

Received June 13, 2017; reviewed; accepted September 11, 2017

## Decomposition of the Kenticha mangano-tantalite ore by HF/H<sub>2</sub>SO<sub>4</sub> and KOH fusion

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**Abstract:** In this study, the decomposition behavior of Ta and Nb from the mangano-tantalite ore was investigated using HF/H<sub>2</sub>SO<sub>4</sub> mixture and KOH fusion. The effects of reaction time, decomposition temperature, acid and alkaline concentrations, and particle size on the dissolution process were examined. Higher decomposition rates were achieved at 6:2 mol/dm<sup>3</sup> ratio of HF and H<sub>2</sub>SO<sub>4</sub>, and with the addition of 10 g KOH in the alkali fusion step. In addition, similar trends in decomposition rates between two agents (HF/H<sub>2</sub>SO<sub>4</sub> and KOH) and comparable dissolution performances were observed. The increase in the decomposition temperature initially increased the decomposition rate of mangano-tantalite for the two systems considered in this study. However, no significant change in the leaching rate of Ta and Nb was observed beyond 50 °C for the HF/H<sub>2</sub>SO<sub>4</sub> system and above 400 °C when using the KOH fusion process. The elemental and compositional analyses of the leached residues using XRF, XRD, and FT-IR spectroscopy indicated that the acid (mixture of HF and H<sub>2</sub>SO<sub>4</sub>) decomposition resulted in slightly better dissolution performance for Ta compared with the alkaline fusion method. Overall, the results indicated that KOH can be a suitable alternative decomposition agent to the volatile, corrosive and toxic HF in the hydrometallurgical processing of tantalite ores.

**Keywords:** decomposition, Kenticha pegmatite, mangano-tantalite, Ta, Nb

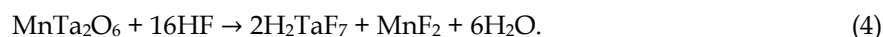
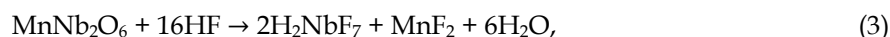
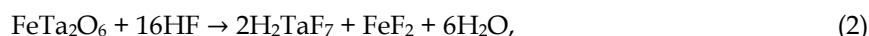
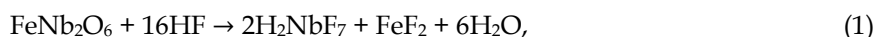
### 1. Introduction

Tantalum and niobium are widely used in many cutting edge technologies such as aerospace industry for jet engines and turbine parts, skin for spacecraft and missiles, rocket nozzle, and the modern electronics due to their good mechanical strength, ductility, and high melting points (Bose and Gupta, 2001; El-Hazek et al., 2012). The estimated annual global production of tantalum has increased from 765 Mg in 2012 to 1,200 Mg in 2014 (Jewell and Kimball, 2015). Most of identified resources of tantalum are found in Australia, Brazil, Canada, and Ethiopia amount to >150,000 Mg. Brazil and Canada are by far the world's leading niobium producers accounting for 99% of production in 2013 and 2014 (Jewell and Kimball, 2015).

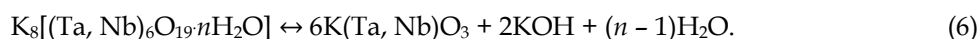
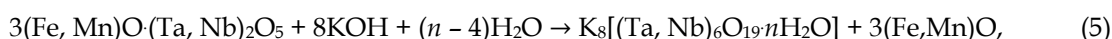
The most important tantalum containing minerals are generally columbite-tantalite, microlite, ixiolite, pegmatites and wodginit (Cerny and Ercit, 2005; Kuster et al., 2009). The Ethiopian Kenticha pegmatite is the site of current open pit tantalum mining operations, and is the main subject of this

study. The Kenticha pegmatites are characterized by quartz-feldspar-muscovite pegmatites of the beryl–columbite sub-type, containing tourmaline, beryl, Fe–columbite, and Mn–tantalite (Zerihun et al., 1995; Tadesse and Zerihun, 1996; Cerny and Ercit, 2005; Kuster et al., 2009; Kim et al., 2013). The Fe–columbite (composition of niobium > tantalum) and Mn–tantalite (composition of niobium < tantalum) are separated by the use of magnetic field and the resulting non-magnetic ore is mainly Mn–tantalite (Tadesse and Zerihun, 1996). Kenticha pegmatite contains SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MnO, CaO, Na<sub>2</sub>O, K<sub>2</sub>O, Li<sub>2</sub>O, TiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, and trace amounts of Ga, Be, Sn, Nb, Ta, Zr, Hf, Zn, Th, Li, and U (Kuster et al., 2009; Kim et al., 2013). In contrast to most equivalent rare-earth element pegmatites (e.g., Tanco, Canada and Greenbushes, Australia), the Kenticha pegmatite contains little or no cassiterite and pollucite (Zerihun et al., 1995; Kuster et al., 2009).

