РУДАРСТВО И ГВОЛОГИЈА ДАЦАС

међународни симпозијум (

MINING AND -/ GROLOGY TODAY

international symposium

Уредници / Editors S. Vujić, M. Radosavljević, S. Polavder, S. Maksimović

Beorpait //Belgrade







СІР - Каталогизација у публикацији - Народна библиотека Србије, Београд

55(082)622(082)

grafx). - [11], 362 стр. : млустр. ; 24 ст. Балканска академија рударских наука: Академија инжењерских наука Србије, 2017 (Beograd : Colorsymposium, Belgrade ; [урсдници, editors Sloboсюлюзијум, Београд, 18-20.09.2017. = Mining and Geology Today / [proceedings]; International геологија данас : [зборник радова] /Међународни геологија данас" (2017 ; Београд)Рударство и МЕЂУНАРОДНИ симпозијум "Рударство dan Vujić et al.]. - Београд: Рударски институт:

Тираж 300. - Текст ћир. и лат. - Стр. [6]: Уводна реч = Foreword / уредници = editors. - Библиографија уз сваки рад. - Apstracts.

ISBN 978-86-82673-13-2 (PM)

а) Геологија - Зборници b) Рударство - Зборници

COBISS.SR-ID 244649484

Prof. dr Slobodan Vujić Dr Svetlana Polavder Dr Milinko Radosavljević Уредници / Editors Зборник радова / Proceedings MINING AND GEOLOGY TODAY РУДАРСТВО И ГЕОЛОГИЈА ДАНАС

Dr Svetomir Maksimović

Организатори и издавачи / Organizers and publishers









ACADEMY OF ENGINEERING SCIENCES SERBIA АКАДЕМИЈА ЈИСКИЊЕРСКИХ НАУКА СРБИЈЕ

Executive organizer and publisher Извршни организатор и издавач

E. aдреса / e. mail: office@ribeograd.ac.rs Aдреса / adress: Batajnički put 2, 11080 Beograd Mining institute Ltd Belgrade Рударски институт доо Београд

director of the Mining institute директор Рударског института Dr Milinko Radosavljević, Za izdavače / For the publishers

Graphic design, text capture and processing by Grafički dizajn i priprema za štampu

Mining institute Ltd Belgrade Рударски институт доо Београд

Godina izdanja / Issued in: 2017 Stampa / Printed by: Colorgrafx, Beograd Tiraž / Circulation: 300

CIP 55(082), 622(082); COBISS.SR-ID 244649484

ISBN 978-86-82673-13-2(РИ)

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MMXVII БЕОГРАД BELGRADE

KARAKTERIZACIJA PRIRODNIH MATERIJALA U PROCESU PRIPREME RUDE GVOŽĐA U RUDNIKU OMARSKA

CHARACTERIZATION OF NATURAL RAW MATERIALS IN THE PROCESSING OF IRON ORE FROM OMARSKA MINE

DOI:10.25075/SI.2017.31

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Сажетак: У овом раду су проучаване карактеристике узорака примарних природних сировина из рудника Омарска (Босна и Херцеговина). Ови материјали су означени као: "лимонитска руда", "кварц" и "глина", и представљају главне компоненте које се јављају приликом прераде руде гвожђа у руднику Омарска. Како би се прецизно одредиле присутне минералне фазе и њихови хемијски састави, примењене су анализе рендгенске дифракције на праху (XRPD), инфрацрвена спектроскопија (FTIR) и скенирајућа електронска микроскопија-енергетски дисперзивна спектроскопија X-зрацима (SEM-EDS). Резултати рендгенске дифракције су показали да се узорци "лимонитске руде" састоје углавном од гетита који доминира над хематитом, и са мањим количинама магнетита, кварца и минерала глина. Узорци "кварца" се састоје углавном од кварца који доминира над мањим количинама минерала глина, фелдспатима и хематитом. Узорци "глина" састоје се углавном од кварца и минерала глина који доминирају над мањим количинама фелдспата, амфибола, гетита и хематита. Минерали глина су идентификовани углавном као илит-серицит који преовлађује над каолинитом, као и хлорит који се само јавља спорадично. FTIR и SEM-EDS анализе су у врло доброј сагласности и у великој мери потврђују резултате добијене XRPD методом.

