

CHARACTERIZATION OF AN ORE SAMPLE FROM CONGO BASED ON TANTALUM AND NIOBIUM FOR POSSIBLE FURTHER TECHNOLOGICAL TESTS

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ABSTRACT – In this study, two samples of "Ta-Nb" ore from Congo (MMR-1 and MMR-2) were analyzed. Various physical and chemical properties were determined for these samples, including gross and hygroscopic moisture, specific gravity, granulometric composition of the initial samples and after each level of crushing, bulk density of the initial samples and after each crushing stage, and chemical composition. Qualitative mineralogical analysis was conducted using X-ray diffraction (XRD), while polished sections were prepared for optical microscopy, scanning electron microscopy (SEM), and mineralogical analysis by particle size classes. Detailed physicochemical and mineralogical tests were carried out on the MMR-1 and MMR-2 samples to comprehensively define the parameters necessary as a foundation for further technological testing of "Ta-Nb" ore from Congo. The results showed that the examined samples have appropriate characteristics for further technological tests.

Keywords: "Ta-Nb" ore-Congo, Physico-Chemical, and Mineralogical Investigation.

INTRODUCTION

Tantalum (Ta) and niobium (Nb) are transition metal elements whose economic importance continues to grow due to the increasing demand for their use in various technological applications [1-3]. The unique properties of Ta and Nb, such as their high dielectric constants, stability of their oxides, and corrosion resistance [3-6], make these metals highly valuable in electronics, aerospace, nuclear, and automotive industries, as well as in military and medical applications [2, 3, 7, 8]. Tantalum is invariably associated with niobium, and both elements are primarily extracted from columbite-tantalite group minerals, which are minor components found in primary granites, granite pegmatites,

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and greisen rocks [1,3]. In nature, tantalum coexists with niobium as oxides, often associated with iron and manganese oxides, and sometimes includes elements such as tin, titanium, lithium, cesium, and trace amounts of tungsten, uranium, and thorium in pegmatitic and related lithologies [3, 9, 10]. Columbite-tantalite (commonly referred to as coltan) group minerals $[(\text{Fe},\text{Mn})\text{O}(\text{Ta},\text{Nb})_2\text{O}_5]$ are the primary sources for tantalum and niobium production. These include [tantalite-(Fe) or tantalite-(Mn)] and [columbite-(Fe) or columbite-(Mn)] [2, 3, 11].

Additionally, tantalum, tin, and niobium are found in other minerals such as pyrochlore, ixiolite, and wodginite, which are complex oxides and hydroxides [3, 10]. In 2022, global tantalum production, expressed as Ta_2O_5 content, was approximately 1,726 tons, with Congo contributing around 550 tons (31.90%), followed by Brazil (20.30%), Rwanda (17.40%), Nigeria (8.10%), China (5.30%), Ethiopia (5.20%), Mozambique (4.80%), and others (7.0%). In the same year, global niobium production, expressed as Nb_2O_5 content, totaled 118,209 tons, with Brazil accounting for 110,000 tons (93.05%), Canada (5.75%), and other countries (1.20%) [12]. Both Ta and Nb are classified as critical raw materials (CRM) for the European Union [13].

Ta and Nb-based ore samples from the Kivu Belt (KVB) region of the Congo were analyzed by different test methods. The Kivu Belt is a geological part of the Kibara Belt, which extends over 700 kilometers from Katanga Province in southeastern Congo to Uganda [14]. The paper will present the results of testing ore samples based on Ta and Nb, with the aim of establishing the characteristics of the mentioned metals and their possibility in further technological applications.