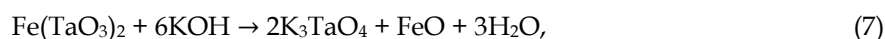
Following the beneficiation of the ore, a number of processes may be required based on the mineralogy and the chemical composition of the ore. The primary step in hydrometallurgical processes is to breakdown the constituents of the ore to solution. This may depend on the rate of the decomposition and leaching of the tantalite ore and/or the solubility of the solute to be extracted (Agulyansky, 2004; Zhu and Cheng, 2011; Nete et al., 2014). The decomposition of the concentrated Mn–tantalite may be carried out via several complicated procedures, such as alkali fusion, chlorination, reduction, and acid leaching. Currently, most of the niobium–tantalum ores are decomposed by concentrated hydrofluoric acid (HF) (Wang et al., 2009; Rodriguez et al., 2015). The dissolution of columbite–tantalite in HF can be represented by the following reactions (Gupta and Suri, 1993):



However, these decomposition processes are associated with the loss of HF due to volatilization (Agulyansky, 2004; Wang et al., 2009; Zhu and Cheng, 2011; Nete et al., 2014), which is toxic to plant operators and causes equipment corrosion. A number of studies have been reported in the literature with the aim of eliminating HF pollution at the source leading to cleaner Ta and Nb production processes (El-Hussaini and Mahdy, 2001; Wang et al., 2010). For example, the decomposition of niobium–tantalum ores with KOH sub-molten salt has been proposed as an alternative method with the objective of eliminating HF (Zhou et al., 2005; Zhu and Cheng, 2011). The decomposition reaction of niobium–tantalum ore with KOH has been suggested to proceed through the following steps (Zhou et al., 2005):



The K<sub>8</sub>[(Ta, Nb)<sub>6</sub>O<sub>19</sub>.nH<sub>2</sub>O] produced in the first stage (Eq. 5) is soluble while the K(Ta, Nb)O<sub>3</sub> produced in the second stage (Eq. 6) is insoluble, and whichever of these two species present in the leaching system is determined by the reaction conditions. The niobates and tantalates species produced during the alkaline decomposition of columbite–tantalite mineral are readily converted to more soluble species as shown below:



In light of the above reaction mechanism (Eq. 1 – 10), the aim of the present study was to compare the performance of both HF and KOH fusion methods on the decomposition efficiency of manganotantalite, and subsequent leaching of Ta and Nb in aqueous solutions. Although the use of HF on industrial scale is well established, the use of other alternatives such as KOH fusion on a larger scale is still scarce. Extensive research on the applicability of KOH to various Nb/Ta ores with different mineralogy is required to realize its use on industrial scale as well as for process optimization. In this

paper, the optimal decomposition and leaching conditions for Kenticha pegmatite ore using acidic (HF and H<sub>2</sub>SO<sub>4</sub>) and alkaline (KOH) at various decomposition time, temperature, and concentrations of alkaline and acid leaching agents were presented.

