Mini Review

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Green extraction of niobium and tantalum for Ethiopian kenticha ores by hydrometallurgy process: a review

Abstract

This review presents an overview of the currently mined tantalite ores in Ethiopia (in particular Kenticha ores) and potentialities to extract niobium (Nb) and tantalum (Ta) products using green technologies. The foremost source of niobium and tantalum is the columbite-tantalite mineral or "coltan". Since hydrometallurgical methods are most commonly used to recover these metals from raw materials, Solvent Extraction (SX) processes have been used for producing pure niobium and tantalum products. All commercialized "SX" processes are exclusively conducted in the presence of fluoride ions, most frequently in a mixture with a mineral acid such as sulphuric or hydrochloric acid. Due to increasingly stringent regulations concerning the protection of human health and environment, there is an urgent need to develop novel aqueous and organic systems to reduce or eliminate the use of harmful fluorides. Because the Kenticha pegmatite spodumene has an excess percent of uranium, certain markets stopped importations of Ethiopian coltan. So, investigations are required to indentify aqueous complex systems and solvent extraction systems to enable the purification of niobium and tantalum without the use of fluorides. This paper summarizes the common dissolution and extraction agents used and suggest some "friendly" alternative lixiviant to be used for the extraction of Ta and Nb products from Kenticha tantalite ores.

Keywords: tantalite, kenticha, solvent extraction, coltan, lixiviant

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Introduction

Like other economical metals, tantalum (Ta) and niobium (Nb) are broadly used in electronic and other high-tech industries.[1–4] Ta-Nb concentrates are mainly composed of columbotabatlite (Fe,Mn)(Ta,Nb)$_2$O$_6$ and other classes of mineral.[1–7] Ethiopia has world class tantalite deposits, which are found only in some countries of the world such as Australia, Brazil, Canada and Mozambique. In Ethiopia, specifically at Kenticha, Adola area, Southern Ethiopia, there is rare-element pegmatite which represents a globally important tantalum source. Ethiopia has been mining and exporting tantalite via Ethiopian Mineral Development Share Company (EMDSC) about 100-120 T of Ta_2O_5 per year, equivalent concentrate containing 50-60 wt % Ta_2O_5 to the metallurgical industries, which are providers Nb and Ta for 60 wt % production of capacitors.[8] The extraction of these metals from mineral ores has been significantly studied[5–35] This extraction process may begin by physical beneficiation such as sizing, gravity, floatation and magnetic separation followed by chemical breakdown or decomposition or leaching of the ore.[2–4]

The bases for decomposition and separations processes are breakdown of the ore followed by solvent extraction (SX).[1–7] The technique also for higher and powerful separation.[1–7] Before Sx to decompose the ore he binary acid system of H_2SO_4-HF is normally applied to leach niobium and tantalum from their raw materials. The presence of HF in the leach solution also plays an important role in the extraction of Nb and T and will be better if salting agent is added (eg. H_2SO_4) a using various extractants.[1–3] However, due to the strong volatility (about 6-7%), the HFis lost during the decomposition process, which is harmful to human beings and causes equipment corrosion. As well, a large amount of wastewater containing fluoride is generated which needs to be treated.[2] More importantly, this method is only appropriate for high-grade niobium-tantalum ores, and it is difficult to decompose low-grade ores by hydrofluoric acid.[1,2] New process for the leaching of low-grade ores with KOH to eliminate fluorine pollution at the decomposition,[1,2] was proposed. However, a large amount of KOH solution is required to be evaporated and recycled in this process, which is very energy intensive and high reaction temperature is required.[1,2] To avoid this disadvantage, a hydrothermal method has been studied and ores can also be processed using chlorination, fusion with ammonium fluoride and bifluoride, direct acid dissolution with H_2SO_4, or a combination of H_2SO_4 and HF.[7,8] Another method is being investigated, but not still validated, to remove the use of hydrofluoric acid as dissolvent of tantalite using alkali and fluoride salts and replacing the petroleum derived solvents, what could be a new "environmental friendly" solvent using ionic liquids for low and high grade ores and a minimizer of wastes production by process integration and added value sub-products recovery.[10–12] The aim of this paper is to review recent trends and potentials for extraction of Nb and Ta from Ethiopian Kenticha pegmatite ore using green processes.