EXPERIMENTAL

Materials and methods

The physicochemical and mineralogical properties of two "Ta-Nb" ore samples from Congo called MMRS-1 and MMRS-2 were examined by the Institute (ITNMS) for a Slovenian company. Each sample weighed approximately 25 kg. The tests aimed to determine the key properties of the mineral raw material, assess its potential as a resource, and evaluate the technological possibilities for processing the "Ta-Nb" ore into commercial products for various industrial sectors. The analysis included determining gross and hygroscopic moisture, specific gravity, granulometric composition of the initial samples and after each crushing stage, and bulk density. Granulometric composition was determined by dry sieving using Tyler's series of sieves for the initial samples. Due to the high clay content in the ore, samples were washed, and the granulometric composition was re-evaluated using the wet method.

Mineralogical analyses were conducted using X-Ray Diffraction (XRD), Reflected Light Microscopy, and Scanning Electron Microscopy (SEM), providing both qualitative and quantitative data on the samples. All analyses adhered to internationally recognized standards for evaluating the quality of mineral raw materials.

Chemical analyses of the "Ta-Nb" ore samples were performed at the ICP laboratory of the Jožef Stefan Institute. Both samples were prepared identically for characterization, with the sample preparation method outlined in Figure 1.

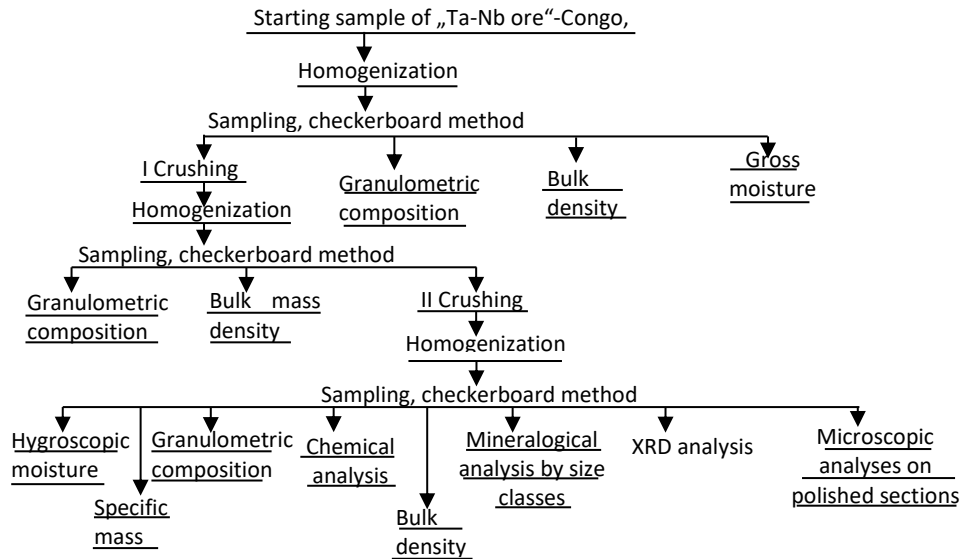


Figure 1 Written scheme of preparation “Ta-Nb ore samples” - Congo (MMR Sample 1 and MMR Sample 2) for physico-chemical and mineralogical investigation

Physical characterization of the sample

As part of the physical characterization, the gross moisture (Hg) content of the initial samples of the “Ta-Nb” ore—Congo was first determined. Sample MMR-1 and sample MMR-2 had Hg presence of 0.30 and 0.12%, respectively. The content of hygroscopic moisture in the sample MMR-1 and MMR-2 was 0.32 and 0.39%, respectively.

The granulometric compositions for both samples of “Ta-Nb” Congo (MMR sample 1 and MMR sample 2) were determined on the initial samples, after the first crushing and after the final crushing. The particle size distribution of these samples was determined by wet sieving on sieves (Tyler sieve series). During these tests, upper size limit (d_{95}) and the mean grain diameter (d_{50}) were determined for each of the samples. In addition, the bulk density of the samples was measured during these tests (SRPS EN ISO 3252:2014). All these parameters for each of the samples are shown in Table 1.