## 2. Experimental

### 2.1 Materials

The ore sample used in this study was obtained from the Kenticha processing plant in the regional state of Oromia, Ethiopia, currently mined by the Ethiopian Mineral Development Share Company (EMDSC). The mineralogy of the ore was investigated using a Shimadzu Maxima-XRD-7000 X-ray Diffractometer (XRD) with CuK $\alpha$  (1.540600Å) radiation. The XRD analysis revealed that the Kenticha pegmatite ore was dominated by a manganotantalite phase as seen in Fig. 1. An Energy Dispersive X-Ray Fluorescence (EDXRF 2800) was used for the elemental analysis of the ore. The sample was mainly composed of Ta<sub>2</sub>O<sub>5</sub>, Nb<sub>2</sub>O<sub>5</sub>, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and other radioactive impurities such as ThO<sub>2</sub> and U<sub>3</sub>O<sub>8</sub> (Table 1). A more detailed mineralogical analysis and suggested methods for the removal of radioactive oxides from the ore have been discussed elsewhere (Berhe et al., 2017). A Shimadzu FT-IR-8400S spectrophotometer was used to check the presence or absence the Nb/Ta oxygen bond frequency. The particle size distribution of the sample was determined using sieves (PAT) by dry screening in the size range of 38 – 74  $\mu$ m. The decomposition process was carried out in stainless batch reactor (SUS316) in a high temperature furnace at 400 °C. Analytical grade HF, H<sub>2</sub>SO<sub>4</sub> and KOH (Sigma-Aldrich) were used without further purification. De-ionized water (surface tension 72 mN m<sup>-1</sup> and resistivity of 18.2 M $\Omega$  cm) was used for leaching of the decomposed ore.

Table 1. Typical chemical composition of Kenticha pegmatite ore and beneficiated non-magnetic tantalite fraction

Ore (sample code)	Percentage, Wt%						
	Ta <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Nb <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	ThO <sub>2</sub>	U <sub>3</sub> O <sub>8</sub>
Pegmatite ore (1)	2.32	3.47	0.45	3.56	25.54	0.09	0.80
Tantalite concentrate (3)	55.71	0.67	5.51	0.36	4.31	0.03	0.68

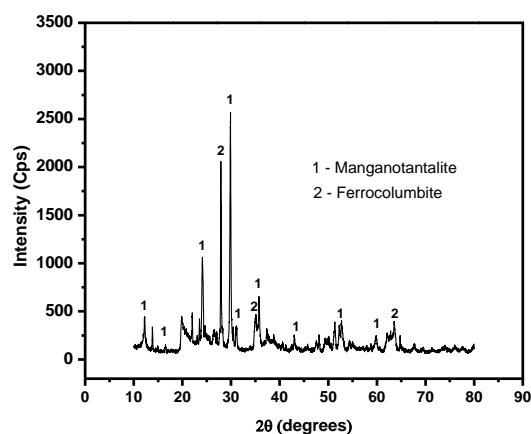


Fig. 1. XRD pattern of the Kenticha pegmatite ore (sample 1)

### 2.2 Methods

The Kenticha pegmatite ore (sample 1) was first crushed with a jaw crusher and then milled manually with a mortar and pestle until the desired particle size of < 74  $\mu$ m was obtained. This was followed by washing and decantation to reject filtrate. The residues were then treated by gravity separation using a shaking table to separate the heavy brown-black portion from the light fraction. Further beneficiation was conducted on the heavy portion of the sample using a dry magnetic barrier laboratory separator (Frantz, model LB1). The non-magnetic fraction was then taken for decomposition studies according to the scheme in Fig. 2. In order to conduct the acid digestion tests at various temperatures, acid and alkaline concentrations, and particle sizes, 2.5 g of non-magnetic

sample (sample 3) was used. The filtration was carried out following the acid digestion to obtain a residue (sample 4) and filtrate. The alkaline fusion steps were carried out in accordance with previously published procedure (Wang et al., 2010). The KOH was placed in ceramic crucibles which was then transferred to a batch reactor. When the desired temperature was reached, a mechanical stirrer was started, and 2.5 g of tantalite (sample 3) with a known particle size distribution was added. The mixture was then placed inside a furnace, heated to 400 °C and allowed to fuse for 80 min. The product was cooled to room temperature, and then leached with 100 cm<sup>3</sup> de-ionized water (resistivity of 18.2 MΩ cm). The filtrate and residue (sample 5) were obtained by filtration. All residues were compared by gravimetric, XRF, XRD, and FT-IR analyses.

