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Green extraction of Niobium and Tantalum from Kenticha Tantalite ore using 1-Ethyl-3-Methyl Imidazolium Chloride ionic liquid

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Abstract

The decomposition of the Ethiopian Kenticha tantalite ore was studied using the KOH fusion and H_2O leaching system at 400 ^oC for a 1 h reaction time. The experimental results obtained showed that 27 wt% of Ta₂O₅ was mainly transformed into insoluble KTaO₃, not to be KNbO₃, and as well, into the dissolved liquor containing 94.73 vol% of Nb₂O₅ and 75.80 vol% of Ta₂O₅. The observations, supported by the EDXRF, XRD, and ICP-OES techniques, showed that the ratio of the dissolved mixture of Nb and Ta metal ions was beneficial to the solvent extraction from both Ta₂O₅ and Nb₂O₅. Niobium and tantalum were extracted from their neutralized alkaline dissolved and hydrolyzed solution by a new process using the EMIC/AlCl₃ ionic liquid as the extractant using selective stripping, precipitation, and calcination of a highly crystalline pure 99.84 wt% of Nb₂O₅ and amophours 90.81 wt% of Ta₂O₅. The compositions of the dissolved metal ions and solids were analyzed by the ICP-OES, EDXRF, XRD, and FT-IR techniques.

Keywords: Decomposition, Ionic Liquid, Kenticha Tantalite Ore Mine.

1. Introduction

Niobium (Nb) and tantalum (Ta) are two group VB transition metals that have attracted a high demand in the recent years due to their applications in many industries [1-4]. These critical and economic elements occur together in leading types of ores such as "calton". manganocolumbite, ferrocolumbite, and pyrochlore. The chemical separation of these two metals from their ores and each other is usually challenging but feasible [4-7]. The decomposition of the host ore is the key step in the extraction of Nb and Ta from their target ore. Currently, the major hydrometallurgical process used to produce Ta and Nb involves the use of fluorinated media and a mineral acid [1-4, 8, 9]. However, the fluoride solutions (F) are highly poisonous, and a large amount of sewage is released from the processing plants, which pose significant

environmental problems due to the high volatility (up to 7 vol%) of these wastes [4, 10-14] In particular, this method is appropriate for highgrade tantalite ores, and it is difficult to decompose low-grade ores by hydrofluoric acid [2, 4, 11, 12]. Therefore, it is imperative to develop alternative methods and green lixiviants for the production of purer target metals [15, 16]. Other alternative processes with lower environmental impacts compared with the fluoride solutions have recently gained an increasing attention for the production of Nb and Ta from low-grade tantalite ores [7, 11, 12, 17]. For instance, Zhou et al., 2005 have proposed the use of KOH sub-molten salt for leaching low-grade refractory niobium-tantalum ores.

Assortments of organic solvents have been extensively studied in the recent years in SX of Ta

and Nb-containing liquors. Examples include methyl iso-butyl ketone, tri-butyl phosphate, cyclohexanone, 2-octanol, and high molecular weight amines. Comparatively, due to its solubility and stability, the former organic extractant is the most commonly used worldwide [4, 11, 12].

Ionic liquids (ILs) or the so-called "green solvents" can be considered as suitable alternatives to fluoride solutions as they possess a very low vapor pressure, versatility, and high capacity for separating organic compounds and metal ions from aqueous phases. The potential benefits of ILs in the recovery of metal ions have been summarized in a number of reviews elsewhere [11, 12, 18-25].

studies Further on the separation and determination of Nb and Ta are required regarding efficiency, selectivity, and sensitivity. The intention of this work is to demonstrate the feasibility of using 1-ethyl-3-methyl imidazolium chloride (EMIC) as an extracting phase. Metal extraction was performed by varying acid concentrations with a constant mass of EMIC in the presence of the Lewis acid AlCl₃ in the KOH feed solution followed by separation and purification of Nb and Ta from other metal ion impurities in ILs in the light of their further use and prospective development [19]. Currently, there is no previous work reported on the separation of Ta and Nb from dissolved alkaline solutions, and there still exists a luck of quantitative data on the solubility, separation, and precipitation of the hexaniobate or hexatantalate salts from high-grade ores.

