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POTENTIALITY OF OBTAINING MICA FLOTATION CONCENTRATE FROM KAOLINISED GRANITE

BADANIE MOŻLIWOŚCI OTRZYMYWANIA KONCENTRATU POFLOTACYJNEGO ZAWIERAJĄCEGO MIKĘ ZE SKAOLINIZOWANEGO GRANITU

This study shows the results of flotation concentration of mica minerals from kaolinised granite taken from the “Bašića bare” deposit – Kobaš, Srbac, The Republic of Srpska (B&H). Mineralogical composition of kaolinised granite is as follows: kaolinite, feldspar, quartz, and mica. After separating >0.630 mm, and <0.043 mm size class where kaolinite is concentrated, the rest is –0.630+0.043 mm class containing quartz, feldspar and mica. The mica concentrate was obtained by the flotation concentration, while feldspar and quartz were in the flotation underflow. According to the mineralogical analysis, the most abundant minerals are mica and chlorite/clays, while quartz and feldspar occur much less, and accessory minerals are represented in trace. The semi-quantitative mineralogical analysis obtained by the X-ray powder diffraction (XRPD) method of the mica concentrate amount to: mica ≈55%, chlorite/clays ≈35%, quartz ≈5%, feldspars (plagioclase and K-feldspars combined) ≈5%.

Keywords: kaolinised granite, muscovite, biotite, flotation concentration, mica concentrate

W pracy omówiono wyniki badania koncentratu poflotacyjnego zawierającego minerały z grupy miki ze skaolinizowanego granitu pozyskiwanego ze złoża Bašića bare w Kobas, w Republice Serbskiej (B&H). Skaolinizowany granit zawiera następujące minerały: kaolinit, skałen, kwarc i mika. Po wyodrębnieniu klas ziaren o wymiarach >0.630 mm i <0.043 mm, zawierających największe ilości kaolinitu, pozostały produkt stanowi klasa ziaren o wymiarach –0.630+0.043, zawierających kwarc, skałen oraz mikę. Koncentrat zawierający mikę otrzymano z koncentratu poflotacyjnego, skałen i kwarc zebrano w produkcie dolnym procesu flotacji. W oparciu o analizy składu mineralogicznego stwierdzono, że minerałami występującymi w największych ilościach są mika oraz chloryty/iły, kwarc oraz skalenie występują w znacznie mniejszych ilościach podczas gdy pozostałe minerały występują jedynie w ilościach śladowych. Przeprowadzona analiza mineralogiczna koncentratu miki w ujęciu ilościowym w oparciu

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o wyniki rentgenowskiej dyfraktometrii proszkowej (*X-ray powder diffraction – XRPD*) ujawniła że skład jego jest następujący: mika $\approx 55\%$; chloryty/ity $\approx 35\%$; kwarc $\approx 5\%$; skalenie (łącznie plagioklasy i skalenie potasowe) $\approx 5\%$.

Słowa kluczowe: skaolinizowany granit, muskowitz, biotyt, koncentrat w procesie flotacji, koncentrat zawierający mikę

1. Introduction

1.1. Mica minerals

Micas are group of silicate minerals belonging to the rock-forming minerals. They are incorporated in a vast number of Earth's crust rocks, and it is considered that they participate in its structure with 4 %. They represent important part of large group of igneous rocks created by cooling of hot melted magma. Micas can also be formed by the process of metamorphism under the influence of high pressure, heat and hydrothermal solutions (Pavlica & Draškić, 1997). According to the chemical composition micas are hydrated aluminosilicates of following elements: K, Na, Mg, Fe, Li, Ce, Rb with OH (hydroxy) group. The most abundant minerals are:

- muscovite $\text{KAl}_2(\text{Al}, \text{Si}_3\text{O}_{10})(\text{OH})_2$,
- biotite $\text{K}(\text{Mg}, \text{Fe})_3(\text{Al}, \text{Si}_3\text{O}_{10})(\text{OH})_2$,
- phlogopite $\text{KMg}_3(\text{Al}, \text{Si}_3\text{O}_{10})(\text{OH})_2$,
- illite $\text{K}_x\text{Al}_4(\text{Si}_{8-x}\text{Al}_x)\text{O}_{20}(\text{OH})_4$,
- zinnwaldite $\text{KLiFe}^{2+}\text{Al}(\text{AlSi}_3\text{O}_{10})(\text{F}, \text{OH})_2$, paragonite $\text{NaAl}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$,
- lepidolite $\text{KLi}_2\text{Al}(\text{Si}_4\text{O}_{10})(\text{F}, \text{OH})_2$.