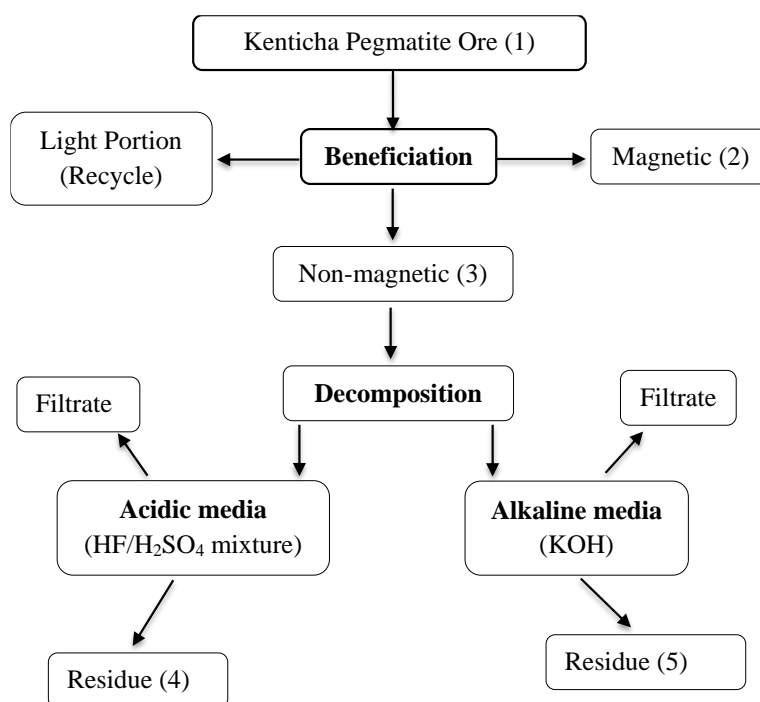


Fig. 2. Schematic diagram of a connected approach for the beneficiation and decomposition of Kenticha pegmatite ore

### 3. Results and discussion

#### 3.1 Beneficiation of the ore

The rate of leaching is affected by a number of factors such as particle size, the nature of the leaching agent, temperature, agitation time, and degree of acidity or alkalinity of leaching agents (Wang et al., 2009; Zhu and Cheng, 2011; Nete et al., 2014). However, prior to direct leaching repeated beneficiation of the pegmatite-spodumene ore is required to upgrade the tantalite portion (Cerny and Ercit, 2005; Kuster et al., 2009). In this study, gravity and magnetic separation methods were used to upgrade the ore and separate the magnetic and non-magnetic constituents from the ore. The distribution of magnetic and non-magnetic minerals within the heavy portion of the ore with respect to particle size is given in Fig. 3. The magnetic and non-magnetic mass fractions shown in Fig. 3 were obtained from a 10 kg sample according to the beneficiation scheme shown in Fig. 2. It can be seen from Fig. 3 that a higher magnetic fraction was recovered in the finer size fraction (<38 μm) while the non-magnetic portion slightly concentrated in the coarser size fraction (74 μm).

The XRD spectra (dominant phases) of the magnetic and non-magnetic components of the ore concentrated at different size fractions are shown in Fig. 4. As expected, the XRD spectra shows the magnetic ferrocolumbite as the dominant phase in the magnetic fraction (Fig. 4(a)) and the non-magnetic manganotantalite as the main phase in the non-magnetic fraction (Fig. 4(b)). The non-magnetic portion (manganotantalite ore) of the sample was used for further studies.

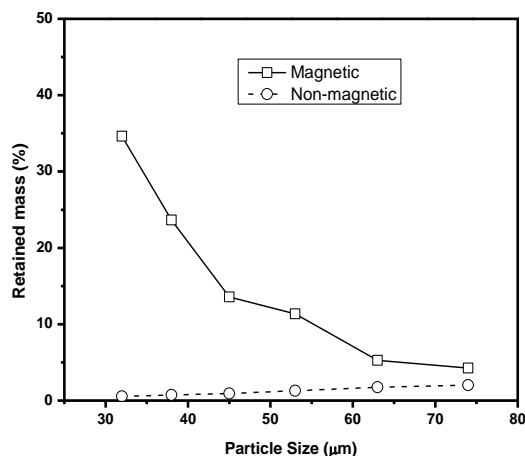


Fig. 3. Distribution of magnetic and non-magnetic mineral fractions with respect to particle size for the heavy portion of the Kenticha pegmatite ore after beneficiation

