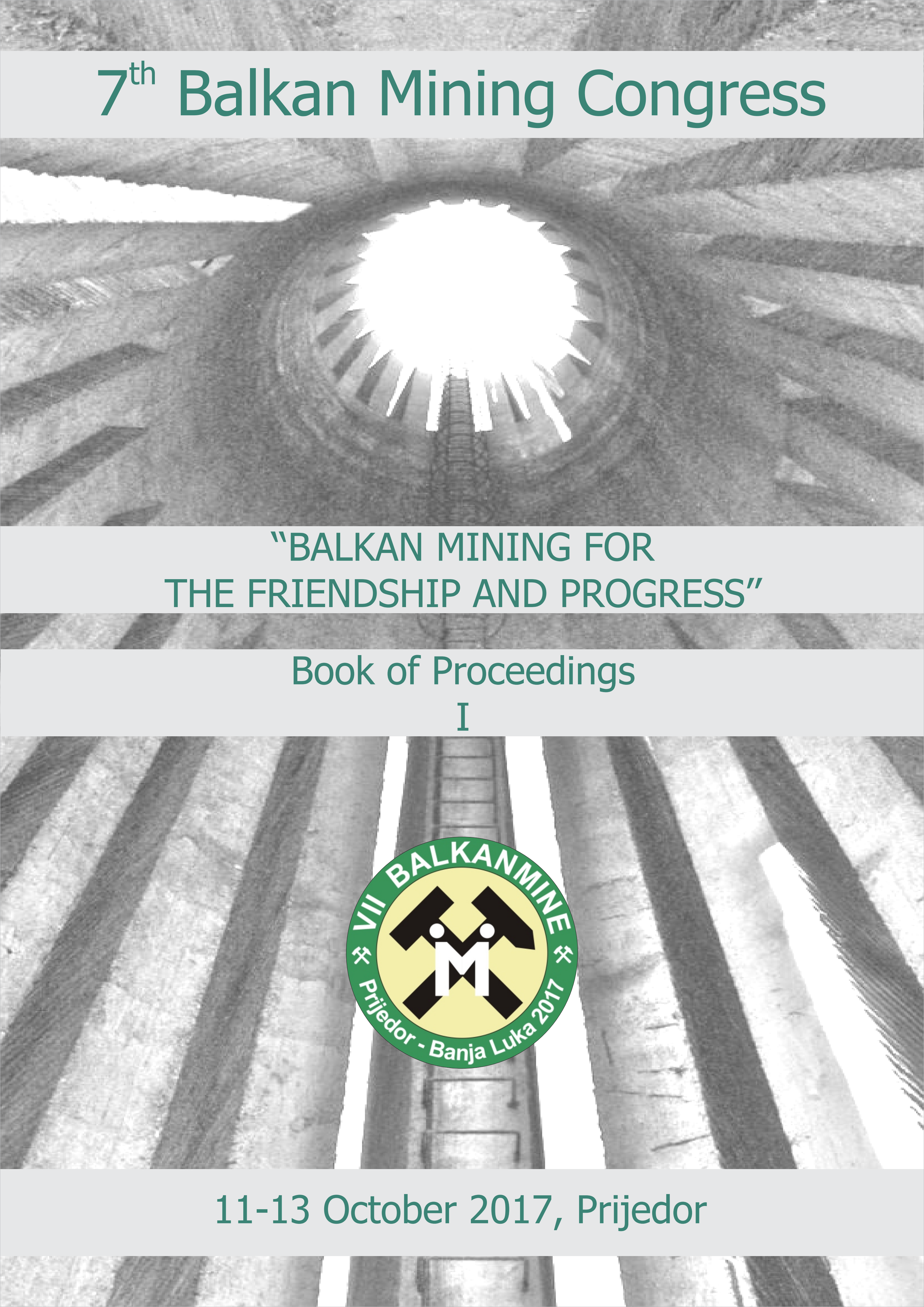

7th Balkan Mining Congress

"BALKAN MINING FOR THE FRIENDSHIP AND PROGRESS"

Book of Proceedings
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II Balkan Mining Congress

September 10-13, 2007.
Belgrade, Serbia
President of the Balkan Coordinating Committee:
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III Balkan Mining Congress