Micas have perfect cleavage, pearly luster (Fig. 1a), hardness 2.5, and they are all monoclinic (Fig. 1b) (link 1 and 2). They have various colors, from colorless to yellow, red, green, brown and black. USA, Canada, India, Madagascar, Russia, Brazil and SAR are countries with the largest mica deposits. Because of their features micas have great practical application. Micas are excellent non-conductors, they neither conduct heat nor electricity. Based on these properties, they are used in the production of electrical devices and materials resistant to fire.

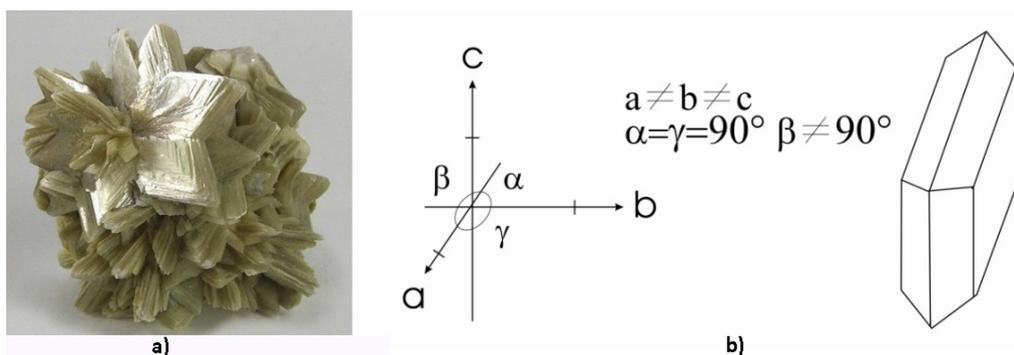


Fig. 1. a) Silky luster of muscovite (link 1) and b) monoclinic crystal habitus (link 2)

According to the mineralogical analyses, the main micas of this study are muscovite and biotite. Considering that, the following study gives overview of the properties for these two minerals in detail.

1.1.1. Muscovite and Biotite

Chemical formula of muscovite is $\text{KAl}_3\text{Si}_3\text{O}_{10}(\text{OH})_2$. It is white with silky (pearly) luster, perfect cleavage, hardness 2.5, monoclinic (S.G. $C2 (5)$). Muscovite usually occurs in flaky aggregates, originates from igneous processes and can be also formed by metamorphism of clay minerals. It is very resistant to the weathering processes and tends to concentrate as a fine grained sericite. The crystal structure of muscovite is composed of two tetrahedral sheets and one octahedral sheet located in-between (so-called 2:1 or T-O-T layer Fig. 2b). Since the octahedral cation is Al^{3+} it belongs to dioctahedral phyllosilicates.

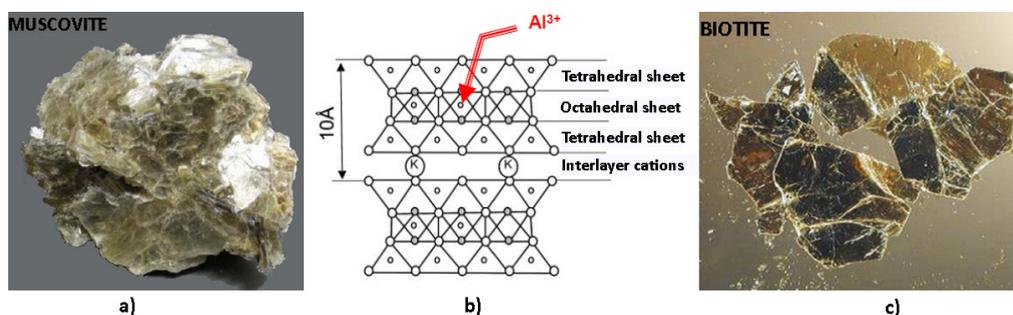


Fig. 2. a) Muscovite, b) The crystal structure of muscovite, and c) Biotite (link 3)