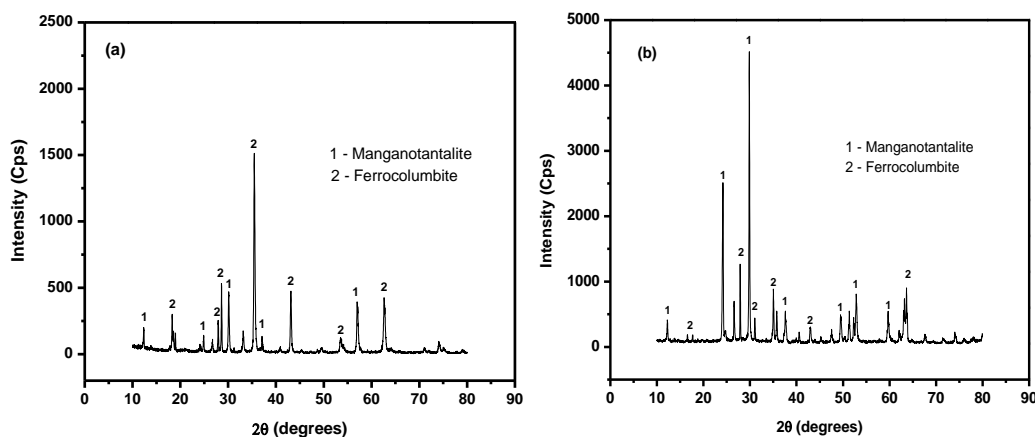


Fig. 4. XRD spectra of (a) magnetic (sample 2) and (b) non-magnetic (sample 3) fractions of the Kenticha pegmatite ore after magnetic separation

### 3.2 Effect of HF:H<sub>2</sub>SO<sub>4</sub> ratio and KOH concentration on decomposition of tantalite

The effect of HF:H<sub>2</sub>SO<sub>4</sub> ratio (mol/dm<sup>3</sup> H<sup>+</sup>) and KOH concentration on the decomposition behavior and water leaching of tantalite ore was carried out on the coarser particle size range. The decomposition studies were initially carried out at 400 °C and 80 min reaction time. In the high acid concentration with higher HF proportions, a higher decomposition efficiency was observed (Fig. 5(a)). Similarly, higher KOH concentration (10 g) lead to higher decomposition as can be seen from the smaller amount of residue in Fig. 5(b). In addition, similar trends in the decomposition rates and comparable dissolution performances between HF:H<sub>2</sub>SO<sub>4</sub> and KOH were observed. The remaining small amount of residues with the use of 6:2 H<sup>+</sup> ratio of HF and H<sub>2</sub>SO<sub>4</sub> and upon the addition of 10 g KOH indicated that the majority of the tantalite ore was effectively dissolved. Thus, 6:2 HF to H<sub>2</sub>SO<sub>4</sub> ratio and 10 g KOH were chosen for subsequent studies on the effect of reaction time on the decomposition rate of tantalite ore.

### 3.3 Effect of reaction time on decomposition of tantalite

Fig. 6 shows the effects of reaction time on the decomposition of tantalite using HF and H<sub>2</sub>SO<sub>4</sub> in 6:2 ratio and using alkaline fusion in 10 g KOH. It can be seen from Fig. 6 that the rate of decomposition increased with the increasing reaction time until a decomposition time of 80 min, after which it reached a steady state. Thus, the decomposition time of 80 min appears to be the most efficient condition for tantalite ore dissolution in both acidic and alkaline media. Higher decomposition rates were observed at the early stages of reaction when the HF and H<sub>2</sub>SO<sub>4</sub> system was used in comparison

with KOH fusion. However, it is important to note that reaction kinetics may also depend on particle size, alkali-to-ore ratio, temperature, and agitation rate.

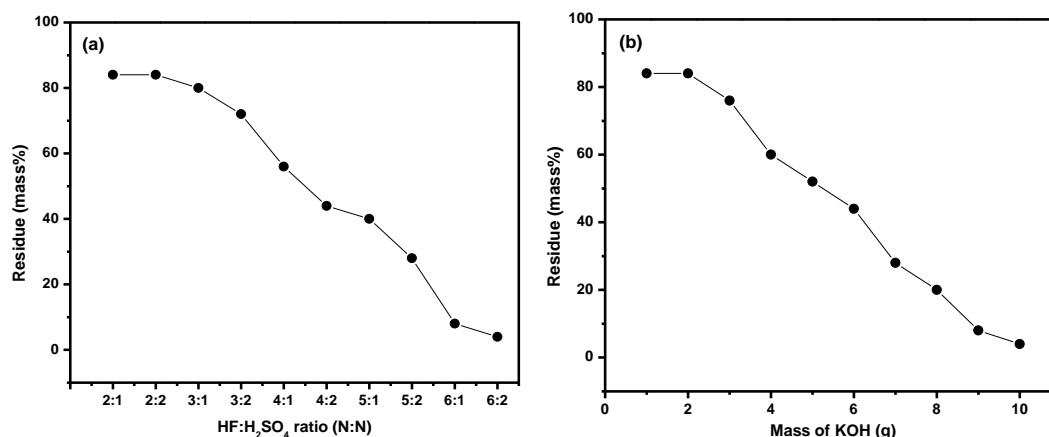


Fig. 5. Effect of acid and alkaline concentration on the decomposition of tantalite using a) HF:H<sub>2</sub>SO<sub>4</sub> and b) KOH

