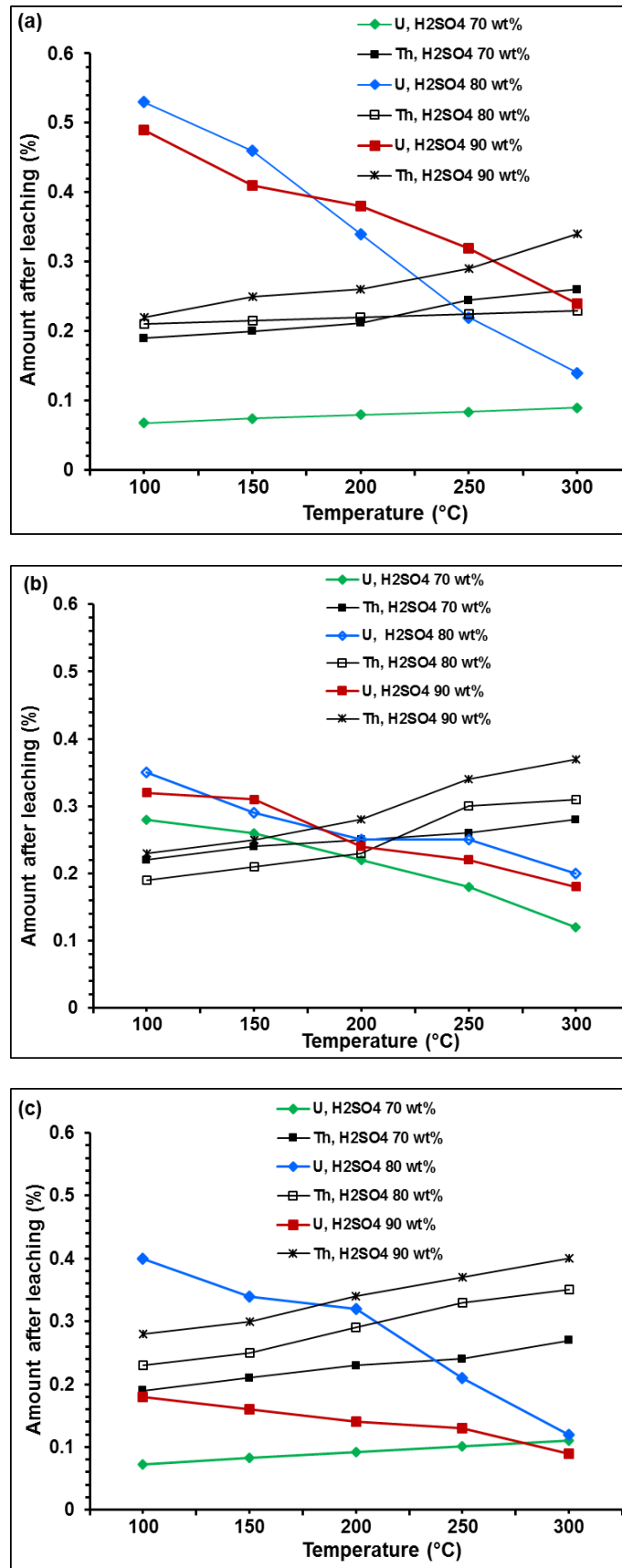
Fig. 8. Effects of temperature and H_2SO_4 concentration on the leaching of U and Th at various contact times, (a) 1 hr, (b) 2 hrs and (c) 3 hrs
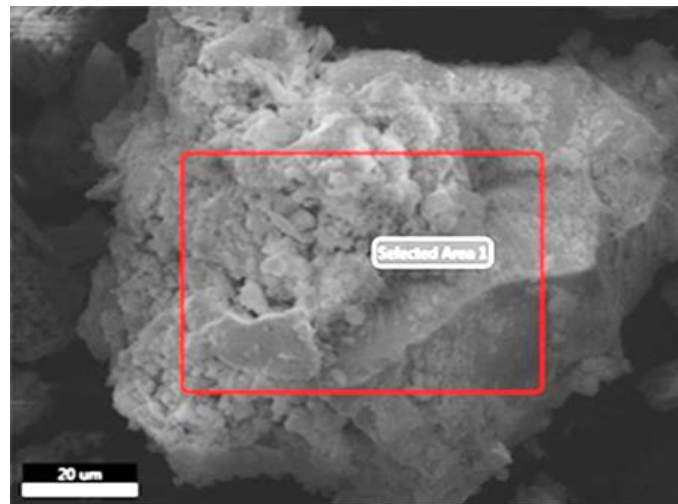
Fig. 9. SEM micrograph of sulfuric acid leached tantalite ore (sample 4 in Table 1) after digestion with 90 wt% sulphuric acid at 300 ºC, and 2 hrs of contact time
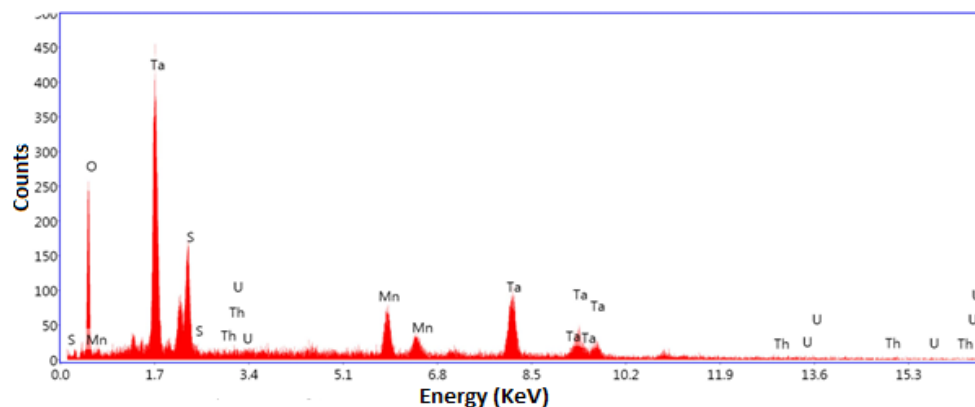

