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Original research article

STUDY OF MINERALOGICAL AND PHYSICOCHEMICAL PROPERTIES OF SAMPLES OF FLOTATION TAILINGS "RUDNIK" MINE

Dragan S. Radulović[1#], 0000-0001-8210-8100,
Jovica Stojanović[1], 0000-0002-2752-6374,
Grozdanka Bogdanović[2], 0000-0002-1013-4075,
Vladimir Jovanović[1], 0000-0001-6069-1630,
Dejan Todorović[1], 0000-0002-2776-3388,
Branislav Ivošević[1], 0000-0001-9445-8159,
Vladimir Simić[3], 0000-0002-2483-1779,
[1]ITNMS, 11000 Belgrade, Serbia
[2]University of Belgrade, Technical faculty in Bor, Bor, Serbia
[3]Faculty of Mining and Geology, University of Belgrade, 11000 Belgrade, Serbia

ABSTRACT – In this study, two samples of flotation tailings from the "Rudnik" Mine (one from boreholes and one from the surface) were examined. Various physical and chemical properties were analyzed, including gross and hygroscopic moisture, specific mass, bulk density in both loose and compacted states, and specific weight. Additionally, granulometric and chemical compositions were determined. A qualitative mineralogical analysis was conducted using X-ray diffraction (XRD), along with the preparation of polished sections for optical and SEM microscopy. Comprehensive physicochemical and mineralogical tests were performed to define all parameters necessary for the valorization and recycling of flotation tailings from the "Rudnik" Mine.

Keywords: Flotation Tailings, Physico-Chemical And Mineralogical Investigation, Recycling.

INTRODUCTION

The extraction and processing of ore generate large amounts of mine waste materials, including tailings, metallurgical waste, and waste rock [1-3]. While these materials may present economic opportunities for re-use and re-mining, they can also cause severe environmental damage and contamination if not properly managed [3-10]. Sulfidic mine waste has the potential to generate Acid Rock Drainage (ARD) and release toxic elements into the environment under oxidizing conditions, posing significant risks to both ecosystems and human health. Therefore, the re-use of mine waste materials can play a crucial role in mitigating environmental hazards [3].

The "Rudnik" Mine has been engaged in the exploitation and processing of Pb-Zn ore since 1953. This activity inevitably led to the construction of a tailings pond, which has expanded over time and now covers an area of more than 30 hectares. Over 11,000,000 tons (more than 7 million m^3) of flotation tailings have been deposited in the Rudnik Mine

[#] corresponding author: d.radulovic@itnms.ac.rs

tailings pond. This fine-grained material is a residue from the flotation process, which produced K/PbS, K/$CuFeS_2$, and K/ZnS concentrates.

Based on previous preliminary tests, a program of technological studies on the flotation tailings of the Rudnik Mine has been developed. The objective of these studies is to valorize valuable components from the tailings, including precious metals, non-ferrous metals, and critical elements, while also utilizing the remaining aluminosilicate residue for the production of construction materials—ultimately striving to achieve the Zero Waste principle.

In 2023, the Science Fund of the Republic of Serbia, under the Prisma Program in the field of technical and technological sciences, approved funding for the project **"Characterization and Technological Procedures for Recycling and Reusing of the Rudnik Mine Flotation Tailings - REASONING"** (2024-2026). The planned activities within the REASONING project are outlined in a written scheme presented in Figure 1.