2. Experimental

2.1. Materials

The ore sample sourced from Kenticha in the regional state of Oromia (Ethiopia). The mineralogical purity of the ore was investigated using a Maxima-XRD-7000 X-ray Diffractometer from Shimadzu with Cu K_{α} (1.540600A) radiation generated at 40.0 kV and 30.0 mA. The resulting spectra were interpreted using Diamond (version 4.2.2), Match (Version 3.2.2) with COD reference database and Origin 9.0. An Energy Dispersive X-Ray Fluorescence (EDXRF 2800) and an ICP-OES optima 7000 DV (Parkin Elimer 15,000k) were used for the percentage elemental analysis of the mineral ores. A high-temperature furnace was used for calcination in order to decompose the associated oxides, and a Shimaduzu FTIR-8400S spectrophotometer in KBr was used to support the presence or absence of the Nb/Ta oxygen bond

frequency. The particle size was analyzed using a 200-mesh sieve (No 72 PAT). The mass of the ores was measured by Sartorius ENTRIS323-1S chemical weighing. The pH measurements were performed using a pH-meter (827), and calibrated with standards at pH 4-9. The dissolution process was carried out in a high-temperature furnace at 400 °C inside a stainless batch reactor (SUS316). Analytical grade EMIC, H₂SO₄, and KOH (Sigma-Aldrich) without further purification, and de-ionized and doubly distilled water were used.

2.2. Procedure

Based on previously published papers [8, 9, 26], 5 g of tantalite was obtained by beneficiation from a pegmatite-spodumene ore (Scheme 1 and Table 1). The alkaline fusion steps were carried out in accordance with the previously published works [1, 4, 7, 9-14, 17, 19, 27]. A given mass of KOH was transferred to the reactor using a ceramic crucible, and was kept at 25 °C. While the temperature was reached, a mechanical stirrer was started, and 5 g of tantalite (sample 14) was added to each different mash size to obtain an optimum ratio of the lowest retained mass of residue. The mixtures of given proportions were placed inside a furnace, heated to 400 °C at a constant string speed at atmospheric pressure, and allowed to fuse for 1 h. While the products were cooled to the room temperature, they were leached with 100 mL de-ionized water, the filtrate was quantitatively transferred to a 100-mL volumetric flask, and a residue was obtained upon filtration followed by washing with doubly distilled water to pH 7 (Labeled as sample 4.1). All residues were compared by gravimetric analysis, and from the filtrates, the highest compositions of Nb and Ta were chosen, and their dissolved solution and the residues were analyzed by ICP-OES and EDXRF, respectively, and by FT-IR.

The solvent extraction processes were done by modifying the research work on solvent extraction fluoride media. and for preparation, in performed as described EMIC/AlCl₃ was previously for the electro-deposition of Nb and Ta [19]. 50 mL of the filtrate (sample A) was neutralized using H_2SO_4 to pH = 1-7 by shaking for 30 min. Biphasic of this neutralized solution was prepared by dissolving 10 mL of the neutralized filtrate with 2 g of EMIC/Al₂O₃ by shaking 3 times for 30 min (samples Aa, Ab, and Ac) in an atmosphere-dry box. The organic phase (sample B) was separated using a separatory funnel, and the raffinate was stored for further studies. Biphase was obtained upon adding 6N H_2SO_4 and 2g EMIC/AlCl₃ to the organic phase, and separated by a separatory funnel (the aqueous phase was labeled as D1 and the organic phase as sample A1). Striping of the aqueous and organic phases was conducted by adding 2 N H_2SO_4 and deionized water (labeled as samples D2 & A2, respectively). The dissolved Nb and Ta were analyzed by ICP-OES. Both striped phases were precipitated by adding a 30% ammonia solution and then filtered, dried in an oven for 3 days at 100 °C, and calcined in a furnace at 1,200 °C for 1 h. The calcined solids were analyzed by EDXRF.

3. Result and discussion

3.1. Characteristics of caustic conversion reaction of tantalite ore

In order to discuss the alkaline fusion phenomenon and phase analysis based on the research works of Wang et al., 2009 [7] and 2010 [17] and our works [9], the decomposition of the tantalite ore by molten KOH was followed by hydrolysis. Analysis of the residue showed an insoluble portion of KTaO₃ and KNbO₃ and a soluble portion of K₃NbO₄ and K₃TaO₄ (Eqs.1 and 2) including impurities such as K₂SiO₃, K₂SnO₃, KFeO₂, K₂MnO₃, and K₂TiO₃ [4, 7, 17].