October 1-3, 2009.
Izmir, Turkey
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IV Balkan Mining Congress

October 18-20, 2011.
Ljubljana, Slovenia
President of the Balkan Coordinating Committee
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V Balkan Mining Congress

September 18-21, 2013.
Ohrid, Republic of Macedonia
President of the Balkan Coordinating Committee:
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VI Balkan Mining Congress

September 20-23, 2015.
Petrosani, Romania
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IN MEMORIAM

Assistant professor Tomo Benović was born on January 06, 1958 in Bogutovo Selo in Ugljevik. From 01.02.1982.Tomo Benović was employed in Rudnik i termoelektrana Ugljevik in the following works: trainee, shift manager, technical manager of coal production, assistant director (for mines and technical business), manager for mining and geological service, director of RiTE Ugljevik, coordinator for coordination with Regulatory Authorities, Team Leader of Project Implementation and realization of investments and projector for the mine. Tomo Benović was the first Mayor of Municipality Ugljevik and in the period 2000-2002 he had been a member of the National Assembly of Republic Srpska. Tomo Benović had been in the following scientific and professional organizations and associations: the Chairman of the Alliance of Engineers and Technician of Mining - Geological and Metallurgy Profession, the membership of the International Coordination Committee of the Balkan Mining Congress from Bosnia and Herzegovina in two mandates.

At the Senate of the University of Banja Luka session, held on August 25, 2016, Tomo Benović PhD in mining was elected as Assistant professor for scientific research - Surface exploitation of the mineral raw materials. Assistant professor Tomo Benović tragically died on 27 November 2016 in a traffic accident.

Preface

Dear Colleagues,

On behalf of the University of Banja Luka, the Faculty of Mining Prijedor and the International Coordination Committee of the 7th Balkan Congress, we welcome you as respected and dear guests of the University and Faculty, Prijedor, Republic Srpska and Bosnia and Herzegovina. The 7th Balkan Mining Congress has a motto **"Balkan mining for the friendship and progress"**, which speaks enough about the basic idea of organizing and holding this event. This Conress has been held biennial in the Balkan countries.

This international meeting is an opportunity for Congress participants - authors of works, sponsors, exhibitors, representatives of institutions and companies to meet each other, exchange experiences in solving problems and issues related to the development of mining, geology, and the work of companies. Every opportunity to hear something new, something that is applied in other countries and conditions is the chance to find a chance in this transition period which is difficult for the work and development of mining companies. The exploitation of mineral resources could be beneficial, for the producers themselves,and for local communities and countries where are these mines located.

In contemporary trends in the mining and geology development, there are dilemmas to reconcile certain, at first sight, completely opposed and incompatible activities: mining, environmental protection, optimal economic effects of mining activities for the concedents and concessionaires. The Balkan Mining Congress is a unique opportunity to talk about these issues, exchange experiences, find solutions and align certain models of more rational solutions.

Wishing to feel comfortable and pleasant in Prijedor, and after the end of the Congress, you go home happy and with the view that it was worth being here, I greet you in my personal name and the name of the University in Banja Luka-Mining Faculty Prijedor and others co-organizer of the Congress.

Prijedor, October 2017.

Assoc. prof. Vladimir Malbašić

Chairman of the Organizing Committee
of the 7th Balkanmine Congress

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PARTICLE SIZE DISTRIBUTION OF IRON ORE SLUDGE DETERMINED BY USING DIFFERENT METHODS AND IRON CONTENT BY SIZE CLASS

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ABSTRACT

This paper presents the results of the study of the particle size distribution of the limonite sludge that occurs as a hydro-cyclone overflow in the Omarska iron mine. The primary role of the precise characterization of grains of mineral raw material is in obtaining quantitative data about their particle size and analysis of the distribution of mineral grains by volume, which is a necessary step in controlling the processes of the iron ore beneficiation. Since the overflow of the hydro-cyclone size class is -25 μm, in this paper the following methods of sub-sieve analysis are used: sedimentation method-Beaker decantation; pipetting method by Andreasen-Borner; and Warman cyclosizer. Different methods were used in order to analyze the compliance of the results, as well as comparison of them, because expensive and rapid instrumental methods are often unavailable in practice. Also, the content of iron in the sludge varies, so the correlation of iron content with granulometric particle distribution was analyzed. These analyzes were carried out on two samples of sludge with different average iron content (29.43% and 41.19%). On both samples the iron content was determined per size class. The results showed a largest iron content in the smallest classes (-9+6 microns). These results indicate the necessity of applying some of the methods for selectively separating fine particles (such as selective flocculation, for example) in a further process of preparation.