Chemical formula of biotite is $\text{K}(\text{Mg},\text{Fe})_3(\text{Al},\text{Si}_3\text{O}_{10})(\text{OH})_2$. It is dark brown to black (Fig. 2c), perfect cleavage and elasticity in thin lamina, hardness 2.5, monoclinic (S.G. $C2/m (12)$). It originates from igneous and pegmatitic-pneumatolytic processes (Fe-rich). It also occurs in metamorphic rock of regional and contact metamorphism. In hydrothermal conditions biotite most commonly alters into chlorites, while during the weathering processes it loses iron and alkali metals, (link 2). The crystal structure of biotite is also composed of 2:1 or T-O-T layers. The only difference is in cation of octahedral sheet, namely Mg and Fe^{2+} are in biotite instead of Al in muscovite. Therefore, biotite belongs to trioctahedral phyllosilicates.

1.2. Mineral processing of micas

A vast number of researchers have been engaged in processes of obtaining mica by flotation concentration (Vidyadhar & Hanumantha, 2007; Bogdanov & Podnek, 1959a, 1959b; Polkin, 1987; Gaudin, 1957). In practice, there are two methods of obtaining mica products: hand sorting (Pavlica & Draškić, 1997) and flotation concentration (Pavlica & Draškić, 1997; Wang et al., 2014; Hanumantha & Forsberg, 1997). The hand sorting is a procedure that is performed after the mining of the ore and its sieving. The flotation concentration is applied in pegmatite ore when mica is accompanied with feldspar and quartz, but much less abundant (Raymond, 1997;

Sekulic et al., 2004; Akzochemie 1986; Salmawy et al., 1993; Xu et al., 2013). This technique is significant because it is possible to obtain three high-quality concentrates of mica, feldspar, and quartz with the application of appropriate collectors and flotation conditions (Hanumantha & Forssberg, 1993; Hanumantha & Forssberg, 1995).

1.3. Market demands for the use of mica concentrates obtained by flotation

Mica manufacturers form assortment according to quality defined by chemical composition and gives them the appropriate labels for easy presentation with a recommendation for which purposes they can be used (link 4; link 5). Moreover, besides chemical composition, physical properties are also important for quality determination. It is particularly stated by the mica manufacturers for use as functional filler in different areas. Namely, the parameters that determine the quality of the obtained mica concentrate, besides the type of active material (refers to the mica mineral species), are whiteness, density and specific surface (link 6).

Moreover, following mica properties are essential in construction: chemical inertness, high thermal stability, elastic, but firm particles, high whiteness, high dielectric strength and electrical insulation (link 7). Generally, in the absence of mica quality standards obtained by flotation, users experientially come to the conclusion which quality suits them for their own use.

2. Experimental

2.1. Material

The sample used for this study is kaolinised granite from the “Bašića bare” – Kobaš, Srbac, Bosnia and Herzegovina.

2.2. Methods and reagents

The chemical analyses were obtained by Atomic Absorption Spectrophotometer (AAS) “AAS Analyst 300” (“Perkin Elmer“-SAD”).

The XRPD patterns of powdered samples were obtained using a “Philips” PW-1710 automated diffractometer with a Cu tube operated at 40 kV and 30 mA (“Philips”-the Netherlands). The instrument was equipped with a diffracted beam curved graphite monochromator and a Xe-filled proportional counter. The diffraction data were collected in the 2θ Bragg angle range from 4 to 65°, counting for 3 s at every 0.02° step. The divergence and receiving slits were fixed to 1 and 0.1, respectively. All the XRPD measurements were performed at room temperature in a stationary sample holder.

Qualitative mineralogical analyses were obtained using “Carl-Zeiss” polarizing microscope by the immersion (xylol) method, model “JENAPOL-U” equipped with 10X, 20X, 50X, 100X objectives and a system for a photomicrography (“Axiocam105 color” camera and “Carl Zeiss AxioVision SE64 Rel. 4.9.1.” software package with „Multiphase” module, “Carl-Zeiss”, Jena-Germany).

Sulfuric acid (5 %) was used for pH adjustment in flotation tests. In the flotation process AERO 3030C was used as a collector (“Cytec”-USA) (link 8; Yang et al., 2016).

2.3. Procedure of obtaining mica concentrate

In order to obtain the mica concentrate, studies of the raw sample from the “Bašiča bare” deposit were carried out (schematic diagram in Fig. 3a).