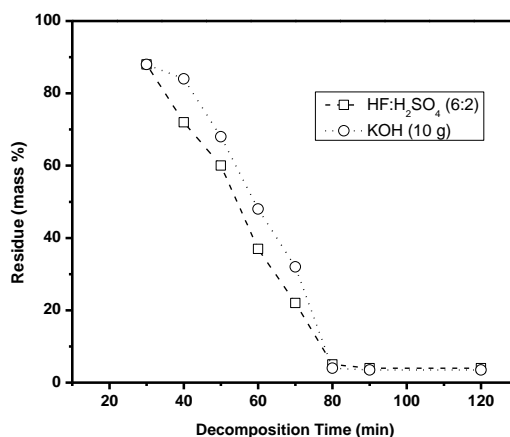


Fig. 6. Effect of reaction time on decomposition of tantalite using 6:2 ratio of HF to H<sub>2</sub>SO<sub>4</sub> and alkaline fusion in 10 g KOH

### 3.4 Effect of temperature on tantalite dissolution

The effect of temperature on the decomposition of tantalite was studied in the range of 20 - 90 °C for HF/H<sub>2</sub>SO<sub>4</sub> system and in the range of 200 - 500 °C using the KOH fusion method. A 2.5 g tantalite ore sample, -74+63 μm size particle, 6:2 ratio (mol/dm<sup>3</sup> H<sup>+</sup>) for HF/H<sub>2</sub>SO<sub>4</sub>, 10 g KOH, 300 rpm agitation speed, and 80 min reaction time were used at each of the decomposition temperature investigated. Figs 7(a) and (b) show that for both acidic and alkaline fusion systems, increasing the decomposition temperature initially increased the rate of dissolution of Ta and Nb. However, the decomposition temperature above 50 °C for HF/H<sub>2</sub>SO<sub>4</sub> system showed no significant changes in the dissolution of Ta and Nb (Fig. 7(a)). Similarly, no substantial change in % metal dissolution was observed upon increasing the temperature above 400 °C when using 10 g KOH as can be seen from Fig. 7(b). While no significant change in % dissolution was noted for Nb between the two systems, the % dissolution of Ta in the acidic leaching agent was higher than in alkaline fusion. The differences in the dissolution rates between Nb and Ta in KOH fusion (Fig. 7(b)) was attributed to the differences in reaction behaviors of Nb<sub>2</sub>O<sub>5</sub> and Ta<sub>2</sub>O<sub>5</sub> with KOH. Wang et al. (2009) reported that KOH fusion and water leaching of Nb<sub>2</sub>O<sub>5</sub> leads to high dissolution of Nb due to the conversion of Nb<sub>2</sub>O<sub>5</sub> to the soluble K<sub>3</sub>NbO<sub>4</sub>. By contrast, under similar conditions, large amount of residue was obtained upon KOH fusion and water leaching of Ta<sub>2</sub>O<sub>5</sub>. This relative insolubility of Ta<sub>2</sub>O<sub>5</sub> was attributed to the formation of the insoluble KTaO<sub>3</sub>. The formation of KTaO<sub>3</sub> during KOH fusion was confirmed in this study (Fig.

8(a)). Nevertheless, in the current experimental conditions, the decomposition of tantalite ore with KOH fusion at 400 °C and with HF/H<sub>2</sub>SO<sub>4</sub> mixture at 50 °C appeared to be effective.