Key words: particle size distribution, limonite sludge, Beaker, Andreasen-Borner, Warman

1. INTRODUCTION

Particle size is one of the most important factor in determining suitable technique in mineral processing. Many properties are dependent on particle size, such as: electrical properties (chargeability, surface conductivity, dielectric), dispersivity, specific surface area, etc., and they are decisive for the selection of an appropriate method of separating of useful minerals from gangue in stable dispersions, such as sludge [1-3]. Sludge, defined as –20 μm particles, are an unseparable part of finely ground minerals and may contain significant amounts of valuable minerals. Conventional techniques, such as: gravity concentration, magnetic separation and flotation, are generally ineffective in treating sludge. Thus, sludge are often discarded prior to concentration. Discarded sludge are not only an economical loss, but their disposal may be an environmental concern and costly to manage, as well. Many techniques have been investigated to treat sludge, but these systems are complex and each case is subject of special research [4-8].

In mineral processing plant of Omarska mine (Bosnia and Herzegovina), the iron ore from mineral deposit "Buvač" is prepared by the classification of the coarse classes and magnetic concentration of the classes (-0.500+0.025 mm). Class -0.025 mm represents tailings, but sometimes it contains up to 30% mass with high content of iron. Filter-press selectively treats the rich part of sludge and compensates for the mass loss at new magnetic separators. The structural-texture characteristics of limonite ore from the mine "Buvač" cause to obtain, already upon excavation, the major amount of smaller classes, with grit below 25 μm. Every further procedure enlarges the part of fine classes from which the concentrate of sufficient quality can't be extracted in the existing plant for mineral processing in Omarska, and as such it represents tailings sludge. This sludge is not suitable for deposition because it represents stabile suspension and related to that, a problem for the environment. On the other hand, formation of sludge, with approximately 50% Fe or over 75% limonite as waste, represents a major loss of limonite and decrease of economic effects. In this stable suspension the particles below 10 μm (ultrafine) remain dispersed for a long time. Because the movement of the smallest particles through fluid is slowing down, the traditional procedures of mineral processing of this material are not applicable [9]. Before choosing adequate methods for the treatment of this sludge, it is necessary to know the particle size distribution and iron content by size class.

2. MATERIALS AND METHODS

2.1. Materials

For this study, we used two samples of limonite sludge (Sludge I and Sludge II), which have different iron contents (29.43% and 41.19%) and densities (3.000 g/cm^3 and 3.526 g/cm^3), respectively. A samples of the sludge were taken as an overflow on hydro-cyclone plant for the preparation of iron ore in Omarska mine, Bosnia and Herzegovina. The natural pH of the samples ranged from 7 to 7.2. The mineral phases present in the sludge were investigated and characterized earlier [10]. According to the obtained results of XRD, IR and SEM-EDS analysis, both of sludge samples are composed of major goethite and quartz, less clay minerals, and minor magnetite and todorokite. Also, it was established that the phases composition of two analyzed sludge samples are almost the same, but that their content varies [10]. Therefore, the obtained densities in this paper are in excellent concordance with the obtained qualitative – semi-quantitative sludge mineral compositions, and especially to the ratio of goethite (and other minor Fe minerals) to quartz (and other silicate minerals, such as clay) [10]. It should be mentioned here that such sludge compositions are primarily derived by natural raw materials compositions, which were previously also precisely characterized [11].
As dispersant, we used sodium hexametaphosphate, $Na_6P_6O_{18}$, analytical grade, manufactured by Lach-Ner, s.r.o. (Czech Republic). The working solutions are made with distilled water.