Scheme 1. Systematic process of fusion, separation, precipitation, and calcination of Kenticha pegmatitespodumene ore.

According to X. Wang (2009) [7], Eqs. (1) and (2) suggest that the dissolution rate of the Nb ions is much higher than that for the Ta ions. The variables involved in the fusion step such as the particle size, solid-to-solid ratio, and pH of phase formation were studied, and the parameters such as the ore-to-solvent ratio, temperature, speed, and reaction time were accepted based on the works of [4, 7, 9, 17].

 $(Fe,Mn)O.(Ta,Nb)_2O_5 + 6KOH =$ $2K_3(Ta,Nb)O_4 + (Fe,Mn)O + 3H_2O$ (1)

3.1.1. Effects of particle size and alkali-to-tantalite mass ratio

The particle size of tantalite plays a major role in the decomposition process, which cannot be neglected. The effect of particle size was not included in the separation process but in order to keep constant, the effect was checked in Table 1 to show a constant particle size for further separation techniques. The effect of particle size on the dissolution and retained mass of the tantalite residue was studied from mesh sizes less than 200 to greater than 300 at the recommended decomposition parameters (Table 1), which were a temperature of 400 $^{\circ}$ C, a 330 rpm stirring speed, an 80 min reaction time, and leaching using water to reach 100 mL [9].

| Table | 1. | Effect of | КОН | on | tantalite ore. | |
|-------|----|-----------|-----|-----|----------------|--|
| | | | Mag | ь 6 | Sizo Dongo (g) | |

| VOU . Tontalita | Mesh Size Range (g) | | | | | |
|------------------------|---------------------|-----------|--------|--|--|--|
| KOII: l'antante | < 200 | (200-300) | >300 | | | |
| 2:1 | 0.0298 | 0.0386 | 0.0396 | | | |
| 3:1 | 0.0256 | 0.0374 | 0.0392 | | | |
| 4:1 | 0.0234 | 0.0354 | 0.0377 | | | |
| 5:1 | 0.0211* | 0.0311 | 0.0323 | | | |
| *G 11 / 1/ | | | | | | |

*Compared lowest result

The results shown in Figure 1 indicate that the changes in the particle size slightly affects the fusion of tantalite ore in the predicted range of mesh size; this can be attributed to the increase in the mesh size to 200 and over, having a significant effect on the dissolution of tantalite since under such conditions, the mesh size200 leads to a maximum degree of dissolution in each KOH:tantalite ratio. However. the alkaline-to-tantalite ore ratio largely affects the fusion and leaching process, which means a higher KOH-to-tantalite ratio, resulting in a less mass residue retained as well as leaching of the target metals and impurities. These results agree with the other works [4, 5, 7, 11, 12, 17]. In this work, the dissolved solution and residue of 5:1 KOH:tantalite ratio and mesh size 200 were preferred for further parameters such as the leaching temperature and time.



Figure 1. Particle size and mass ratio of residue in alkaline-fused tantalite.

3.1.2. Dissolved metal ions using KOH fusion

The decomposition temperature for Nb and Ta was checked from 400 ⁰C using an alkaline leaching agent with 2 g tantalite, mesh size 200, 10 g KOH, a 300 rpm string speed, and an 80 min reaction time to separate Nb and Ta from the dissolved alkaline solution, as shown in Table 2.

It is known that increasing the digestion temperature in the alkaline leaching process is effective in increasing the dissolved percent of target metal ions as well as impurity in metal ions, which can be attributed to an increase in the decomposition rate of the ore and to a decrease in the mass of undissolved metal oxides in the residue as well as using the acidic decomposition process [9, 11, 12]. However, Table 2 shows that the percentage decomposition of Nb oxide is higher than that of Ta oxide in the dissolved alkaline fused solution (sample A), as compared with the composition of the starting tantalite ore (sample 14). From this, it is possible to conclude that when tantalite ore is fused using KOH, there is around 15.22 wt% mass of undissolved mass of Ta oxide in the residue, and the decomposition of Nb oxides is the reverse. This gives evidence that there are Ta-containing oxides in the residue and that there are almost negligible Nb-containing oxides. This means that KOH fusion is an effective leaching agent to decompose the Nb ions compared with the Ta ions from high-grade tantalite ore. Therefore, when tantalite (Ethiopian tantalite ore that has the worldwide highest