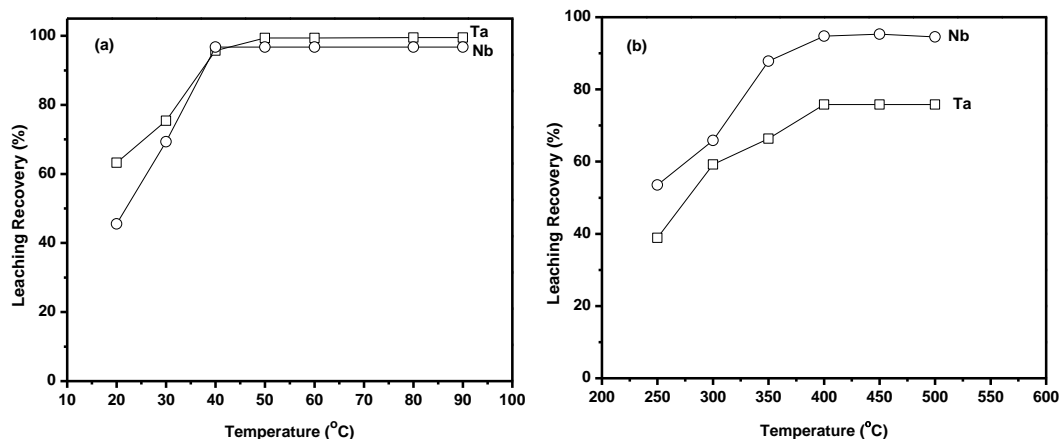


Fig. 7. Effect of decomposition temperature on the % dissolution of Nb and Ta (a) HF:H<sub>2</sub>SO<sub>4</sub> and (b) KOH

### 3.5 Characterization of ore and residues

The chemical composition of the magnetic and non-magnetic portions of the ore, and the residues leftover after the decomposition by acidic media and alkaline fusion followed by water leaching were determined using XRF, XRD, and FT-IR. Table 2 shows that the composition of the target metals (Nb and Ta) was higher in the water leached alkaline fusion residue as compared with the residue from acidic decomposition. These results indicated that the acidic leaching agent (mixture of HF and H<sub>2</sub>SO<sub>4</sub>) would be a better option for the dissolution of Nb and Ta from Mn-tantalite. The residue of alkaline fusion had higher amount of Ta (14 wt% of Ta<sub>2</sub>O<sub>5</sub>) suggesting that not all of the tantalite was decomposed by the alkaline fusion and water leaching.

Table 2. Elemental analysis of beneficiated ore, and residues after decomposition with HF and H<sub>2</sub>SO<sub>4</sub>, and KOH

Sample (code)	Wt%						
	Ta <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Nb <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	ThO <sub>2</sub>	U <sub>3</sub> O <sub>8</sub>
Magnetic (2)	6.25	0.41	10.15	5.33	80.23	0.06	0.10
Non-magnetic (3)	55.71	0.67	5.51	0.36	4.31	0.03	0.68
Acidic residue (4)	0.23	15.56	0.01	Nd	Nd	Nd	Nd
Alkaline residue (5)	14.20	15.61	0.35	Nd	0.22	0.01	Nd

Nd - Not Detected.

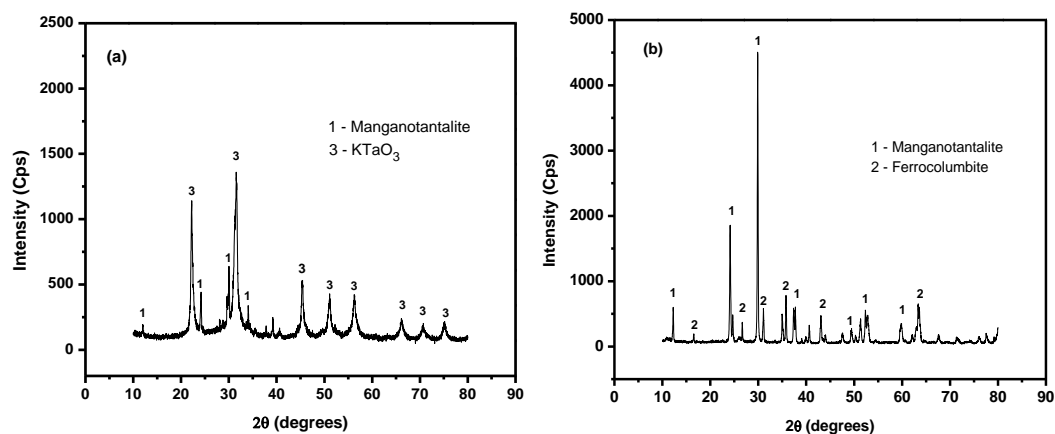


Fig. 8. XRD spectra of leaching residues from (a) KOH fusion (sample 5) and (b) HF:H<sub>2</sub>SO<sub>4</sub> decomposition (sample 4)

The XRD spectra of the residues after the alkaline fusion (sample 5) and acidic decomposition (sample 4) are shown in Fig. 8(a) and (b), respectively. It can be seen from Fig. 8 that crystalline phases in the residues are different from the original ore shown in Fig. 1 and the non-magnetic tantalite fraction shown in Fig. 4(b). The main phase in the residues was attributed to  $\text{KTaO}_3$  instead of  $\text{KNbO}_3$ . This observation is in agreement with previous studies published on the alkaline fusion related tantalite ore decomposition processes (Wang et al., 2009; Wang et al., 2010).