2.2. Methods

For particle size analysis of fine particles, different methods were used, which can generally be divided into traditional sedimentation methods (based on Stokes' Law) and different instrumental methods. All of the methods have their advantages and/or disadvantages. In this study three common methods of sub-sieve analysis have been used: (1) Beaker decantation (sludge I and II); (2) Andreasen-Borner pipette method (sludge I and II); and (3) Warman cyclosizer-elutriation method (only sludge II). A detailed description of the above methods is given in the cited literature [1, 12-14]. Here, we present briefly description of each of them.

2.2.1. Beaker decantation

One of the most commonly used procedures for determining the particle size distribution of the raw material is certainly "Beaker decantation" proposed by E.J. Robert in 1953. [12]. This is a tehnique used to separate mineral sample in two or more size fraction according to the differences in setlling velocities of the particles. It is tehnique which can be used to accurately split a sample at a pre-determined cut size, but has some disadvantages because it is a time consuming method, and especially when spliting including a fine sizes. Also, a dilute solids content is required to stop natural coagulation from occuring.

For the procedure of examination using Beaker, we used 100 g limonite sludge, dried on 105 ^{o}C, two cups of 2 liters and six cups of 1 liter volume. For dispersant, we used 0,002 M sodium hexametaphosphate. The pulp was gently stirred to disperse the particles through the whole volume and then was allowed to stand for the calculated time. The water above the end of the tube was syphoned off, dried and weighed.

Velocity of the particles is calculated according to the form:

$$v_0 = 545(\rho_m - \rho_t) \cdot d^2 \left[mm/s \right] \quad (1)$$

Thime of settling:

$$t_1 = \frac{H_1}{v_0 \times 1{,}05} \left[s \right] \quad (2)$$

2.2.2. Andreasen-Borner method

Determining the granulometric distribution of materials by the Andreasen-Borner method belongs to a group of differential methods. This method is based on the same principle as the sedimentation scale, but here the deposition rate is not automatically registered and continuous. In the pipette method, concentration changes occurring within a settling suspension are followed by drawing off definite volumes, at predetermined times and known depths, by means of a pipette. Although, theoretically, errors can be reduced by the use of more complicated construction and operation, it is highly debatable as to whether this is worthwhile for routine analyses, since conventional apparatus is reproducible to ±2% if operated with care. This technique is a standard procedure since both the Stokes diameter and the mass undersize are determined from first principles. The method is versatile, since it can handle any powder that can be dispersed in a liquid, and the apparatus is inexpensive. The analysis is, however, time consuming and intensive.

For the procedure of examination using Andreasen method, we used Andreasen pipette, which represents a cylindrical vessel of about 500 ml volume and 2.5 g limonite sludge, dried on 105 ^{o}C. For dispersant, we used 0,002 M sodium hexametaphosphate. The suspension was alloved to settle and at given intervals of time, samples are withdrawn by applying suction to the top of the 10 ml reservoir. The samples ware then dried and weighed, and the weights compared with the weight of material in the same volume of the original suspension. Times of settling were calculated using equation *(3)* and shown in Table 1:

$$t = \frac{18 \cdot h \cdot H}{\left(r_m - r_f\right) \cdot 981 \cdot d^2} [s] \tag{3}$$

Table 1: Pipette time for two samples of limonite sludge

Particle size, µm	Pipette time (sludge I)			Pipette time (sludge II)		
	h	m	s	h	m	s
"0" probe						
25		5´	9´´		3´	50´´
18		9´	42´´		7´	19´´
13		18´	11´´		13´	38´´
9		37´	11´´		28´	00´´
6	1	22´	58´´	1	2´	37´´

2.2.3. Warman cyclosizer – elutriation method

One of the widely used methods of elutriation, determination of the granulometric composition of mineral resources, in the laboratories for the preparation of mineral raw materials is in Warman cyclosizer (Finch and Leroux,1982.) [1]. This method of determining the particle size of small diameter is most commonly used for routine testing and control in industrial processes, for particle size ranges of 8-50 micrometers and a density of about 2.7 g/m^3, or 4 micrometer classes for material with higher density.

Particle size distribution of sludge II using Warman cyclosizer were performed in the laboratory Global Research and Development, Mining and Mineral Processing, Maizières-lès-Metz, France. Data were obtained on CYCLOSIZER M_{16} – MARC TECHNOLOGIES, 230 V, 50 Hz under following work conditions: pressure 270 kPa, temperature of water 18.5 ^{o}C, flow rate of water 120 l/min, time for test 15 min, particle size of sample -25 µm, specific density of sample 3.526 g/cm^3.

