

Study on Extraction of Niobium Oxide from Columbite–Tantalite Concentrate

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Abstract—The principal objective of this study is to be able to extract niobium oxide from columbite-tantalite concentrate of Thayet Kon Area in Nay Phi Taw. It is recovered from columbite-tantalite concentrate which contains 19.29 % Nb_2O_5 . The recovery of niobium oxide from columbite-tantalite concentrate can be divided into three main sections, namely, digestion of the concentrate, recovery from the leached solution and precipitation and calcinations. The concentrate was digested with hydrofluoric acid and sulfuric acid. Of the various parameters that effect acidity and time were studied. In the recovery section solvent extraction process using methyl isobutyl ketone was investigated. Ammonium hydroxide was used as a precipitating agent and the precipitate was later calcined. The percentage of niobium oxide is 74%.

Keywords—Calcination, Digestion, Precipitation, Solvent Extraction.

I. INTRODUCTION

NIOBIUM always occurs with tantalum because of the similarity in their atomic size. The minerals columbite and tantalite contain both elements, differing only in their proportion. Niobium is located in the periodic table in the middle of a group of refractory metals that has been intensively investigated during the last few years [1].

Columbite contains more niobium and tantalite contains more tantalum. Columbite was named after Columbia in America, the place of origin of the mineral from which the metal was isolated in 1802. Columbite and tantalite are usually found as opaque iron- black or brownish colored minerals having a hardness of about 6. They have a metallic or brilliant resinous luster. The specific gravity varies between 5.3 and 8.0 according to the proportion of tantalum and niobium present, thus providing a simple means of identification [2].

Niobium has been used mainly as an addition agent to construction steels and as an alloying component of heat-resistant alloys and many high-strength low-alloy steels. High purity niobium is utilized to fabricate radio frequency cavities for the acceleration of electrons in quantum physics, because of the good ductility and high thermal conductivity of thin sheets. It is also employed in the field of applied superconductivity, mainly including energy-related applications, transportation, computers and instrumentation. Niobium alloys such as NbTi alloys and Nb_3Sn intermetallics,

have been the superconducting materials commercially available [2].

II. EXPERIMENTAL PROCEDURE

In this paper, columbite-tantalite concentrate was used for the extraction of niobium oxide. This paper made the study of niobium oxide extraction from columbite-tantalite concentrate from Thayet Kon Area, Pynmana Township. The flow diagram of the extraction of niobium oxide is shown in Fig. 1.

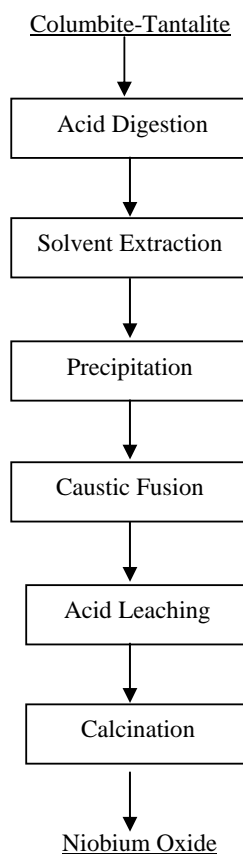


Fig. 1 Flow Diagram of Extraction of Niobium Oxide

Acid Digestion

In the processing, the concentrate was crushed to -325 mesh. Columbite-tantalite concentrate was digested with hydrofluoric acid and sulfuric acid. In this step the normality of acid concentration were varied.

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To get optimum condition, the concentrations of H_2SO_4 and HF were varied. The acid concentration of 6N HF and 1N H_2SO_4 were put in the Teflon beaker and it was heated and then the concentrates were added in the Teflon beaker. The Teflon beaker was covered with the Teflon plate in order to prevent acid losses by evaporation. The slurry was heated to $100^\circ C$ and the temperature was maintained. The slurry was agitated every 15 minutes and the hot distilled water was added to maintain the solution volume. When the leaching time was completed, the slurry was filtered with filter paper and the residue was washed to pH 7 with pH paper. And then the residue was dried in the oven, weighed and determined by XRF and XRD analysis [3-7].

Solvent Extraction

This extraction step is carried out to upgrade niobium oxide by using the extractant, MIBK. After filtration, the filtrate solution was prepared at 2N HF and 8N H_2SO_4 . To obtain one liter solution, the filtrate solution was made to the concentration of 2N HF and 8N H_2SO_4 solution and also distilled water was added. From one liter solution, 100ml of aqueous solution and 200ml of the organic solvent (methyl isobutyl ketone) were put into the polyethylene separating funnel. The funnel was stoppered well and was shaken on the shaker. After shaking for 30 minutes and allowing settling, rich organic solvent and raffinate were separated. Niobium and tantalum were in the organic phase and the impurities were in the raffinate. To remove the impurities, the organic 200ml was scrubbed with 2N H_2SO_4 by the shaker. The niobium rich was extracted into the aqueous solution and remained tantalum containing in the organic phase. The niobium containing solution was mixed with 8N H_2SO_4 for solvent extraction. After that niobium was extracted into the pregnant solution and was separated as in tantalum in organic. Outgoing aqueous with rich niobium pregnant solution was obtained and collected for the precipitation step. This process is shown in Fig. 2 [3-7].