Economic minerals of Nb and Ta

The foremost source of niobium and tantalum is the columbite-tantalite mineral or "coltan", pyrochlore.[7–9] Microlite, ixiolite, wodginite). High grade Ta ore concentrates typically have 10% and 60% Ta_2O_5.[8–11] Granites and pegmatites of the enriched in lithium, cesium, tantalum family or which have the total molecular of C_a O, Na_2O and K_2O could be less than molecular Al_2O_3 and Ta>Nb.[9] Many of the largest tantalum deposits occur in pegmatite swarms and nd.[9–11] All types can contain a range of tantalum minerals Table 1.[11]

Ethiopian Kentichapeg matites are succeeded by quartz-feldspar-muscovite pegmatites of the beryl-columbite subtype, containing black tourmaline, greenish and bluish beryl, Fe-columbite (magnetic) and Mn-tantalite/tantalite (nonmagnetic).[11] Kenticha pegmatite contains major and trace element geochemical data such as; SiO_2, Al_2O_3, Fe_2O_3, MnO, CaO, Na_2O, K_2O, Li_2O, TiO_2, $P_2O_5$and in ppm Ga, Be, Sn, Nb, Ta, Zr, Hf, Zn, Th, Li, U.[11–17] In contrast to most equivalent rare-element pegmatites (e.g., Tanco, Canada or Greenbushes, Australia), the Kenticha pegmatite rarely carries cassiterite and has no known pollucite.[11,14–17] This pegmatite is the site of the open pit, tantalum mining operations and the main subject of this paper Table 2.

The Kenticha tantalum deposit currently mined and exported by EMPBC with 70 T/year. The probable reserve of primary ore is 2400 T at a grade of 0.015% Ta_2O_5 from 1:1 to up to 3:1 of Ta and Nb from LZ to UZ in the spodumene unit which is the focus of the present mining and for future green extraction of Ta.[7,11,13–17] This ratio reflects the Ta mineralization potential and it is highest in the spodumene unit Table 3 & Table 4.

Table 1 Selected minerals of Nb_2O_5& Ta_2O_5 and availability (wt%, based on BGS, 2011).[9]

Mineral Group	Formula	Nb_2O_5	Ta_2O_5
Columbite-tantalite	$(Fe,Mn)(Nb,Ta)_2O_6$	78.72	n.a
Columbite-tantalite	$(Fe,Mn)(Ta,Nb)_2O_6$	n.a	86.17
Pyrochlore	$(Na,Ca)_2Nb_2O_6(O,OH,F)$	75.12	n.a
Pyrochlore	$(Na,Ca)_2Ta_2O_6(O,OH,F)$	n.a	83.53
Tapiolite	$(Fe,Mn)(Ta,Nb)_2O_6$	1.33	83.96
Ixiolite	$(Ta,Nb,Sn,Mn,Fe)_4O_8$	8.3	68.96
Wodginite	$(Ta,Nb,Sn,Mn,Fe)_2$	8.37	69.58
Provskite	$(Ce,La,Na,Ca,Sr)(Ti,Nb)_2O_8$	16.15	n.a
Provskite	$NaNbO_3$	81.09	n.a
Euxenite	$(Y,CA,Ce,U,Th)(Nb,Ti,Ta)_2O_6$	47.43	22.53
Rutile	$(Ti,Ta,Fe)O_2$	11.32	37.65
Rutile	$Fex(Nb.Ta)_2xti1\text{-}xO_2$	27.9	n.a

Table 2 Internal structure and mineral assemblages of Kenticha pegmatite odified by Küster et al.[15]