exportable tantalite concentrate grade having compositions of 55.71 wt% Ta_2O_5 and 5.51 wt% Nb_2O_5 [9, 26]) is decomposed by the KOH and water leaching system, almost all Nb and most Ta ions are dissolved, producing Ta-containing residue due to high percent of the ore (Table 3). The dissolved Nb and Ta ions as well as impurity metal ions are recommended to form biphase from the alkaline solution proceeding the process of separation of Nb and Ta.

 Table 2. Oxide compositional analysis in alkaline fused solution using ICP-OES.

| rused solution using fer OLS: | | | | | |
|-------------------------------|--------------------------------|------------------|--------------------------------|------------------|--------------------------------|
| Sample | Ta ₂ O ₅ | K ₂ O | Nb ₂ O ₅ | TiO ₂ | Fe ₂ O ₃ |
| А | 42.23 | 38.75 | 5.22 | 5.20 | 5.11 |
| В | 41.32 | 6.86 | 5.18 | 1.09 | 3.78 |
| D1 | 0.01 | 2.34 | 4.22 | 0.00 | 0.00 |
| D2 | 0.01 | 1.11 | 0.23 | 0.00 | 0.00 |
| D3 | 0.01 | 0.11 | 0.02 | 0.00 | 0.00 |
| A1 | 37.89 | 3.22 | 0.41 | 1.1 | 1.56 |
| A2 | 1.13 | 2.51 | 0.023 | 0.11 | 1.23 |
| A3 | 0.29 | 1.13 | 0.01 | 0.11 | 1.01 |

Table 3. Chemical analysis of solid samples by EDXRF.

| Sample | Ta_2O_5 | K ₂ O | Nb ₂ O ₅ | TiO ₂ | Fe ₂ O ₃ |
|--------|-----------|------------------|--------------------------------|------------------|--------------------------------|
| 14 | 55.71 | 0.67 | 5.51 | Nd | Nd |
| 4.1 | 15.22 | 14.35 | 0.64 | Nd | 0.00 |
| A5 | 90.81 | 0.01 | 3.05 | Nd | 0.53 |
| D6 | 0.01 | Nd | 99.84 | Nd | Nd |

3.1.3. Characterization of residue

Taking into account the XRD (Figure 2), EDXRF (Table 2), and FT-IR (Table 3) results of the reaction of oxides of tantalum and niobium (OTN) on the fused and leached solution, the composition of the residue may depend on the composition of the ore sample, amount of potash, and reaction temperature (Eqs. 1 and 2) [4, 7, 17]. Figure 1 shows the results obtained for the XRD analysis of the residue left-over after fusion and leaching. The XRD spectra for the residue show different patterns for the parent ore as compared with the tantalite concentrate. In addition, the residue contains KTaO₃ not KNbO₃, which is in agreement with the previous works on the alkaline processes and consistent with that reported in JCPDS No. and different chemical 01-077-0917 [17] compositions with previous works published on fluoride media and different with that reported data base (JCPDS 33-659, 1993) [12]. This indicates that besides converting into soluble K₃TaO₄, a part of tantalum is directly converted into insoluble KTaO₃ in the KOH decomposition procedure, which consequently leads to a low dissolution rate of Ta₂O₅. At the recommended 400 °C temperature, a lower conversion rate to KTaO₃ was observed.

. (Fe,Mn)(Nb,Ta)₂O₆ Manganotantalite * KTaO₃ I rel 1000 tal pattern: Sample_B42 () 500 500 35.00 40.00 45.00 15.00 20.00 25.00 30.00 50.00 55.00 60.00 65.00 70.00 75.00 80.00 Cu-Ka1 (1.540598 A)

Figure 2. Compositional XRD spectra for Kenticha tantalite (sample 14) and residue (sample B4.2) from alkaline fusion.