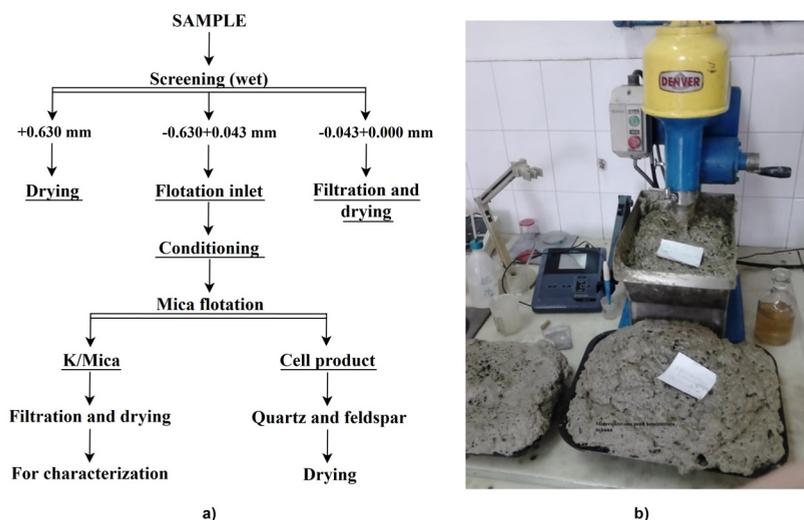


Fig. 3. a) Schematic diagram of the procedure of obtaining mica concentrate from kaolinised granite of the “Bašiča bare” deposit and b) flotation product

Screening of the sample of kaolinised granite on 0.630 and 0.043 mm mesh screens was performed before the flotation process. Three size classes were obtained: +0.630; -0.630+0.043 and -0.043 mm. Inlet for process of flotation concentration and mica eliquation was the -0.630+0.043 mm size class. Mica minerals usually occur as fine grained in pegmatite rocks and they are much less abundant in regards to quartz and feldspars. Therefore, it is considered that these should be separated by flotation, using cationic collectors (Hanumantha et al., 1990; Hanumantha et al., 1995). Thus, conditions are optimal for obtaining quality quartz and feldspar concentrates from a flotation cell product (Pavlica & Draškić, 1997).

2.4. Reagent regime and mica flotation conditions of the -0.630+0.043 mm size class

Experimental conditions for mica flotation are given in Table 1.

TABLE 1

Experimental conditions of the mica flotation concentration test

Parameter	Solid phase content, %	Time, min	5% H ₂ SO ₄ , ml	AERO 3030 C, g/t
Test phase				
Conditioning for mica flotation	50	8	22 ml / pH 3.26	400 g/t
Mica flotation	28	6	3.26	—

Flotation tests were carried out in “Denver” flotation cell. The solid : liquid ratio in a suspension was 50:50. Before the flotation process, the suspension was conditioned (mixed) for 8 min with the addition of 22 ml of H₂SO₄ (5%) in order to achieve pH of 3.26 of the solution. During the flotation process 400 g/t of AERO 3030 C cationic collector was added. After the conditioning the pulp was diluted by adding water (solid phase content was 28%), and then the process of flotation and separation of mineralized mica rich foam started (Fig. 3b) in duration of 6 min. The obtained mica concentrate was further analyzed and the results are presented below.

3. Results and discussion

After the experiments performed, in the following text we will show the results and discussion.

3.1. Screening and flotation mass balance

Mass balance is shown in Table 2.

TABLE 2

Sample „Bašića bare“ mass balance

Raw sample	M, g	% share related to dry raw sample	% share related to size class -0.630+0.043 mm
Wet	19 796.00		
Dry	17 597.00	100.00	
Raw material screening			
For screening		100.00	
+0.630 mm		24.05	
-0.630+0.043 mm		59.58	
-0.043 mm		16.37	
Flotation of mica			
For flotation -0.630+0.043 mm		59.58	100.00
K/mica		6.11	10.25
Cell product		53.47	89.75

As to Table 2, percent of mica concentrate relative to raw material is 6.11%, and 10.25% relative to flotation treated size class.

3.2. Chemical composition

The chemical composition of initial sample of kaolinised granite from the “Bašića bare” deposit by size classes is shown in Table 3.