Table 1 Granulometric composition and bulk density of “Ta-Nb” ore samples

MMR-1 initial sample			MMR-2 initial sample		
$d_{95}=23,914\text{mm}$	$d_{50}=3,507$	$\Delta=1.35\text{g/cm}^3$	$d_{95}=19,647\text{mm}$	$d_{50}=1,683\text{mm}$	$\Delta=1.37\text{g/cm}^3$
MMR-1 after I stage of crushing			MMR-2 after I stage of crushing		
$d_{95}=9,767\text{mm}$	$d_{50}=1,961$	$\Delta=1.31\text{g/cm}^3$	$d_{95}=9,904\text{mm}$	$d_{50}=1,25\text{mm}$	$\Delta=1.34\text{g/cm}^3$
MMR-1 after final crushing			MMR-2 after final crushing		
$d_{95}=4,386\text{mm}$	$d_{50}=0,959$	$\Delta=1.19\text{g/cm}^3$	$d_{95}=4,332$	$d_{50}=0,594\text{mm}$	$d_{50}=1,23\text{g/cm}^3$

The specific mass was determined for each of the "Ta-Nb" ore samples (MMR-1 and MMR-2). The specific gravity value obtained for sample MMR-1 and MMR-2 was 2.55g/cm³ and 2.58g/cm³, respectively.

Chemical compositions of both starting samples "Ta-Nb" ore-Congo

The chemical composition of both initial samples of "Ta-Nb" ore from Congo (MMR-1 and MMR-2) was determined using ICP analysis at the Jožef Stefan Institute. The results of the chemical analysis for major components (high content) are presented in Table 2, while the results for minor components (low content) are shown in Table 3.

Table 2 Chemical composition of the MMR-1 sample "Ta-Nb" ore-Congo, main components

Components	Si	Al	Ca	Na	K	Mg	Fe	Ti
Sample MMR-1, %	29,94	8,39	0,073	0,90	1,57	0,055	1,61	0,052
Sample MMR-2, %	31,18	9,68	0,11	1,48	1,515	0,050	0,83	0,022

Table 3 Chemical composition of the MMR-1 sample "Ta-Nb" ore-Congo, minor components

Components	Mn	Ni	Mo	Nb	Cr	Sn	Ta	W
Sample MMR-1, g/t	193	20	45	111	97	167,9	212,3	45,1
Sample MMR-2, g/t	453,1	23,34	48,37	118,37	85,29	178,97	150,1	44,2

Mineralogical study of two samples MMR S1 and MMR S2 „Ta-Nb“ ore -Congo

As part of the mineralogical analysis, preparations were made for optical and SEM microscopy, XRD analysis, and mineralogical examination by individual size classes.

XRD method-results of the research

The qualitative mineralogical composition of the MMR-1 sample includes quartz, mica (muscovite), illite, kaolinite, dickite, nacrite, plagioclase, alkali feldspar (K-feldspar), columbite-tantalite, and wodginite. Semi-quantitative mineralogical analysis of the crystalline phases, determined using the Rietveld refinement method, is as follows (in weight %): quartz 61.4 (0.49)%, kaolinite 13.1 (0.73)%, muscovite 12.2 (0.6)%, plagioclase 6.8 (0.69)%, dickite 4.9 (0.58)%, illite 0.4 (0.04)%, nacrite 0.4 (0.04)%, K-feldspar 0.8 (0.05)%, with columbite-tantalite and wodginite present in trace amounts.

The qualitative mineralogical composition of the MMR-2 sample includes quartz, mica (muscovite), illite, kaolinite, dickite, nacrite, alkali feldspar (K-feldspar), plagioclase, columbite-tantalite, and wodginite. The quantitative mineralogical analysis of the crystalline phases is as follows (in weight %): quartz 39.5 (0.75)%, plagioclase 24.1 (0.69)%, muscovite 15.9 (0.85)%, kaolinite 8.0 (0.53)%, K-feldspar 7.3 (0.54)%, dickite 1.4 (0.67)%, nacrite 3.1 (0.38)%, illite 0.8 (0.64)%, with columbite-tantalite and wodginite present in trace amounts.