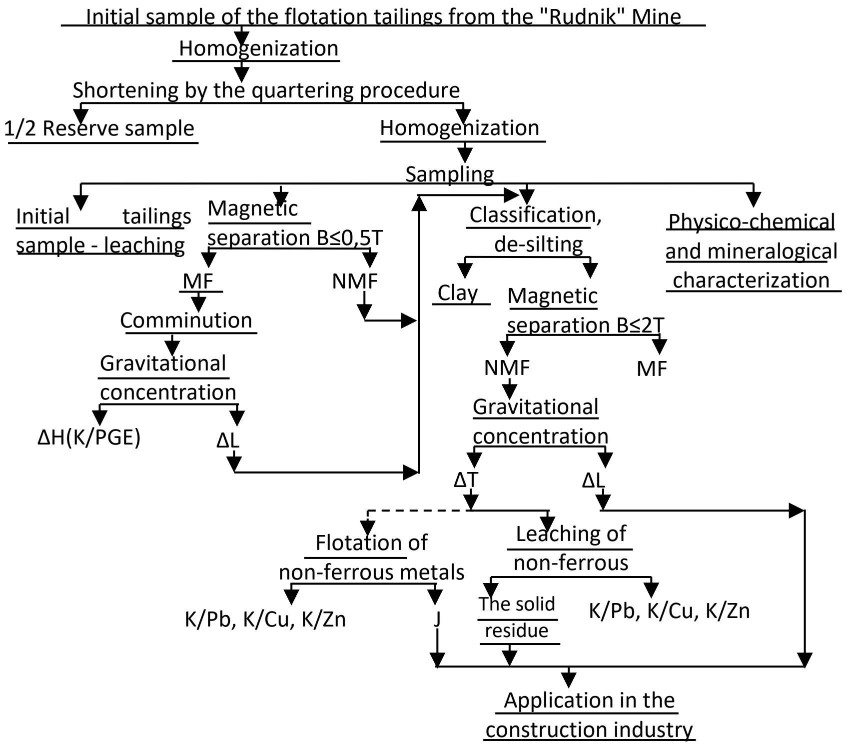

Figure 1 Written scheme of all activities and technological stages for the valorization of tailings according to Project-REASONING

At the end of 2024, two samples of flotation tailings were collected from the tailings pond at the Rudnik Mine. The first sample was obtained from four boreholes by drilling, and a composite sample was created from them. The second sample was taken from the surface of the tailings pond, and a composite sample was also prepared.

EXPERIMENTAL

Materials and methods

The physicochemical and mineralogical properties of two flotation tailings samples were analyzed: a composite sample from four boreholes with a total mass of 260 kg and a composite sample from the surface of the tailings pond with a total mass of 360 kg. These analyses were conducted at the Institute for Technology of Nuclear and Mineral Raw Materials (ITNMS).

Detailed physicochemical and mineralogical tests were performed on the initial flotation tailings samples from the Rudnik Mine to determine the essential characteristics of this secondary mineral raw material. Various physical and chemical properties and parameters were examined, including gross and hygroscopic moisture, specific mass, granulometric composition, bulk density in both loose and compacted states (SRPS EN 1097-3:2009), and specific weight (SRPS EN 1936:2009).

The particle size distribution of both tailings samples was determined using dry sieving with a Tyler series of sieves and laser diffraction analysis. Dry measurements were conducted using Sympatec Helos BR (H1506), Sucell 2; HELOS, while wet measurements were performed using a Malvern Mastersizer 3.0. Both devices operate on the principle of laser diffraction (refraction) of laser beams on micronized sample particles.

Mineralogical analyses were conducted using the following methods:

- X-Ray Diffraction (XRD)
- Reflected Light Microscopy
- Scanning Electron Microscopy (SEM)

These analyses provided qualitative and quantitative data on the mineralogical composition of the tailings samples. All analyses and tests were carried out according to internationally recognized standard methods for assessing the quality of mineral raw materials.

All chemical analyses of the tailings samples were performed using Inductively Coupled Plasma (ICP) spectroscopy at the Institute for Multidisciplinary Research in Belgrade.

RESULTS AND DISCUSSION

Physical characterization of the initial samples

As part of the physical characterization, the gross moisture content of the initial flotation tailings samples from the "Rudnik" Mine was first determined. Sample 1 (composite from boreholes) had a gross moisture content of Hg_1 = 0.32%, while Sample 2 (composite from the surface) had Hg_2 = 0.24%. The hygroscopic moisture content was w_1 = 15.42% for the borehole composite sample and w_2 = 10.61% for the surface composite sample.

The granulometric composition of both initial flotation tailings samples (composite from boreholes and composite from the surface) was determined using dry sieving with Tyler series sieves, covering sieve openings from 1 mm to 0.3 mm. All results obtained from different particle size distribution methods (sieving and laser diffraction) were consolidated. Based on these results, the upper size limit (d_{95}) and average particle diameter (d_{50}) were determined:

- Borehole sample: d_{95} = 0.45 mm, d_{50} = 0.175 mm
- Surface sample: d_{95} = 0.35 mm, d_{50} = 0.17 mm

The bulk density of the borehole tailings sample was:

- Loose state: ΔL = 1.64 g/cm³
- Compacted state: Δc = 1.93 g/cm³
- Specific mass: γ_1 = 3.19 g/cm³

For the surface tailings sample:

- Loose state: ΔL = 1.44 g/cm³
- Compacted state: Δc = 1.66 g/cm³
- Specific mass: γ_2 = 3.13 g/cm³

Chemical Composition of Initial Samples

The chemical composition of both flotation tailings samples from the Rudnik Mine was determined using ICP at the Institute for Multidisciplinary Research. Table 2 presents the major components, while Table 3 shows the minor components.