Results of chemical analysis of kaolinised granite taken from the “Bašića bare” deposit are showing that in every size class SiO₂ and Al₂O₃ oxides are most represented. Forasmuch as these two oxides are the main constituents of kaolinite mineral (Al₂O₃x2SiO₂x2H₂O), it is indicated

TABLE 3

Chemical composition of kaolinised granite from the “Bašića bare” deposit by size classes

Oxides	mm	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	TiO ₂	*
%	Inlet	69.50	17.89	1.24	0.97	0.21	4.76	2.58	0.22	2.51
	+0.630	84.73	8.18	0.28	0.38	0.02	5.31	0.70	0.18	0.18
	-0.630+0.043	70.20	16.82	1.31	1.15	0.22	5.17	3.38	0.19	1.42
	-0.043+0.00	50.67	32.13	2.03	0.942	0.37	2.70	1.76	0.40	8.93

* loss of ignition

that raw material contains kaolinite. For this reason raw material is justified named as kaolinised granite. Furthermore, of three obtained size classes, the -0.63+0.043 mm size class was preferred for the process of flotation concentration in order to separate mica concentrate.

The obtained mica concentrate (according to schematic diagram from Fig. 3) was chemically analyzed, and the results are given in Table 4.

TABLE 4

Chemical composition of the mica concentrate

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	TiO ₂	LiO ₂	*
%	52.70	24.60	6.89	0.84	2.07	5.72	0.94	0.99	0.16	6.96

* loss of ignition

Based on the results of chemical analysis of the mica concentrate obtained by the procedure shown in this paper, the presence of SiO₂, Al₂O₃, K₂O and Fe₂O₃ can be determined with a content of 52.70%, 24.6%, 24.60% and 6.89% respectively. This indicates the dominant presence of mica in the concentrate, bearing in mind that Si and Al are the main building constituents of the crystal lattice of mica minerals. Other oxides indicate the presence of chlorite, quartz and feldspar. This was confirmed by the results of X-ray analysis, as well as the results of mineralogical analysis.

Chemical composition of the mica flotation concentrate from this study conforming to the literature data (link 5; Jia et al., 2018; Schoeman, 1989). Some mica manufacturers, depending on the chemical composition of their product assortments, give recommendations for which industrial branches (industry of paints, plastics, rubber and automotive industry) can be used (link 5). Considering the fact that the mica concentrate of this study has similar chemical composition (both chemical and mineralogical) as which require the mentioned industry it can be concluded that this concentrate can have potential application.

In order to have a better insight into quality of the mica concentrate which was made by the flotation in the laboratory conditions, the authors of this study proposed several mica concentrates obtained in industrial conditions in Serbia:

- „Feldspat“ Bujanovac: SiO₂ 47.85%; Al₂O₃ 36.98%; Fe₂O₃ 1.02%; CaO 0.20%; Na₂O 1.14%; K₂O 8.62%.
- „Feldspat“ Prokuplje: SiO₂ 45.00%; Al₂O₃ 37.00%; Fe₂O₃ 2.00%; CaO 0.20%; Na₂O 0.80%; K₂O 8.10%.
- „Vranjska Banja“: SiO₂ 44.95%; Al₂O₃ 37.40%; Fe₂O₃ 2.12%; CaO 0.23%; Na₂O 0.60%; K₂O 8.25%, (Milošević, 1998).

3.3. X-Ray Powder Diffraction (XRPD) analysis of the mica concentrate sample

In the analyzed sample of the mica concentrate obtained by flotation following mineralogical composition was determined: mica, chlorites, kaolinite, quartz and feldspars. The most abundant minerals are mica, then chlorites and kaolinite, while quartz and feldspars are far less represented. Plagioclases are more represented than K-feldspars. The semi-quantitative mineralogical composition is as follows: mica $\approx 55\%$, chlorites/kaolinite (combined) $\approx 35\%$, quartz $\approx 5\%$, feldspars (plagioclase and K-feldspars combined) $\approx 5\%$. The diffractogram of the analyzed mica concentrate sample is given in Figure 4.