Кључне речи: КАРАКТЕРИЗАЦИЈА, ЛИМОНИТНА РУДА, КВАРЦ, ГЛИНА.

Abstract: In present paper there were studied characteristics of the primary natural raw material samples from Omarska mine (Bosnia and Herzegovina). These materials are labeled as: "limonite ore", "quartz" and "clay", and they are main components which occur in the processing of iron ore in Omarskamine. In order to precisely determine the present mineral phases and their chemical compositions, it was performed XRPD, FTIR and SEM-EDS analysis. The XRPD results showed that the "limonite ore" samples are composed of major goethite which dominate over hematite, and with minor contents of magnetite, quartz and clay minerals. The "quartz" samples are composed of major quartz which dominate over minor contents of clay minerals, feldspars and hematite. The "clay samples" are

composed of major quartz and clay minerals which dominate over minor contents of feldspars, amphiboles, goethite and hematite. The clay minerals were identified as mostly illite-sericite which prevails over kaolinite, and with chlorites which appears only sporadically. The FTIR and SEM-EDS analysis are in very good agreement and mainly confirms the results obtained by XRPD method.

Key words: CHARACTERIZATION, LIMONITE ORE, QUARTZ, CLAY.

INTRODUCTION

The iron ore deposit "Omarska" (Bosnia and Herzegovina) is located in the northeast part of the "Sana paleozoic", or the Ljubija metalogenetic region, and in the Omarska-Prijedor field between the cities of Prijedor and Banja Luka (Figure 1). Prijedor and the "Ljubija" mines are about 20 km away. It was discovered during the regional geological research of the wider area of Omarska started in 1968. The iron ore deposit "Omarska" consists of the iron ore body "Buvač", which is still in exploitation, and the iron ore bodies "Jezero" and "Mamuze", where the exploitations were completed in 2008. At this deposit there are the following types of iron ores: carbonate (massive dark blue siderite, dark siderite, gray grained siderite) and oxide (compact limonite, kidney limonite, powdered limonite) [1].

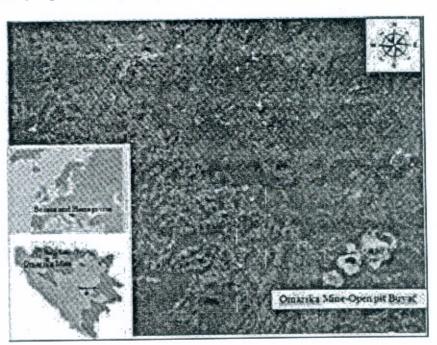


Figure 1, Geographically maps of location of the "Omarska" mine

Processing of ore in Omarska mine is carried out by following methods: washing, sieving, grading and magnetic concentration. The morphological and structural characteristics of limonite ore from the "Buvač" cause the production of a significant amount of small fractions,

with a volume below 25 μ m, in exploitation. Any further process increases the proportion of the range from which the existing plant for the mineral processing in Omarska is not possible to derive concentrate of satisfactory quality, and as such, represents a tailings pond.

Crude ore were firstly be washed in the washing drum, and then continues to wet sieving, grinding and grading by spiral classifier and the mechanical hydrocyclones. The larger classes from hydrocyclone go to thickener and further to magnetic separation. The smaller classes, namely overflow from hydrocyclone (classes -25 µm) go to tailing in forms of sludge. On the one hand, this sludge is an environmental problem, but on the other hand, it contains significant amounts of limonite, which could be eventually used. Studies that we have conducted are focused on evaluation of useful minerals from hydrocyclone overflow. One of the promising methods of the concentration of useful minerals from the sludge is the application of selective flocculation as a prior stage in order to increase the efficiency of the process magnetic ore concentration [2-5].

Our previous investigations [6] shown that sludge samples were composed mostly of goethite-FeO(OH) and quartz (SiO₂), which prevails over clay minerals. The results also indicate quartz as the main impurity. Particles size distribution [7] shows that almost 70 mass% are of -7 µm size class, which further confirms that it is not possible to use conventional methods of mineral processing without the use of some prior stage. The selective separation of impurities and useful minerals in complex system such as the investigated sludge, is very difficult and depend on the individual components of the sludge and their behavior. The removing of clay or quartz from fine iron ores was earlier investigated [8-13], but knowledge about fine limonite ore processing are relatively poor.