The diffractograms of samples "MMR-1" and "MMR-2" are shown in Figures 2 and 3, respectively.

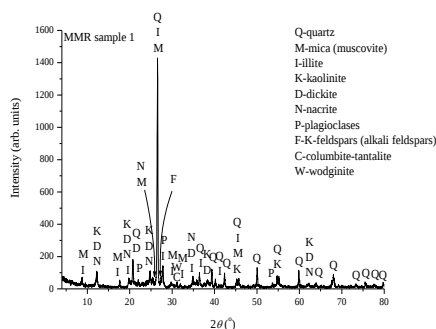


Figure 2 XRD diffractogram of the "MMR S1"

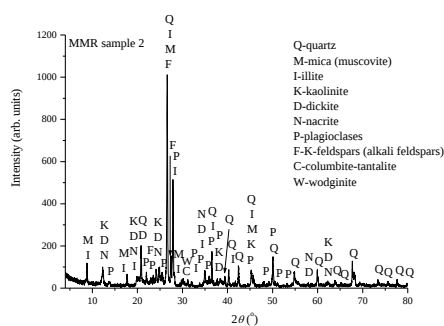


Figure 3 XRD diffractogram of the "MMR S2"

Reflected Light and Scanning Electron Microscopy method

According to mineralogical analyses, the useful minerals identified in both samples (MMR-1 and MMR-2) are columbite, tantalite, wodginite, and gersdorffite, all occurring in small quantities. Microscopic analyses revealed that the MMR-2 sample does not contain niobium-tantalates (columbite-(Fe,Mn)-tantalite-(Fe,Mn) series) but only (ferro)wodginite ($\text{FeSnTa}_2\text{O}_8$), which explains the lower Nb content in the MMR-2 sample.

The grain size of niobium-tantalates in MMR-1 (columbite-(Fe,Mn)-tantalite-(Fe,Mn) series and (ferro)wodginite) ranges from 10 μm to 115 μm , while the grain size of (ferro)wodginite in MMR-2 ranges from 90 μm to nearly 200 μm . A grain liberation study of Nb-Ta-bearing minerals (columbite-tantalite, wodginite) and cassiterite indicates that in the size class $-0.15+0.1$ mm, over 80% of the grains are liberated in both samples.

CONCLUSION

Mineralogical analyses of both samples (MMR-1 and MMR-2) of the "Ta-Nb" ore from Congo indicate that clay minerals (illite, kaolinite, dickite, nacrite) and mica (muscovite) are present as accompanying minerals, constituting approximately 30% of the starting sample by weight. Removing these minerals via washing would be the initial step in processing the "Ta-Nb" ore from Congo.

For the concentration of useful minerals (columbite-tantalite and cassiterite) from the washed "Ta-Nb" ore, gravity concentration and magnetic separation are the most suitable methods.

1. **Gravity Concentration:** Gravity concentration is feasible due to the significant difference in density between the useful minerals and the tailing minerals. The useful minerals, such as columbite ($\sim 6 \text{ g/cm}^3$), cassiterite ($\sim 7 \text{ g/cm}^3$), tantalite ($\sim 8 \text{ g/cm}^3$), (ferro)wodginite ($\sim 7 \text{ g/cm}^3$), and gersdorffite ($\sim 6 \text{ g/cm}^3$), have much higher densities compared to the primary tailing minerals, such as quartz and feldspar, which have

densities below 3 g/cm³. This substantial density contrast makes it possible to efficiently separate the useful minerals from the tailings using gravity-based methods.

2. **Magnetic Separation:** Magnetic separation can effectively separate the minerals tantalite, columbite, cassiterite, and wodginite from each other. Based on their magnetic properties, tantalite, columbite, and wodginite are paramagnetic, while cassiterite is diamagnetic. This distinction allows for relatively easy separation of cassiterite from the other minerals.

Based on the examination of ore samples containing Ta and Nb from Kon, it can be concluded that the examined ore has the capacity to extract useful elements and use them in technological applications of various industrial sectors.

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