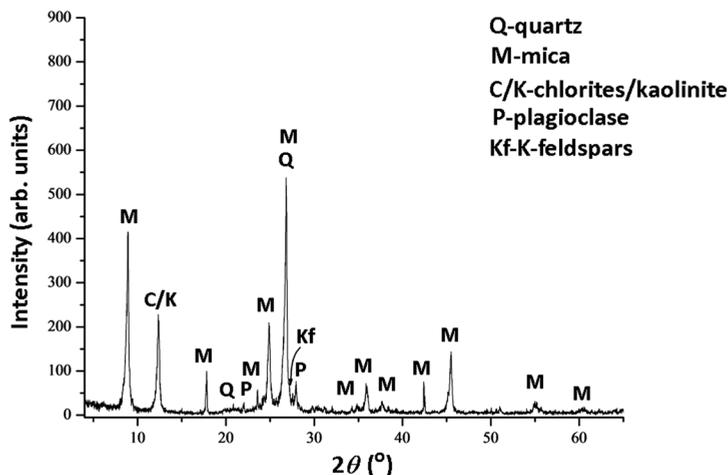


Fig. 4. Diffractogram of the mica concentrate sample

3.4. Microscopic analysis of the mica concentrate sample

According to the qualitative mineralogical analysis the following composition was determined: mica (biotite and muscovite), chlorites, clays, quartz, plagioclase, K-feldspars, opaque minerals, amphiboles, tourmalines, zircon. The most abundant minerals are mica, then chlorites

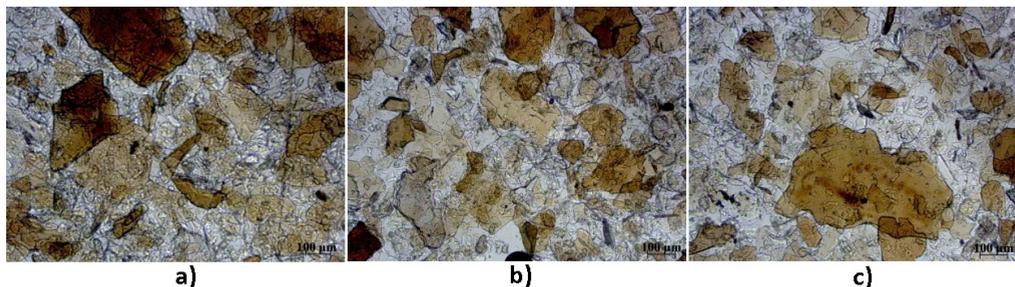


Fig. 5. Mica mass in the flotation concentrate sample (transmitted light, air, // Nicols)

and clays, while quartz and feldspars are far less represented. Accessory minerals (opaque minerals, amphiboles, tourmalines and zircon) appear in trace. Biotite is more represented than muscovite among mica. It is important to emphasize that chlorites are secondary minerals formed by the alteration of biotite (chloritization process). Microphotographs of the mica concentrate sample are shown in Figure 5.

4. Conclusion

According to the obtained results of the kaolinised granite from the “Bašića bare” deposit – Kobaš, Srbac, Bosnia and Herzegovina, the following can be concluded:

- The results of chemical analyses of all three size classes have shown that SiO_2 and Al_2O_3 are dominant, while other oxides are present significantly less.
- The results of chemical analyses of the mica concentrate obtained by flotation have revealed high contents of SiO_2 , Al_2O_3 , K_2O and Fe_2O_3 pointing out that dominant minerals in the concentrate are mica.
- According to the microscopic analyses (in transmitted light) mica (biotite and muscovite) are the most abundant minerals, then chlorites/clays, while quartz and feldspars are the least represented. Accessory minerals are present in trace.
- Semi-quantitative mineralogical XRPD analysis is as follows: mica $\approx 55\%$, chlorites/kaolinite (combined) $\approx 35\%$, quartz $\approx 5\%$, feldspars (plagioclase and K-feldspars combined) $\approx 5\%$.

According to the presented results it is possible to obtain the mica concentrate from kaolinised granite from “Bašića bare” deposit by flotation concentration. However, what is necessary to do is sizing thus providing a satisfactory ore size range that can be flotated. In this case it is $-0.630+0.043$ mm containing fine grained mica, while $-0.043+0.00$ mm contains clays (kaolinite). After the separation of mica concentrate, quartz and feldspars enrich in the flotation underflow, which can be further treated and obtained as particular products. On the basis of comparison of quality with literature data it is confirmed that the obtained mica concentrate may be commercially usable. Namely, it can find its application in the paint, rubber, plastic and automotive industry.

The high mica content of up to 95-96% is a necessary demand for some plastic and synthetic products (powdered mica in mixture with gypsum for cement boards). Having in mind the fact that the obtained mica concentrate contains up to 55%, it is necessary to purify the rough concentrate several times in the rare pulp in order to increase its content. This will be the subject of further research by the authors of this study in order to obtain the highest quality concentrate.

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