In order to better understand this complex system, our primary aim in present paper was characterization of the primary natural raw material samples from Omarska mine, labeled as: "limonite ore", "quartz" and "clay". These materials are the main components of the sludge which is further generated during the iron ore magnetic concentration in Omarska mine.

MATERIALS AND METHODS

There were studied two series of the primary natural raw material samples (I and II) and labeled as "limonite ore", "quartz" and "clay". These samples were hand-picked and carefully selected for further analysis.

The X-ray powder diffraction (XRPD) patterns of samples I were obtained by a Philips PW-1710 automated diffractometer using a Cu tube (λ =1,54060Å) operated at 40 kV and 30 mA. The instrument was equipped with a curved graphite monochromatic diffraction beam, and Xe-filled proportional counter. The diffraction data were collected in the 20 a Bragg angle ranges from 4° to 65°, counting for 0.25 second at every 0.02° steps. The divergence and receiving slits were fixed at 1° and 0.1 mm, respectively. The XRPD measurements were performed ex situ at room temperature in a stationary sample holder. The alignment of the diffractometer was checked by means of a standard Si powder material. Identification of the present mineral phases was done with comparison of the interplanar spacings (d) and relative intensities (I) with the literature data, which is corresponding card from the International Centre for Diffraction Data, the Powder Diffraction File ICDD-PDF database (www.icdd.com).

Fourier Transform Infrared (FTIR) spectroscopy study of samples I and II was performed using Thermo Nicolet 6700, at spectral area 4000-400 cm⁻¹, with resolution of 2 cm⁻¹ and with 32 scans. Samples were powdered in agate mortar. For the spectra recording it was used KBr technique: in 150 mg of powdered KBr it was added 1 mg of the studied sample, then it was homogenized and tablets were made under specific pressure and vacuum. Identification of the present mineral phases was done with comparison of the IR spectra with the literature data for the corresponding mineral phases.

Scanning Electron Microscope-SEM, studies for samples I were carried out in a JEOL JSM 6610LV coupled with Energy Dispersive Spectrometry-EDS by EDS detector model X-Max Large Area Analytical Silicon Drift connected with INCA Energy 350 Microanalysis System.

XRPD and SEM-EDS studies of samples II were performed in the laboratory Global Research and Development, Mining and Mineral Processing, Maizières-lès-Metz, France. XRPD data were obtained on BRUKER D2 PHASER by using software DIFFRAC.EVA V4.0, whereas SEM-EDS data were obtained on HITACHI "TM 3000" Tabletop Microscope with High Sensitivity BSE Detector.

RESULTS AND DISCUSSION

XRPD study

The obtained results of the XRPD studies were shown at Figures 2 and 3, and Table 1.

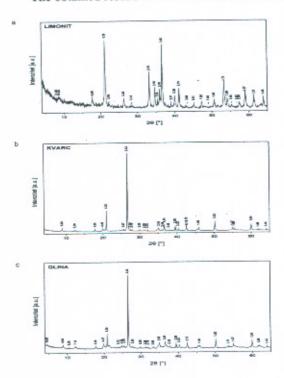


Figure 2, XRPD patterns of the series I samples: a). limonite ore; b). quartz; and c). clay.

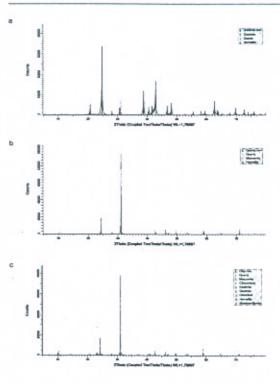


Figure 3, XRPD patterns of the series II samples: a). limonite ore; b). quartz; and c). clay.

Table 1, Identified qualitative and semi-quantitative mineral compositions of the series I and II samples obtained by XRPD studies.