2.2.4. Chemical analyses

Chemical analyses were performed according to BAS ISO 2597-1:2012 standard for determination of iron in iron ore.

2.2.5. Density

Density of sludge samples is determined by the method using a pycnometer. Samples were dried at 105 °C to constant weight prior to determination, and distilled water was used at room temperature at 20 °C.

3. RESULTS

3.1. Results of the particle size analyses using Beaker decantation method

Results of the particle size analyses using Beaker decantation with iron content by size class are shown in Table 2 and Figure1 (Sludge I) and Table 3 and Figure 2 (Sludge II).

Table 2. Particle size distribution of Sludge I

Actual Fraction (µm)	Weight (g)	% Weight	% OS Cum.	% US Cum.	Fe mass%
+25	10.42	10.42	10.42	100	29.05
-25+18	6.77	6.77	17.19	89.58	27.63
-18+13	8.96	8.96	26.15	82.81	27.34
-13+9	12.60	12.60	38.75	73.85	28.71
-9+6	11.88	11.88	50.63	61.25	30.23
-6+0	49.37	49.37	100	49.37	28.73
TOTAL	100	100		0.00	

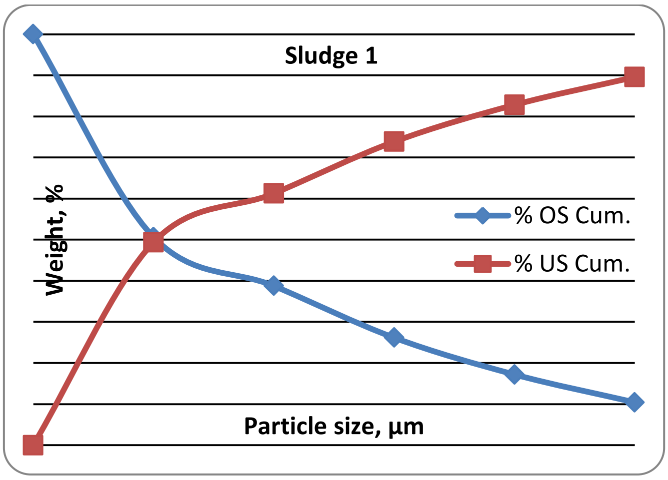

Figure 1. Particle size distribution of Sludge I

The most part of the mass is in fraction -6+0 µm (49.37%, Table 2) which coincides with previous research [15]. This chemical analysis show slight % increase of iron (Fe) in fraction -9+6 µm (30.23%) compared to the initial sample Sludge I (29.43%).

Table 3. Particle size distribution of Sludge II

Actual Fraction (μm)	Weight (g)	% Weight	% OS Cum.	% US Cum.	Fe mass%
+25	13.83	13.83	13.83	100	44.53
-25+18	7.58	7.58	21.41	86.17	38.78
-18+13	7.93	7.93	29.34	78.59	40.09
-13+9	8.29	8.29	37.63	70.66	41.49
-9+6	9.11	9.11	46.74	62.37	43.27
-6+0	53.26	53.26	100	53.26	37.38
TOTAL	100	100		0.00	

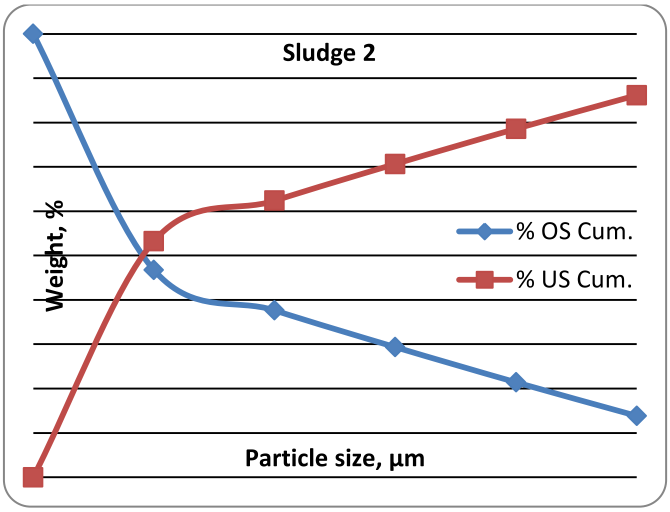

Figure 2. Particle size distribution of sludge II

As in the previous experiment, the most part of the mass is in fraction -6+0 μm (53.26%, Table 3) which coincides with previous research [15]. Chemical analysis show significantly % increase of iron (Fe) in fractions +25 μm (44.53%) and -9+6 μm (43.27%) compared to the initial sample Sludge II (41.19%).