Table 2 Chemical composition of the tailings samples of "Rudnik"-Mine, main components

Components	Al	Ca	Mg	Cu	Fe	Zn	Pb	K
from boreholes, %	2.35	10.39	1.49	0.07	10.74	0.47	0.16	0.75
from surface, %	2.52	7.48	1.52	0.135	11.39	0.22	0.07	1.83

Table 3 Chemical composition of the tailings samples of "Rudnik"-Mine, minor components

Components	Ag	B	As	Ba	Bi	Cd	Co	Cr
from boreholes, g/t	9.2	500	914	172	29	40	35	328
from surface, g/t	7.4	712	3173	395	90	23	45	298

Mineralogical study of the tailings samples

As part of the mineralogical analysis, sample preparations were made for optical microscopy, SEM microscopy, and XRD analysis.

XRD Method – Research Results

Mineralogical Composition of the Borehole Sample - The qualitative mineralogical composition of the analyzed tailings sample from boreholes includes: quartz, calcite, K-feldspars, plagioclase, kaolinite, pyrite, pyrrhotite, arsenopyrite, sphalerite, chalcopyrite, galena, smectite/chlorite minerals, irregularly interstratified clays, and magnetite. Calcite and quartz are the most abundant minerals. Feldspars (K-feldspars and plagioclase) are present in smaller amounts. Among the ore minerals, pyrrhotite, arsenopyrite, and pyrite are the most abundant. Among the valuable minerals, sphalerite is the most common, followed by chalcopyrite in lower quantities, while galena is the least abundant. The diffractogram of the analyzed sample is presented in Figure 2.

Mineralogical Composition of the Surface Sample - The qualitative mineralogical composition of the analyzed tailings sample from the surface includes: quartz, calcite, K-feldspars, plagioclase, gypsum, kaolinite, pyrite, pyrrhotite, arsenopyrite, sphalerite, chalcopyrite, galena, smectite/chlorite minerals, irregularly interstratified clays, magnetite, dolomite, axinite, apatite, and bassanite. Feldspars (K-feldspars and plagioclase), calcite, and quartz are the most common minerals. Clay minerals and gypsum are present in smaller amounts. Among the ore minerals, pyrrhotite, arsenopyrite, and pyrite are the most abundant. Among the valuable minerals, sphalerite is the most common, followed by chalcopyrite in smaller amounts, while galena is the least abundant. All other minerals, if present, are below the detection threshold of this method. The diffractogram of the analyzed sample is presented in Figure 3.

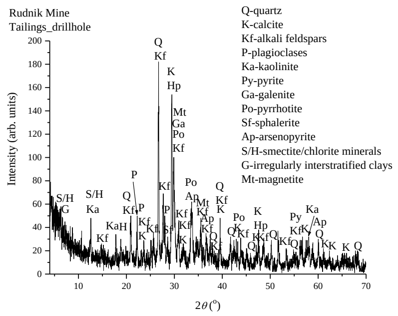

Figure 2 XRD diffractogram of the tailings from boreholes

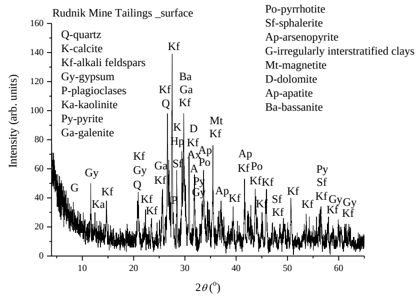

Figure 3 XRD diffractogram of the tailings from surface

Reflected Light and Scanning Electron Microscopy method

According to mineralogical analyses, the valuable minerals present in both samples (from boreholes and the surface) are sphalerite, chalcopyrite, and galena, primarily occurring as intergrowths or inclusions.

CONCLUSION

The determination of gross moisture content revealed that the tailings samples contained a significantly high water content—over 15% in the borehole sample and over 10% in the surface sample.

The granulometric analysis showed that both the borehole and surface tailings samples contained a significant proportion of coarse particles exceeding 0.5 mm.

According to the mineralogical analysis of the Rudnik Mine tailings samples:

- No platinum group elements (PGE) were detected in pyrrhotite.

- Visible gold/silver was observed.
- The most abundant minerals in the tailings are quartz and aluminosilicates.
- Multiple grains of native silver/gold were found in pyrrhotite, sphalerite, chalcopyrite, and galena.
- Sphalerite was identified as the most abundant non-ferrous metal mineral.

Based on the conducted analyses and the mineral composition of the tailings samples, it can be concluded that gravitational and magnetic separation methods can be effectively applied in the concentration process.

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