Sample	Limonite ore I	Limonite ore II
Qualitative and semi-quanti- tative mineral composition	Goethite.	Goethite, hematite and quartz.
Comment	Sample is almost complete- ly consist by mono-mineral goethite with minor contents of hematite, magnetite and clay minerals.	Goethite (~86%) which dominate over hematite (~10%) and with minor contents of quartz (~4%).
Sample	Quartz I	Quartz II
Qualitative and semi-quanti- tative mineral composition	Quartz, clays (of illite-seric- ite - kaolinite composition) and feldspars.	Quartz, illite-sericite and he- matite.
Comment	Quartz (~91%) which dom- inates over clay minera's (~6%) and with minor con- tents of feldspars (~3%).	Quartz (~93%) which dom- inates over illite-sericite (~6%) and with minor con- tents of hematite (~1%).
Sample	Clay I	Clay II

Qualitative and semi-quanti- tative mineral composition	Quartz, clays (of illite-sericite - kaolinite composition),feld- spars and amphiboles.	Quartz, clays (of illite-sericite - kaolinite - chlorite compo- sition), goethite, chloritoid and hematite.
Comment	Quartz (~88%) which dominates over clay minerals (~7%), and with minor contents of feldspars (~3%) and amphiboles (~2%), as well.	Quartz (~50%) and clay min- erals (~44%) which dom- inates overminor goethite (~4%),chloritoid (~2%) and hematite.

By comparison of the presented results from series I and II (Figures 2 and 3, Table 2), it can be clearly seen that in all of the adequate samples (i.e. with same label) there are with more-less similar qualitative mineral composition determined. On the other hand, semi-quantitative mineral compositions are mostly various between series I and II, except for the quartz samples which are almost the same.

The "limonite ore" samples are composed of major goethite-FeO(OH) which dominate over hematite (Fe₂O₃), and with minor contents of magnetite (Fe₃O₄), quartz (SiO₂) and clay minerals. The "quartz" samples are composed of major quartz which dominate over minor contents of clay minerals, feldspars and hematite. The "clay samples" are composed of major quartz and clay minerals which dominate over minor contents of feldspars, amphiboles, goethite and hematite.

Identification of clay minerals indicate that they are mostly of illite-sericite and kaolinite composition, with chlorites which appears only sporadically. Sericite is mainly of the muscovite mica type.

FTIR study

The obtained results of the FTIR studies were shown at Figures 4 and 5, and Table 2.

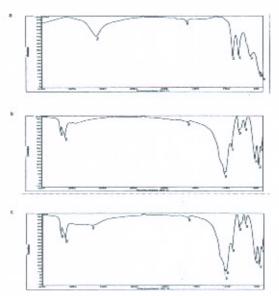


Figure 4, FTIR patterns of the series I samples: a). limonite ore; b). quartz; and c).

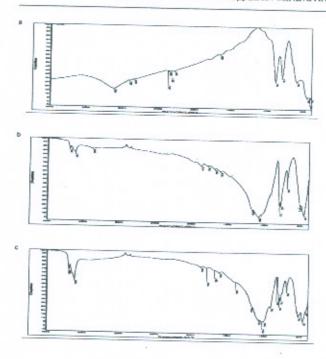


Figure 5, FTIR patterns of the series II samples: a). limonite ore; b). quartz; and c). clay.

Table 2, Identified qualitative and semi-quantitative mineral compositions of the series I and II samples obtained by FTIR studies.

Sample	Limonite ore I	Limonite ore II
Identified mineral composition	Goethite (3110, 897 and 800cm ⁻¹) and magnetite? (607 and 410cm ⁻¹).	Goethite (3124, 893and799cm ⁻¹),he- matite (602and 408cm ⁻¹) and quartz (799cm ⁻¹).
Sample	Quartz I	Quartz II
Identified mineral composition	Quartz (1160, 796 and 693cm ⁻¹) and clay minerals (clays: 3623, 1631, 1033, 1008 and 913cm ⁻¹ ; illite-sericite: 535cm ⁻¹ ; and ka- olinite: 3699, 471 and 433cm ⁻¹).	Quartz (1166, 798and 694cm ⁻¹) and clay minerals (clays: 3622, 1614 and 1083cm ⁻¹ ; illite-sericite: 778 and 535cm ⁻¹ ; and kaolinite: 3699 and 462cm ⁻¹).
Sample	Clay I	Clay II
Identified mineral composition	Quartz (1160, 797 and 694cm ⁻¹) and clay minerals (clays: 3623, 1632, 1032, 1008 and 913 cm ⁻¹ ; illite-scricite: 534 cm ⁻¹ ; and ka- olinite: 3699, 471 and 429cm ⁻¹).	Quartz (1165, 798and 694cm ⁻¹) and clay minerals (clays: 3622, 1620, 1032, 1010 and 913 cm ⁻¹ ; illite-sericite: 778 and 535 cm ⁻¹ ; and kaolinite: 3699, 471 and 435 cm ⁻¹).