3.2. Results of the particle size analyses using Andreasen-Borner method

Results of the particle size analyses using Andreasen-Borner method with iron content in the fraction -6+0 μm, are shown in Table 4 (Sludge I) and Table 5 (Sludge II). It was not possible to make chemical analysis in all particle size class (except in the fraction -6+0 μm), because of the low mass in all of the other fractions using for testing by Andreasen-Borner method.

Table 4. Particle size distribution of Sludge I

Actual fraction (µm)	Weight of material, g		Weight of material, %		Fe mass%
	sum	dif	W,%	Σ W,%	
"0" probe	0.0917		3.70	3.70	
+25	0.0836	0.0081	3.30	7.00	
-25+18	0.0836	0.00	3.30	10.30	
-18+13	0.0724	0.0112	2.90	13.20	
-13+9	0.0687	0.0037	2.75	15.95	
-9+6	0.0687	0	2.76	18.71	
-6+0	Difference to 100%		81.29	100.00	18.80

Table 4. shows that the most part of the mass is in fraction -6+0 µm (81.29%) which is a significant mass fraction in the sample compared with other fractions. This chemical analysis show significantly % decrease of iron (Fe) in fraction -6+0 µm (18.80%) compared to the initial sample (29.43%).

Table 5. Particle size distribution of Sludge II

Actual fraction (µm)	Weight of material, g		Weight of material, %		Fe mass%
	sum	dif	W,%	Σ W,%	
"0" probe	0.0850		4.11	4.11	
+25	0.0790	0.0060	3.82	7.93	
-25+18	0.0785	0.0005	3.80	11.73	
-18+13	0.0681	0.0104	3.30	15.03	
-13+9	0.0652	0.0029	3.15	18.18	
-9+6	0.0576	0.0076	2.79	20.97	
-6+0	Difference to 100%		79.03	100.00	28.94

Table 5. shows that the most part of the mass is in fraction -6+0 µm (79.03%) which is a significant mass fraction in the sample compared with other fractions. This chemical analysis show significantly decrease % of iron (Fe) in fraction -6+0 µm (28.94%) compared to the initial sample (41.19%).

3.3 Warman cyclosizer

The particle size analyses were performed only on sludge II sample. Results of the particle size analyses using this method with iron content by size class are shown in Table 6 and Figure 3. It was listed original data of diameter range and actual fraction.

Table 6. Particle size distribution of Sludge II

Cyclone No	Diameter range (µm)	Actual Fraction (µm)	Weight (g)	% Weight	% OS Cum.	% US Cum.	Fe mass%
UF 1	44	25	4	8	8	92	43.43
UF 2	33	18.75	1.3	2.6	10.6	89.4	37.94
UF 3	23	14.06	3.1	6.2	16.8	83.2	37.76
UF 4	15	10.54	3	6	22.8	77.2	38.52
UF 5	11	7.91	4	8	30.8	69.2	39.77
OF		5.93	34.6	69.2	100	0	40.92
		TOTAL	50	100			