Table 3 shows the results of EDXRF analysis for the ore before and after fusion with the KOH and water leaching system. From the results obtained, it can be seen that the KOH fusion gave around 27 wt% of Ta₂O₅ and 11 wt% of Nb₂O₅ remaining in the residue. Using a mole ratio of 5:1 of KOH-totantalite in the fusion process, 72.68% wt% and 88.39 wt% Ta₂O₅ and Nb₂O₅ were leached in the dissolved solution, respectively. These can be seen in the reduction of Ta₂O₅ from 55.71 to 15.22 wt% and Nb₂O₅ from 5.51 to 0.64 wt%, which implied that the water-leaching system was effective, and more elements and impurities were present in the leached liquors. The essence of the fusion was to get the leached liquor containing tantalum and niobium, which was required for separation of Ta and Nb from the aqueous biphase.

It is clear that most of the FT-IR stretching frequencies of the residue in Table 4 in the region of 1500-600 cm⁻¹ are due to similar functional groups, while some stretching frequencies of TNO (especially in the 500-700 cm⁻¹ region) are comparable to those obtained in the fusion product, parent ore, and tantalite, suggesting their possibility in similar oxide types of solid mineral

[9]. Comparisons between the FT-IR spectra for pegamatite-spodumene (sample 1), tantalite (sample 14), residue (sample 4.1), Nb₂O₅ (sample D5), and Ta₂O₅ (sample A5), clearly show that the compounds formed during the fusion digestion step are chemically and structurally different from

both Nb₂O₅ and Ta₂O₅. However, the stretching frequency at 1463 cm⁻¹ of Nb₂O₅ is different from all samples. This suggests the absence of Nb-O stretching in the residue and agrees with the results of XRD patterns and EDXRF data for the residue.

| Table 4. | FT-IR | results | of se | bilc | sam | nle |
|-----------|---------------|----------|-------|------|-------|----------|
| 1 4010 11 | I I II | I Coulto | 01 50 | JIIU | Stern | $p_{1}c$ |

| Compound (sample) | Frequency, cm ⁻¹ |
|--|--|
| Sample 1 | 3520 (vs); 2954 (s); 1774 (s); 1665(s); 1454 (vs); 732 (vs); 657 (vs), 555 (w) |
| Tantalite (sample14) | 3520 (vs); 2954 (s); 1774 (s); 1665(s); 1454 (s) 721 (vs); 655 (vs); 555 (vs) |
| Residue 1 (sample 4.1) | 3520 (vs); 2954 (s); 1774 (s); 1685(s); 1454 (s); 721 (vs); 655 (w); 555 (vs) |
| Nb ₂ O ₅ (sample D6) | 3520 (vs); 2954 (s); 1774 (s); 1685 (s); 1463 (s); 655 (w); 555 (s) |
| Ta_2O_5 (sample A6) | 3520 (vs); 2954 (s); 1774 (s); 1685 (s); 1454 (s); 721 (s) |

3.2. Solvent extraction of Nb and Ta from KOH-fused solution

3.2.1. Separation and precipitation

Due to the formation of $H_xM_6O_{19}^{x-8}$ (aq.) by water leaching according to Equation 3 [4, 7, 17, 19], it was essential to neutralize the Nb or Ta alkaline solutions formed by excess KOH from the tantalite ore. The pH of the alkaline solution was adjusted to 1-7 by adding H_2SO_4 and 2 g of EMIC/AlCl₃ to the feed solution (sample A) that had 94.73 vol% Nb₂O₅ and 75.80 vol% Ta₂O₅. purified Nb-Ta hydrous oxide, (Nb,Ta)₂O₅.nH₂O [11, 12]. For a better extraction of both elements, collective extraction can be done, while both elements are in the upper phase (UP), not the lower phase (LP). In order to make a clear biphase separation, the composition of the filtrate (Table 2) was analyzed at various pH values from 1 to 7, as shown in Figure 3. A possible extraction mechanism is stated in Eqs. 3-5 [28-31].

Acidification with H₂SO₄ helps to produce a

 $EMIM^+Cl^- + AlCl_3 \leftrightarrow EMIC^+ [AlCl_4]^-$

 $\text{EMIM}^+ [\text{AlCl}_4]^- + \text{AlCl}_3 \leftrightarrow \text{EMIM}^+ [\text{Al}_2\text{Cl}_7]^-$

(3)

(4)

(5)

 $Ta/Nb_{(w)}^{m+} + m_{(0)} + m[EMIM^{+} [Al_{2}Cl_{7}]^{-}]_{(0)} = Ta/Nb_{m(0)}^{m+} + m[EMIM^{+} [Al_{2}Cl_{7}]^{-}]_{(w)}$



Figure 3. Compositional analysis of leached liquor using ICP-OES.