Fig. 10. Energy dispersive spectrum of H_2SO_4 treated tantalite sample (sample 4 in Table 1)

Table 2. Elemental composition of sulphuric acid treated sample determined by EDS

Elements	Weight %	Atomic %	Net Intensity	Error%	K ratio
O K	31.97	71.01	84.52	11.24	0.0967
S K	13.03	14.44	109.54	11.29	0.0646
Th M	0.31	0.05	1.80	70.98	0.0033
U M	0.20	0.03	1.32	72.56	0.0023
Mn K	8.38	5.42	65.16	7.41	0.0857
Ta L	46.11	9.05	107.33	9.06	0.3963

4. Conclusions

The leaching of uranium, thorium, and tantalum from a high grade columbite-tantalite ore collected from the Kenticha area, Southern Ethiopia was conducted using sulphuric acid. The ore contains relatively high amounts of radioactive elements such as U and Th accounting to up to 2 wt% total composition. In this study, the effects of concentration of sulfuric acid, temperature and leaching time on the removal of these radioactive oxides was studied. Increasing temperature from 100 to 300 °C results in increased leaching efficiency of U whereas the leaching efficiency of Th decreased with increasing temperature. The negative effect of temperature on leachability of Th from tantalite at higher temperature was attributed to the formation of insoluble species. It can be concluded that sufficient removal of U (>92 wt%) from tantalite was achieved under the experimental conditions used in this study although the highest removal of Th obtained was only 70.8 wt%. Further studies, particularly at lower leaching temperatures, is required to better understand the leaching behaviour of Th. It is

believed that the present study provides a stepping board for further studies on the removal of radioactive elements from Ethiopian pegmatite ore.

Acknowledgements

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