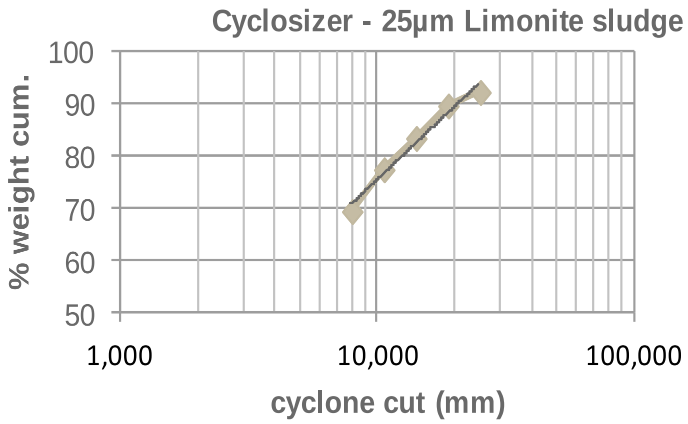

Figure 3. Particle size distribution of sludge II

Results of particle size distribution on Warman cyclosizers also show that the largest proportion of the mass (69.2) is in the class -5.93+0 µm. This chemical analysis show % decrease of iron (Fe) in fraction -5.93+0 µm (40.92%) and % increase of iron (Fe) in fraction +25 µm (43.43%) compared to the initial sample (41.19%).

4. DISCUSSION

As mentioned above, for selecting the most suitable method for mineral processing, one of the most important things is to determine the particle size distribution. The properties that depend on the particle size distribution, such as electrical, reactive and transport properties of particles, are the most important features especially when it comes to fine particles. Also, it is necessary to know the content of useful substances, in this case iron content by size class. Due that the iron content of the overflow in the hydro-cyclone varies, we analyzed the two samples with different initial iron content. Particle size distribution and iron content on sample II were analyzed on a cyclosizer and the first task was to compare these results with the results obtained by some of the other common methods. For this purpose, both samples were analyzed using Beaker decantation and Andreasen-Borner pipette methods.

4.1. Comparison of the used methods

Figure 4 represent comparison of the results of determination of particle size distribution using the three methods (in the brackets are given the original data for particle size limits on the cyclosizer).

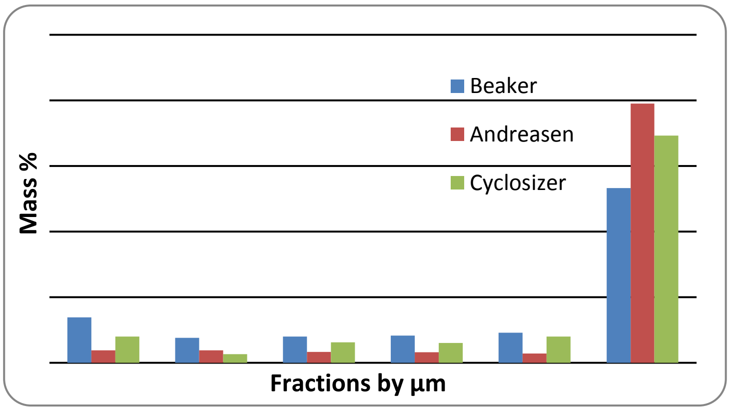

Figure 4: The comparison of content of individual fractions in mass% determined by different methods

The differences between the individual methods were compared using the two-method conversion factor (FC); FC = Method A/Method B. In Table 7 are given calculated values of FC and D (differences in relation to FC = 1 as absolute value), and the sum of D as an indicator of deviation of the results obtained by different methods. Tags B, A and C relate to the methods (B-Beaker decantation, A-Andreasen pipette and C-Warman cyclosizer).

Table 7. Differences between methods by fractions

Particle size (µm)	Compared methods	FC	D	ΣD
+25	B:A	3.6	2.6	4.4
	B:C	1.7	0.7	
	C:A	2.1	1.1	
-25+18(18.8)	B:A	2.0	1.0	3.2
	B:C	2.9	1.9	
	C:A	0.7	0.3	
-18+13(14.1)	B:A	2.4	1.4	3.0
	B:C	2.4	1.4	
	C:A	0.8	0.2	
-13+9(10.5)	B:A	2.6	1.6	2.9
	B:C	1.4	0.4	
	C:A	1.9	0.9	
-9+6(7.9)	B:A	3.3	2.3	4.3
	B:C	1.1	0.1	
	C:A	2.9	1.9	
-6(5.9)+0	B:A	0.7	0.3	0.6
	B:C	0.8	0.2	
	C:A	0.9	0.1	

All three methods have shown the largest mass of tested samples contained in the finest fraction (Figure 4) and the slightest deviation of the result of the methods used to each other just for the finest fraction (Table 7). This indicates that in our case we can apply all three methods. However, in further analysis we rejected the Andreasen pipett method because of the impossibility of determining the content of iron by particle size classes for the above-mentioned reasons.