As it can be seen in Figure 3, pH 6 is suitable for a further biphase separation having 99.23 vol% of Nb₂O₅ and 97.84 vol% of Ta₂O₅ in UP. This high dissolution of the Nb and Ta ions may be referred to the difference in the mineralogical composition of the Kenticha ore and reduction by excess KOH and water leaching. The biphase was achieved by adding H₂SO₄ (6N) and 100 wt% EMIC/AlCl₃ to UP prior to phase separation of LP, and UP 81.46 vol% Nb₂O₅ (sample D1) and 91.69 vol% Ta₂O₅ (sample A1) were obtained from the loaded LP and UP, respectively. In this case, from the extraction of Nb ions from the feed solutions, a higher efficiency was obtained as compared to the fluoride-based leaching and organic phase extraction methods reported in the literature [1, 3, 3]13, 19, 32-41]. This may be due to the action of EMIC/AlCl₃, type of the ore or differences in reaction, and neutralization conditions.

Accordingly, an equal volume of the loaded solvent was shaken for 30 min with H_2SO_4 (2N) and doubly distilled water with 2 vol% EMIC/AlCl₃ (samples D2, D3, A2, and A3), respectively. It was found that a complete stripping of 99 vol% of Nb and 96.51 vol% Ta ions were achieved from the LP and UP solutions. These strip solutions containing Nb (samples D6 and A5. respectively) were directed to precipitation by adding 30 vol% ammonia solution to pH 9 followed by filtration, dried in an oven at 100 °C for 72 h and calcite in furnace at 1,200 ^oC for 1 h to have 99.84 wt% Nb₂O₅ and 90.81 wt% Ta₂O₅. The compositions of the dissolved metal ions analyzed by the ICP-OES (Table 1) and EDXRF (Table 2) data and XRD patters (Figure 4) for Nb₂O₅ and 8 wt/vol% difference in the ICP-OES and EDXRF of Ta₂O₅ are in good agreement. This implies that there is impurity in the Ta₂O₅ solid phase, which was formed during precipitation and would not be sublimated in the calcination range of temperature. Thus it is possible to suggest that alkaline fusion is an efficient and environmentally friendly leaching method for Nb ions. In addition, using EMIC/AlCl₃ as the extractant is more effective for Nb ions than Ta ions to produce crystalline pure Nb_2O_5 . In conclusion, replacement of organic solvents (MIBK) using EMIC reduces the volatilization of solvents. The majority of the impurities of the extraction system

remain in the filtrates, and the residue will be used to recycle.

3.2.2. Characterization of Nb₂O₅ and Ta₂O₅

Figure 4 presents the XRD pattern for Ta_2O_5 , revealing a non-crystalline nature. This indicates that the separation and precipitation processes of Ta_2O_5 yield other metal oxides or impurities, and this is in agreement with the EDXRF data shown in Table 3. The XRD analysis of Nb₂O₅ suggests that it exhibits a totally crystalline behavior, while Ta_2O_5 has an amorphous nature, and this is confirmed by the EDXRF 99 wt% of pure Nb₂O₅ and 90 wt% Ta_2O_5 . This reveals that the alkaline fusion and solvent extraction using IL is an excellent process for production of Nb₂O₅, and is reasonable for production of Ta_2O_5 from highgrade tantalite ore.