Zone	Major minerals	Ta ore Minerals
UZ	Albite-quartz (lepidolite, zinnwaldite)-spodumene-muscovitemicrocline-pegmatite, quartz-feldspar with large spodumene (Figure 1 & Figure 2) and quartz crystals	Mn-tantalite, ixiolite/wodginite Ta>Nb
IZ	Muscovite-quartz-albitemicrocline Pegmatite	Fe-columbite, Mn-columbiteNb>Ta
LZ	"Alaskitic" muscovite-albite granite to layered albite-quartz aplite	ColumbiteNb>Ta

Table 3 Mineralogical analysis of tantalum ore samples from the Kenticha deposit high concentration lots (wt %). Source: EMDSC, Feb. 2016

Lot Number	Ta_2O_5	Nb_2O_5	U_3O_8	TiO_2	SnO_2	ThO_2	Sb (ppm)
36	69.71	7.21	0.3	0.25	0.085	0.026	<20
52	67.42	10.84	0.56	0.28	0.21	0.088	<20
37	62.98	11.61	0.61	0.32	0.09	<0.005	<20
53	61.57	12.95	0.86	0.39	0.32	0.086	<20
39	60.63	9.81	0.56	0.52	0.07	0.015	<20
54	60.23	12.55	1.19	0.41	0.34	0.091	<0.001
32	60.13	15.19	0.55	0.24	0.09	0.025	<20
33	59.23	17.38	0.8	0.15	0.081	0.017	<20
35	58.93	9.06	0.42	0.42	0.079	0.024	<20
29	57.76	13.93	0.67	0.64	0.037	<0.01	<20

Table 4 Mineralogical analysis of tantalum ore samples from the Kenticha deposits low concentration lots (wt %). Source: EMDSC, Feb. 2016.

Lot Number	Ta_2O_5	Nb_2O_5	U_3O_8	TiO_2	SnO_2	ThO_2	Sb (ppm)
67	34.89	10.56	0.44	2.53	0.45	0.068	<20
80	34.86	21.87	0,63	1.58	0.15	0..220	0.17
68	34.83	13.32	0.9	0.9	0.69	0.019	<20
70	33.75	21.34	0.77	0.43	0.61	0.049	30
81	33.27	24.24	0.4	0.85	0.11	<0.02	<20

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Table Continued...

Lot Number	Ta_2O_5	Nb_2O_5	U_3O_8	TiO_2	SnO_2	ThO_2	Sb (ppm)
71	33.01	26.33	0.52	0.39	0.63	0.015	<20
76	32.07	18.92	0.48	5.17	0.13	0.034	<0.002
72	31.8	23.03	0.85	0.3	0.63	0.044	<20
83	30.87	26.72	0.43	1.31	0.1	0.2	<20
82	26.33	31.01	0.26	1.24	0.07	0.023	<20

As can be seen from, the Ta_2O_5 content in highly concentrated zones can be as high as 70 wt % while the Ta_2O_5 content in other zones may be as low as 26 wt %. On the other hand, the composition of Nb_2O_5 in the low Ta_2O_5 concentration zones may be up to 31% although Nb_2O_5 content as low as 7% have been reported. It is also worthwhile noted the amounts of U and Dioxides in the ore as these are the major penalty elements. In the Kenticha pegmatite certain parts of the ore do carry low and above the critical level of 0.5 wt % U and lithium is above 1.64 wt % in the spodumene. Certain markets (e.g. Europe Union members) does not allow import of coltan with uranium contents above 0.5 wt % whereas other markets e.g. China allows higher uranium contents.[16,17] To date production can be increased, stored and recycled large tailings dam hosting the remaining 30% of the columbite from the ore. The EMPBC now seeks partners to develop a beneficiation plant to produce value added commodities (K_2TaF_7, K_2NbF_7, Ta_2O_5, Nb_2O_5, U_3O_8, etc.) as well as Li Figure 1.[13–17]

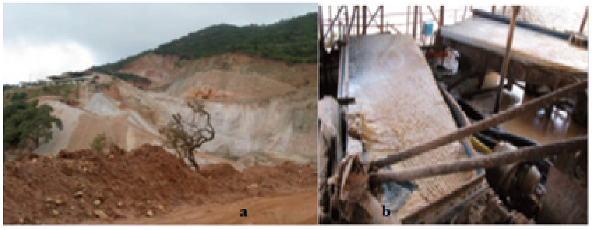

Figure 1 a) Tantalum ore body and b) Shaking tables producing tantalite concentrate.[13–17]