4.2. Determination of iron content by the class size

In Table 8 are given iron contents by the class size for both of samples. There are some deviations in the iron content by the class size using the different methods for particle size distribution in the case of sludge II (Warman cyclosizer and Beaker). In some classes, the difference is significant, as we can see from Table 8. Compared to the initial content of iron, better matching of average is in the case of the Beaker method for sludge II.

Table 8. Iron content by the class size

Actual fraction (μm)	Sludge II			Sludge I
	%Fe (C)	%Fe (B)	Deviations between C and B (%)	%Fe (B)
Initial sample	41.19			29.43
+25	43.43	44.53	2.47	29.05
-25+18(18.8)	37.94	38.78	2.16	27.63
-18+13(14.1)	37.76	40.09	5.81	27.34
-13+9(10.5)	38.52	41.49	7.15	28.71
-9+6(7.9)	39.77	43.27	8.08	30.23
-6(5.9)+0	40.92	37.38	7.52	28.73
Average	39.72	40.92		28.62

4.3. Comparison of two samples of the sludge iron content

Finally, the results of two samples of sludge with different initial iron content can be discussed. The particle size distribution was determined by the Beaker decantation method for samples with different initial iron content. On both samples the iron content was determined per size class. Calculated average iron contents give a satisfactory agreement in comparison with the initial content. Due to the transparency, the results for two samples are summarized in Table 9.

Table 9. Particle size distribution and iron content by size class for two investigated samples

	Sludge I		Sludge II	
Actual Fraction (μm)	mass %	Fe mass%	mass %	Fe mass%
Initial	100	29.43	100	41.19
+25	10.42	29.05	13.83	44.53
-25+18	6.77	27.63	7.58	38.78
-18+13	8.96	27.34	7.93	40.09
-13+9	12.60	28.71	8.29	41.49
-9+6	11.88	30.23	9.11	43.27
-6+0	49.37	28.73	53.26	37.38
Average	-	40.92	-	28.62

Although in some classes a slightly higher iron content is observed compared to the initial, these differences are not sufficient reason for further research to be done on separate classes. A large mass percentage of the finest class in both samples also supports this. Bearing in mind that the separation of such fine classes is very difficult and complicated, this procedure is justified only in the case of large differences in the content of iron. Particularly interesting is that there is no big difference in the classes of the particle size distribution of the two samples although the mineral phases are present in various contents. This indicates that all mineral phases, both useful and useless, are present in the sludge as very fine particles which makes this system very complex for further separation process. Therefore, our future research will be aimed at defining the surface charge of natural minerals that have been already identified [10,11] as the main components of the system and their behavior in the presence of different dispersants and flocculants.

5. CONCLUSION

The particle size distribution and iron content by size classes of the limonite sludge that occurs as a hydro-cyclone overflow in the Omarska iron mine were studied on two samples with different initial iron content. According to the obtained results, it can be concluded that the both samples are composed mainly of the most finest particles (below 6 microns). The content of iron by particle size classes mostly corresponds to the content of iron in the initial samples, but there is no significant difference in iron content by classes in any sample. The results showed a large iron content only in the finest classes. The rather uniform distribution of iron by class and a large percentage of the smallest particles confirm that all mineral phases in the sludge are present as fine particles regardless of the initial sample. There are not sufficient reasons for further research to be done on separate classes and it will be done on bulk samples. Future research will be focused on the study of surface properties (primarily electrical properties), as for sludge, as well as for the individual phases of which are part of the sludge.

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Subject: Confirmation letter

TO WHOM IT MAY CONCERN

We hereby confirm that Review paper entitled "PARTICLE SIZE DISTRIBUTION OF IRON ORE SLUDGE DETERMINED BY USING DIFFERENT METHODS AND IRON CONTENT BY SIZE CLASS", with authors: Tankosić, Lj., Tančić, P., Sredić, S., Nedić, Z., Torbica, D.; has been published (after positive reviews) in the Congress Book of Proceedings I, ISSN: 2566-3313, Year 7, No 7 (2017), DOI: 10.7251/BMC 170701129T (available at: http:/balkanmine2017.com/) of the Balkan Mining Congress "BALKANMINE 2017".

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