It can be seen from the FI-IR stretching frequencies of Ta_2O_5 (sample A5) and Nb_2O_5 (sample D6) shown in Table 4 and Figure 5 that the region 1500-3600 cm⁻¹ has mostly similar stretching frequencies compared to the parent ore (samples 1 and 14). The strong stretching frequencies at 555-721 cm⁻¹ and at 800-1000 cm⁻¹ for the vibrations of M-O-M bridges and M-O bond of the metal oxide, respectively, are similar to the literature values [5, 9, 11, 12], indicating possible frequencies of tantalate and niobate oxides. The characteristic stretching frequencies observed at 721 cm⁻¹, 655 cm⁻¹, and 555 cm⁻¹ may be attributed to the presence of M=O stretching, M-O bending deformation, and M-O stretching frequencies. In comparison, the absence of the 721 and 1463 cm⁻¹ stretching frequencies of Ta_2O_5 confirms the Ta-O stretching, while the presence of strong bands at 721 and 1463 cm⁻¹ in Nb₂O₅ is attributed to the Nb-O stretching in Ta_2O_5 , whereas in Ta_2O_5 , it is attributed to the presence of Nb₂O₅ due to the presence of stretching frequencies at medium bands 655 and 555 cm⁻¹ attributed to the Nb-O bond stretching. The very clear stretching frequencies in the 1,500-1,000 cm⁻¹ region suggest that there is a homogeneous distribution of Nb₂O₅ particles in Nb₂O₅. The absence of very strong or unique stretching frequencies for both oxides does not make this FT-IR very effective for the characterization of the products but some useful information can be obtained by comparison.





Figure 4. Compositional XRD spectra for Ta₂O₅ (sample A5) and Nb₂O₅ (sample D6).

Figure 5. FI-IR stretching frequencies of Ta₂O₅ (sample A5) and Nb₂O₅ (sample D6).

4. Conclusions

1. This work confirms that the alkaline fusion can be used as an alternative to fluoride media in the extraction and separation of the Nb and Ta ions in a GREEN and new process. The dissolved tantalite in KOH fusion and H₂O leaching was reacted at 400 0 C for 1 h, and 15.22 wt% of Ta₂O₅ was converted into insoluble KTaO₃, and around 94 vol% of Nb₂O₅ and 75 vol% of Ta₂O₅ were dissolved for beneficial solvent extraction processes. 2. For the extraction using EMIC/AlCl_3 , IL for Nb and Ta from KOH fusion was dependent on the neutralization pH, and the highest Nb separation was achieved at pH 6.

3. The extraction was improved considerably with the introduction of sulfuric acid and water into the feed solution. For a nearly complete extraction of tantalum and niobium with the ionic liquids as a green extractant and recovery of 99.84 wt% Nb₂O₅ and 90.81wt%, Ta₂O₅ was attained by precipitation and calcination from the strip solution. That alkaline fusion is a powerful approach for solvent extraction of niobium from high grade tantalite ore.

4. To develop further works on loading, the kinetic and thermodynamics of solubility, scrubbing, and increasing purity of Ta_2O_5 are required. However, considering the state of the art of EMIC/AlCl₃ ionic liquids as Nb and Ta extracting agents, their application in real water systems is still limited to the basic knowledge regarding their stability, solubility, and metal-uptake mechanism.

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استخراج نیوبیوم و تانتالیم از سنگ معدن تانتالیت Kenticha با استفاده از مایع یونی کلرید 1-Ethyl-3-Methyl Imidazolium

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چکیدہ:

در این پژوهش، تجزیه سنگ تانتالیت Kenticha اتیوپی با استفاده از سیستم همجوشی KOH و H₂O در دمای ۴۰۰ درجه سانتی گراد برای یک ساعت واکنش مورد مطالعه قرار گرفت. نتایج تجربی به دست آمده نشان داد که ۲۷٪ وزنی از Ta₂O₅ به طور عمده به KTaO₃ نامحلول تبدیل می شود و همچنین به محلول مایع حاوی ۹۴٫۷۳٪ از Nb₂O₅ و ۷۵٫۸۰٪ از Ta2O5. مشاهدات که توسط روش های XRD، EDXRF و ICP-OES پشتیبانی شده است، نشان داد که نسبت مخلوط محلول یونهای فلزات Nb و Ta بر روی استخراج حلال از هر دو T2O5 و Nb2O5 سودمند است. نیوبیوم و تانتالیم از محلول حل شده و هیدرولیز شده قلیایی خنثی شده توسط فرآیند جدید با استفاده از مایعات یونی EMIC/AICl3 به عنوان استخراج کننده به دست می آید. همچنین ترکیبات یونهای فلز محلول و جامدات با روشهای EDXRF JCP-OES و RT-IC/2 مورد تحلیل قرار گرفت.

كلمات كليدى: تجزيه، يونيك مايع، سنگ معدن تانتاليت Kenticha.