Fig. 2 Extraction with MIBK

Precipitation

The pregnant solution was put in a beaker and heated for one hour. By adding 30% NH_4OH to the pregnant solution, precipitation took place until pH 11 was reached. The precipitate was allowed to settle and filtered. The precipitates were washed with boiled distilled water to pH 7 and dried in the oven [3-7].

Caustic Fusion

The dried precipitate ($Nb(OH)_5$) and sodium hydroxide ($NaOH$) were put in a porcelain crucible in the ratio of 1:3. The crucible was placed in the muffle furnace at $800^\circ C$ for one hour [3-7].

Acid Leaching

The fused mass was leached with HCl (30%) on the hot plate. The solution was filtered and the precipitate was washed to pH 7 and dried in the oven [3-7].

Calcination

The niobium hydroxide was crushed to powder. Calcination was done at $1200^\circ C$ for one hour. The niobium pentoxide was obtained [3-7].

III. RESULTS AND DISCUSSION

Effect of Acid Concentration on Digestion

Columbite-Tantalite concentrate from Thayetkon Area, Pyinmana Township, contained 19.29 % Nb_2O_5 . Heavy concentrate contained 5 % columbite-tantalite mineral. Columbite-tantalite mineral was separated from other mineral by Frantz Isodynamic Magnetic separator. After separation, heavy concentrate contained about 62% columbite tantalite mineral.

By mineralogy result, the average minerals composition of concentrate is shown in Table I. The concentrate was pulverized to -325 mesh and then sample were analyzed by XRD and XRF analysis. The XRF analysis result is shown in Table II.

TABLE I
AVERAGE MINERALS COMPOSITION OF HEAVY CONCENTRATE

Minerals	Percent
Columbite-Tantalite	62.88
Cassiterite	10.98
Xenotime	9.81
Monazite	5.84
Garnet	3.91
Ilmenite	3.60
Brookite	2.98

TABLE II
XRF ANALYSIS OF COLUMBITE-TANTALITE CONCENTRATE

Columbite-Tantalite Concentrate	
Nb_2O_5	19.29 %
Ta_2O_5	9.93 %
ZrO_2	2.17 %
Y_2O_3	1.75 %
Fe_2O_3	22.99 %

Columbite-Tantalite concentrate was leached with a mixture of hydrofluoric acid and sulfuric acid. The variation of acid concentration and leaching time were studied. The filtrate solution was used for solvent extraction with MIBK. The pregnant solution was precipitated, caustic fusion, HCL leaching and calcined.

The various concentrations of hydrofluoric acid and sulfuric acid were tested to obtain a condition to extract maximum amount of niobium in the filtrate and minimum amount of niobium in the residue.

The concentration of 1N H₂SO₄ was held constant and the concentration of HF were varied as 1N, 2N, 3N, 4N, 5N, 6N, 7N, 8N, 9N, 10N respectively. To study the effect of sulfuric acid, the concentration of H₂SO₄ was varied from 1N to 5N.

According to the leaching tests, the concentration of 6NHF and the concentration of 1NH₂SO₄ were chosen because these conditions gave minimum amount of niobium oxide in residue.

Table III shows as that the percentage of niobium oxide in residue was slightly less at 1N H₂SO₄ and 6N HF than other acid concentration. So the optimum condition is 1N H₂SO₄ and 6N HF.

TABLE III
ANALYSIS OF NIOBIUM OXIDE IN RESIDUE

Acid Concentration		Niobium oxide in Residue (%)
H ₂ SO ₄ (N)	HF(N)	
1	6	0.09%
2	6	0.11%
3	6	0.13%
4	6	0.1%
5	6	0.1%

In order to recover niobium oxide from the pregnant solution, solvent extraction method using MIBK was carried out. Two stages were employed.

By adding NH₄OH to the pregnant solution, precipitation took place until pH 11 was reached. The precipitate and sodium hydroxide were put in a porcelain crucible and was placed in the muffle furnace.

HCL digestion was necessary to remove impurities. The fused mass from caustic fusion was put in a beaker and leached for ½ hr. Calcinations was further performed for the production of pure niobium oxide.

IV. CONCLUSION

The head sample was first characterized and analyzed by X-ray diffraction (XRD) and X-ray fluorescence (XRF). The head sample was found to contain 19.29% Nb₂O₅. Columbite-Tantalite concentrate was digested with various concentrations of hydrofluoric acid and sulphuric acid. The leached solution was purified by solvent extraction process to obtain a high percent purity and yield. The percentage of niobium oxide in residue and purity of niobium oxide were determined by XRF

and XRD. The purity of niobium oxide obtained is 74% Nb₂O₅. The application of solvent extraction separation gave high purity for niobium oxide.

After solvent extraction, the pregnant solution was precipitated with ammonia. The precipitate was filtered and dried.

Caustic fusion of the dried precipitate followed by hydrochloric acid leaching, filtration, washing, drying and calcination at 1200 °C.

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