The observations from the XRF and XRD analyses were also supported by FT-IR stretching frequencies in the range of  $1665$  to  $655\text{ cm}^{-1}$  for the residues (Table 3) which implies differences in structure of metal oxide bonds between both residues and the tantalite ore. As indicated in Table 2, only  $0.23\text{ wt}\%$   $\text{Ta}_2\text{O}_5$  was detected in the residue after the acid leaching (sample 4). Thus, the absence of stretching spectra at  $721\text{ cm}^{-1}$  for the acid leached residue as compared with that of the tantalite ore (with  $55.71\text{ wt}\%$   $\text{Ta}_2\text{O}_5$ ) confirmed the very low  $\text{wt}\%$  of  $\text{Ta}_2\text{O}_5$  in the acid leached residue. The  $721\text{ cm}^{-1}$  stretching frequency corresponds to Ta-O bond in samples of pegmatite ore, tantalite ore and alkaline leached residue.

Table 3. FT-IR analysis of pegmatite ore, tantalite concentrate, and water leached residues collected following acidic decomposition and alkaline fusion

Sample (code)	Frequency, $\text{cm}^{-1}$
Pegmatite ore (1)	3520 (vs); 2954 (s); 1774 (s); 1665(s); 1454 (vs); 732(vs); 657(vs), 555(w).
Tantalite (3)	3520 (vs); 2954 (s); 1774 (s); 1665(s); 1454 (s); 721(vs); 655(vs); 555(vs)
Acidic residue (4)	3520 (vs); 2954 (s); 1774 (s); 1685(s); 1454 (s); 721(vs); 655(w); 555(vs).
Alkaline residue (5)	3520 (w/0); 2954 (s); 1774 (s); 1685(s); 1465(s); Nd; 655(w); 555(vs).

From the XRF, XRD, and FT-IR analyses, it can be concluded that the residues from the acidic decomposition and alkaline fusion routes were chemically different from each other, and thus each system follows a characteristic dissolution reaction.

#### 4. Conclusions

In this study, the performance of  $\text{HF}/\text{H}_2\text{SO}_4$  mixture and  $\text{KOH}$  fusion as decomposition agents for tantalite ore was investigated. The decomposition step was followed by water leaching to study the rate of dissolution of Ta and Nb. The dissolution rate was found to depend on the proportion of  $\text{HF}$  and  $\text{H}_2\text{SO}_4$ , the concentration  $\text{KOH}$ , decomposition time, and temperature. The non-magnetic tantalite fraction appears to be slightly concentrated in the coarse particle size range. Higher ratio of  $\text{HF}$  to  $\text{H}_2\text{SO}_4$  in the acidic system and higher  $\text{KOH}$  concentration during the alkaline fusion step lead to smaller amounts of residues leftover after decomposition. The elemental and compositional analyses of the residues after leaching by water suggested that the acidic decomposition approach resulted in better dissolution of Nb and Ta metal ions compared with the alkaline fusion approach. The use of 6:2 ratio of  $\text{HF}$  and  $\text{H}_2\text{SO}_4$  at  $50\text{ }^\circ\text{C}$  and  $10\text{ g}$   $\text{KOH}$  addition in the alkali fusion step at  $400\text{ }^\circ\text{C}$  resulted in better decomposition rates. These results indicate that  $\text{KOH}$  fusion has the potential to serve as a suitable alternative decomposition agent to the commonly used toxic and corrosive  $\text{HF}$  in the hydrometallurgical processing of tantalite ores.

#### Acknowledgments

The authors would like to thank the Geological Survey of Ethiopia, the Ethiopia Ministry of Mines, Petroleum and Natural Gas, the Ethiopian Mineral Petroleum and Biofuel Corporation, and Adama Science and Technology University for allowing them to use their analytical and research facilities. The financial support from Jimma University (grant number MEGA project 2008 JIT), and the Norwegian Agency for Development Cooperation and Mekelle University is gratefully acknowledged.

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