Identification of the present mineral phases were done using the adequate references [14-16]. The obtained results are in very good agreement and mainly confirms XRPD results of the previously determined qualitative and semi-quantitative mineral compositions. Some differences between series I and II, especially for the "limonite ore" samples, are also clearly visible with this method.

SEM/EDS study

Scanning electron microscope (SEM) coupled with Energy Dispersive Spectrometry (EDS) was used for morphological, structural and chemical composition study of natural series I and II samples of "limonite ore", "quartz" and "clay". For defining the more precise elemental and mineral composition, it was done analysis of selected points on the surface. The results were shown at Figures 6-9 and Tables 3-8.

As it is well known, one of the most limiting factors for determination of the mineral phases from EDS data is its inability to measure hydrogen, due that goethite and clay-mica minerals contain appreciable amounts of OH groups and/or H_2O . According to that, we can only speculate about these possible phases. However, we believe that the obtained SEM-EDS results are also in very good agreement and mainly confirms XRPD results of the previously determined qualitative and semi-quantitative mineral compositions presented in this paper.

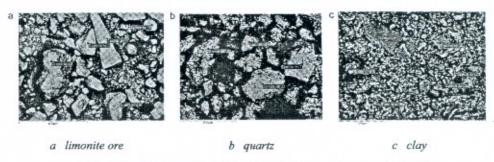


Figure 6, SEM images of the series I samples with analyzed selected points

Table 3, EDS microanalysis data from the selected points 1-5 of the limonite ore I shown in Figure 6a (in wt. %). Analysis of the whole studied area is also presented.

Element	1	2	3	4	5	Whole area
0	44.67	32.03	50.10	49.05	46.11	38.05
Si	0.72	0.47	0.97	0.73	0.49	0.79
Al	n.d.	n.d.	n.d.	1.96	0.72	0.44
Fe	54.05	66.71	47.61	43.58	40.37	56.69
K	n.d.	n.d.	n.d.	n.d.	0.47	n.d.
Mn	0.55	0.79	1.33	4.68	11.83	4.03

n.d. - not detected

The most of the selected points of limonite ore I (spectrums 1-3, Table 3) contain Fe as the most dominant element with minor amounts of Mn and Si. This is in good agreement with XRPD results and corresponds to goethite as the main phase. Some of the selected points (spectrums 4 and 5) contain less Fe and different amounts of other elements (Mn, Al, Si and K) and could be assigned as mixture of goethite and other phases, such as Mn-minerals or impurities, and minor Al-silicates and quartz.

Table 4, EDS microanalysis data from the selected points 1 - 4 of the quartz I shown in Figure 6b (in wt. %). Analysis of the whole studied area is also presented.

Element	1	2	3	4	Whole area
0	52.46	53.94	60.23	49.07	59.27
Si	42.72	28.19	20.74	33.18	27.02
Al	3.64	12.73	13.89	11.73	8.52
Fe	n.d.	1.25	1.31	0.70	1.70
Na	0.43	n.d.	n.d.	0.41	n.d.
K	0.74	2.93	2.87	4.53	2.44
Mg	n.d.	0.70	0.96	0.38	0.59
Ti	n.d.	0.26	n.d.	n.d.	0.47

n.d. - not detected

X-ray microanalysis (EDS) data of the selected point 1 (Table 4) confirms that the quartz phase is dominant over small amounts of Al-silicates. Other selected points (2-4) are also identified as quartz, but with something higher amounts of Al-silicates. Elemental composition indicates that the Al-silicates are of clays and/or micas and/or feldspars composition.

Table 5, EDS microanalysis data from the selected points 1-6 of the clay I shown in Figure 6c (in wt. %). Analysis of the whole studied area is also presented.