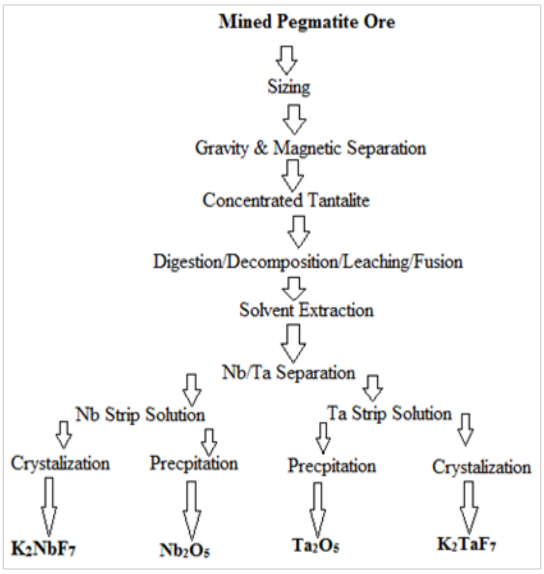

Figure 2 Scheme 1.Conceptual flowchart of a commercial process to produce pure Nb and Ta products (based on Zhaowu and, Chu, 2011).[6]

This review outlines some beneficiation approaches before exporting, alternative methods of separation of Nb, mechanisms of production of oxides of Nb and Ta with reduced uranium and further options for refining into niobium and tantalum metals.

Mineral processing of tantalite ore

A large number of chemical treatment procedures for the breakdown of primary sources have been studied and all these processes can essentially be divided into reduction to metallic or compound form via chlorination, alkaline fusion or acid dissolution (leaching).[5] According to a recent review, the success beneficiation of tantalum and niobium ores normally depends on the physiochemical properties of the ore, such as the presence of radioactive materials, its response to a magnetic field, the Ta and Nb contents, and the nature of the ore. The beneficiation process of an ore usually starts with an enrichment step, which may involve gravity and magnetic separation steps. And the presence of radioactive elements such as thorium and uranium in tantalite complicates the transportation, handling, and processing of these minerals. These radioactive materials are removed using acid leaching.[1–6] There is none to limited research on the beneficiation and acid leaching of Ethiopian-Kenticha tantalite ore. The Kenticha tantalite ore have higher concentrations of Li and U and other oxides of alkaline, transition and rare earth elements (REE).Thus, the beneficiation of this ore needs new design for better enrichment and the production of high quality oxides and metallic forms of Nb and Ta.[19] 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 most common process in hydrometallurgical processes is to dissolve (digestion or decomposition or solid-liquid extraction) the constituents of the ore to form a 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. Obviously, the better the leaching, the higher the quantity of tantalum and niobium to be extracted. The difficulty of Ta and Nb extraction from the ore is due to the fact that only a few solvents can leach the ore and mostly in the presence of the fluorine ion.[5,7,20] The process of leaching or decomposition of or may be used to produce dissolved metal ions of valuable solid materials and to decrease an insoluble solid or considered as residue. This rate of leaching is affected by a number of other factors such as; particle size, the nature of solvent, temperature and agitation.[21]