Element	1	2	3	4	5	6	Whole area
0	48.06	56.08	55.90	45.58	57.28	49.71	55.22
Si	23.67	39.64	17.51	25.75	40.51	22.44	22.59
A1	16.61	1.82	13.86	18.70	0.63	18.20	10.94
Fe	1.71	2.28	10.07	2.13	1.07	1.55	6.89
Na	0.53	n.d.	n.d.	n.d.	n.d.	0.96	n.d.
K	8.08	0.18	1.41	7.35	n.d.	6.49	2.57
Mg	1.05	n.d.	n.d.	0.49	n.d.	n.d.	0.60
Ti	0.29	n.d.	0.57	n.d.	n.d.	0.22	0.65
Cu	n.d.	n.d.	0.68	n.d.	0.51	0.42	0.54

n.d. - not detected

The content of Si, Al and other elements, such as K, Na and Mg, indicate clays-micas intimately associated with quartz (spectrums 1, 3, 4 and 6, Table 5). Correlation of Si/Al in some selected points (spectrums 2 and 5) indicates to an almost pure quartz which dominates over clay-mica minerals. It should be mentioned that there is also increase of one Fe mineral at spectrum 3, and that there are significant Cu contents at spectrums 3, 5 and 6.

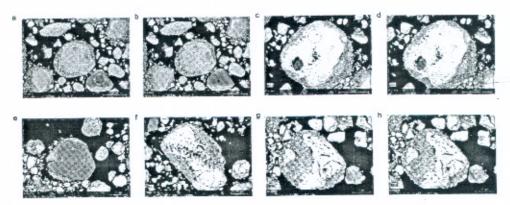


Figure 7, SEM images of the limonite ore II samples with analyzed a-h selected points.

Table 6, EDS microanalysis data from the selected points a-h of the limonite ore II shown in Figure 7 (in wt. %).

Element	a	b	С	d	e	f	g	h
0	42.47	32.69	30.93	49.82	42.33	29.37	28.27	30.87
Si	57.53	1.52	0.89	41.67	57.67	0.59	13.27	0.69
Al	n.d.	0.25	n.d.	n.d.	n.d.	n.d.	11.40	n.d.
Fe	n.d.	65.54	68.18	8.51	n.d.	70.12	44.06	68.44
K	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	3.00	n.d.

n.d. - not detected

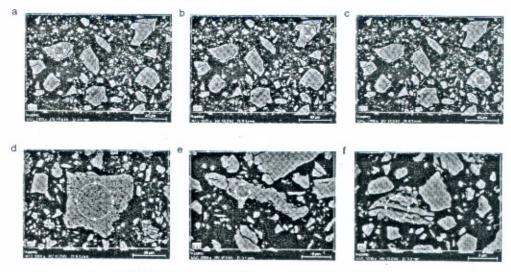
Same as in the limonite ore I, the most of the selected points of limonite ore II (spectrums b, c, f and h, Table 6) contain Fe as the most dominant element with minor amounts of Si and Al. This is in good agreement with XRPD results and corresponds to goethite as the main phase. Some of the selected points (spectrums a and e) are adequate to pure quartz. Spectrum d is adequate to the mixture of quartz and goethite, i.e. more precisely to the inclusion of quartz in goethite which is clear visible; whereas spectrum g is adequate to the mixture of goethite and Al-silicates.

Table 7, EDS microanalysis data from the selected points a-f of the quartz II shown in Figure 8 (in wt. %).

Element	a	ь	С	d	е —	f
0	43.15	43.09	41.90	41.62	44.22	42.62
Si	56.85	56.91	58.10	58.38	55.78_	28.76
Al	n.d.	n.d.	n.d.	n.d.	n.d.	20.56
K	n.d.	n.d.	n.d.	n.d.	n.d.	8.06

n.d. - not detected

Most of the selected points (spectrums a-e, Table 7) are adequate to the pure quartz. Only spectrum f most probably corresponds to some of the Al-silicates.



Figure~8, SEM~images~of~the~quartz~II~samples~with~analyzed~a-f~selected~points.

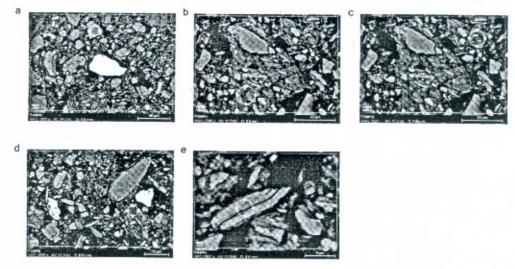


Figure 9, SEM images of the clay II samples with analyzed a-e selected points.

Table 8, EDS microanalysis data from the selected points a-e of the clay II shown in Figure 9 (in wt. %).

Element	a	b	С	d	e
0	50.29	45.41	41.61	41.49	42.55
Si	49.71	29.66	36.20	36.26	26.66
Al	n.d.	19.43	17.50	17.26	18.99
K	n.d.	5.50	4.69	5.00	n.d.
Mg	n.d.	n.d.	n.d.	n.d.	1.16

n.d. - not detected

The contents of Si, Al and other elements (spectrums b-e, Table 8) indicate to the clay-mica minerals. Only one of the selected points (spectrum a) indicates to the pure quartz.

SEM images and EDS chemistry of series I and II samples (Figure 6-9 and Tables 3-8) show heterogeneity of morphology, structural and mineral composition with the predominant oxide and/or hydroxide minerals consisting mainly of iron, silicon and aluminum, and confirming the XRPD and FTIR results. Namely, the "limonite ore" samples are composed of Fe minerals which dominate over quartz and Al-silicates, and with significant amount of Mn. The "quartz" samples are composed of major quartz which dominate over Al-silicates and Fe minerals. The "clay samples" are composed of major quartz and Al-silicates which dominate over minor contents of Fe minerals.

Such identified and characterized compositions of these natural raw materials are also in very good agreement and further explains the sludge composition generated during the processing of iron ore in Omarska mine.

This heterogeneity makes complex dispersion system, in which one of the basic parameters for the selective flocculation process is dispersive and flocculating behavior of the individual components of the system. One of the most important features is surface charge, which depends on the morphological, structural and chemical characteristics of minerals that form the main components of the system. Some earlier studies of these behavior of dispersion systems are mainly performed on mixtures of pure synthetic minerals [17-20]. Therefore, our future research will be aimed at defining the surface charge of natural minerals that have been identified here as the main components of the system and their behavior in the presence of different dispersants and flocculants.

CONCLUSION

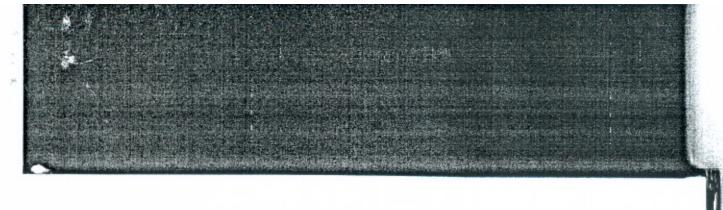
The primary natural raw material samples from Omarska mine, were studied and characterized by XRPD, FTIR, SEM and EDS analysis. According to the obtained results, it can be concluded that the "limonite ore" samples are composed of major goethite which dominate over hematite, and with minor contents of magnetite, quartz and clay minerals. The "quartz" samples are composed of major quartz which dominate over minor contents of clay minerals, feldspars and hematite. The "clay samples" are composed of major quartz and clay minerals which dominate over minor contents of feldspars, amphiboles, goethite and hematite. The

clay minerals were identified as mostly illite-sericite which prevails over kaolinite, and with chlorites which appears only sporadically.

Acknowledgment: The authors are grateful to Company ArcelorMittal in Prijedor for technical support and enabling the study visit Lj. Tankosić in the laboratory Global Research and Development, Mining and Mineral Processing, Maizières-lès-Metz, France for the purpose preparation of their PhD. The authors are also grateful to the SEM LAB of Faculty of Mining and Geology, University of Belgrade, for making SEM and EDS measurements.

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Београд, 12. јуни 2019.

Потврђујемо да је рад CHARACTERIZATION OF NATURAL RAW MATERIALS IN THE PROCESSING OF IRON ORE FROM OMARSKA MINE / КАРАКТЕРИЗАЦИЈА ПРИРОДНИХ МАТЕРИЈАЛА У ПРОЦЕСУ ПРИПРЕМЕ РУДЕ ГВОЖБА У РУДНИКУ ОМАРСКА, аутора Љ. Танкосић, П. Танчић, С. Средић, З. Недић и В. Малбашић, DOI:10.25075/SI.2017.31, саопштен на Мерђународном симпозијуму Рударство и геологија данас, Београд, 18 - 20. 09.2017, и после рецензије као научни рад публикован у Зборнику радова РУДАРСТВО И ГЕОЛОГИЈА ДАНАС, издавачи Рударски институт Београд, Балканска академија рударских наука и Академија инжењерских наука Србије, ИСБН 978-86-82673-13-2(РИ), 2017, стр. 316-329.

Потврда се издаје на захтев аутора због библиографске документованости.

Потпредседник Организационог комитета и уредник

Др Светлана Полавдер, научни сарадник