The decomposition of a tantalite ore involves several complicated procedures, such as alkali fusion, chlorination and acid leaching. Alkali fusion is one of the first methods that was industrially adopted to achieve simultaneous breakdown of columbite-tantalite.[1–5] Alkaline fusion based processing routes have certain advantages over the HF-based decomposition processes. For instance, the HF is not needed for decomposition of the raw materials, and the amount of fluorine would be calculated or adjusted by alkaline salts or hydroxides and ammonium biflouride. Since the leaching is performed with water, a significant fraction of the impurities can be precipitated in the form of insoluble compounds which can then be separated by filtration. The most important aspects of alkaline fusion are that the solution has a low acidity level, eliminate the need of HF and such a solution can be treated by liquid-liquid extraction (SX) using selective methods after an appropriate acidity adjustment.[5,6] As the Kenticha tantalite ore is composed of various metallic oxides Table 4, it is expected that the decomposition rate may vary significantly with the location of the mined zone. It is of interest to investigate reasonably stable and efficient potential leaching solvents with minimum environmental footprints. During SX process an organic solution is contacted with the filtrate produced from the leaching process by checking a number of criteria such as, selectivity, stability, fire hazard, cost and solubility in water (Hussaini and, 2001; Krismer and Hoppe, 1984). Only methyl is-butyl ketone (MIBK), tri-butyl phosphate (TBP), cyclohexanone (CHN) and 2-Octanol (OCL) are widely used industrially. Many other extractants such as high molecular weight amines and long chain alcohols have also been studied extensively.[1–6,23–26] An parameters fate extract ant a number of parameters must be taken into consideration apropriently such as pH, type of dissolved metal ions , concentration, ionic strength, presence, solubility, flash point, density, etc. According to Zhaowu and Chu, 2011; Olushola et al. 2011; Krismer and Hoppe, 1984, the main properties of the four commercialized extract ants are compared in.[5,6,23] It should be noted that MIBK is very stable and its cost is much lower than TBP. However; one significant problem with SX is that no solvent is completely insoluble in another solvent. In practice, one additional step is usually carried out before recycling the extractant solvent. Hydrometallurgical processes are successful in the production of pure niobium and tantalum, but have serious disadvantages.[24–38]

These include

I. Type and quantity of impurities in solution,
II. Expensive regeneration of fluorine and the substantial loss of fluorine quantities in these processes.
III. Generation of large amounts of residue,
IV. The permanent loss of reagents, and
V. The environmental and hazardous nature of HF as a reagent.

Most of the previous investigations lack a clear understanding of the mechanism and basic chemistry involved in the extraction process as well as the properties which allow for the separation of the two metals Figure 2.

Conclusion

Tantalum is a strategic metal due to its use in one of the most widespread gadgets in modern society, the mobile phone. Tantalum is also used in other types of electronic equipment and is thus in high demand. At present, Ethiopia supplies close to ten percent of the world consumption of tantalum and has a good potential for a considerable expansion of the production. The Kenticha tantalum deposit in Southern Ethiopia, the main focus of this review, has Ta/Nb ratio of up to 3:1 which makes the deposit economically attractive. Today the Ethiopian Mineral Development Share Company produces tantalite concentrate and sells the product on the international market. The rather high uranium and lithium contents of some of the Kenticha ore pose a problem since not all countries allow import of ore with significant amounts of uranium and lithium. Thus, urgent solutions are sought in order to upgrade the concentrate into a saleable final product. The first step is to produce a uranium-free product which can be sold to the European and North American markets. The next step is to produce tantalum and niobium compounds such as K_2TaF_7 and K_2NbF_7, and the final step will be to produce Ta_2O_5 and Nb_2O_5 powder. This will significantly increase the earnings. The primary beneficiation of tantalite using magnetic separation and acid leaching procedures also results in the successful removal of reasonable amounts of Fe, Ti, U from tantalite ores and thereby reducing the levels of impurities present in the tantalite mineral.

The extraction and separation of niobium and tantalum by SX has proven to be simple, express and very competent and largely applied in the purification processes in chemical and metallurgical industries and it likewise provides selective extraction and recovery of Nb and Ta from aqueous solutions. This present review also shows that the extraction and separation of niobium and tantalum from their ores involves the breakdown treatment of the source, extraction and separation by varying experimental conditions, precipitation, filtration, washing, drying and calcinations. Other techniques such as gravity, magnetic and electrostatic separation techniques may be coupled as adjunct to obtain a purer niobium and tantalum. Future development of hydrometallurgical processes of Nb and Ta will consider the omission of leaching by the use of HF and replacement of traditional organic solvents by environmental friendly or "Green Solvents".

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Conflict of interest

There is no conflict of interest in this work.

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