

Limestone of „Međeđa“ - Nikšić as filler in various industries

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This paper presents the results of testing the possibility of using limestone from the „Međeđa“ - Nikšić deposit in different industries. During the examination, the coarse moisture, chemical composition, granulometric composition, bulk and particle density, degree of whiteness, oil and water absorption, granulometric composition of micronized samples and specific surface area were determined. All tests were performed on 8 samples taken from different parts of the deposit and the mean values of the obtained results are presented here. Based on the physico-chemical characteristics, it can be stated that this limestone has a high content of CaO and CaCO₃ of 99.01%, as well as a relatively low content of MgO of 0.32% and a low content of SiO₂ of 0.26% and alkali and R₂O₃, with also low content of heavy metals, which is very favorable for limestone. With these physico-chemical properties, this limestone can be used in the following industries: paints and varnishes, paper, rubber and PVC, foundry and foundry industry, sugar industry, metallurgical purposes, glass production, animal feed production and mineral fertilizer production. A serious defect of „Međeđa“ limestone is the low degree of whiteness, which does not exceed 89.6%, which prevents its use in the pharmaceutical and chemical industries, while the relatively high content of K₂O, Fe and Mn prevents its use in calcification of acid soils.

Key Words: limestone, filler, industrial use, standards, comminution and classification

1. INTRODUCTION

Sedimentary rock limestone is composed mostly of calcium carbonate, most often in the form of minerals calcite and aragonite, which represent its various crystalline forms [1, 2]. It can be formed as a biological sedimentary rock by the deposition of shells, coral, algae and other organic residues [3]. It can also be formed by chemical sedimentation processes, i.e. the deposition of calcium carbonate from lake or sea water [2, 4, 5].

Calcite is the most abundant mineral in the Earth's crust, in which it participates with about 4% by weight [6]. Calcite is the most abundant mineral in limestone and is therefore the subject of research in various scientific fields: mineralogy, chemistry, physics, materials science [2, 7-10]. Calcite crystallizes rhombohedrally, in a hexagonal-scalenohedral class and space group $R\bar{3}c$ [11-15].

The Republic of Montenegro has large reserves of limestone [16]. Limestone deposits are very widespread in both the coastal and southern part of the Republic, from the border with Albania to Hercegovina, while the occurrence of limestone in the northern part of the Republic is less.

Despite the fact that Montenegro has huge reserves of limestone that are tied to a large part of the territory of the Republic, they are still relatively little used.

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Limestone is mainly used in construction in Montenegro as a technical-construction and to some extent as an architectural-construction stone [17]. Given the growing use of limestone in the world, in various industries, both micronized and coarse material, the competent Ministries of Montenegro have begun to consider this issue [18, 19].

The quality of limestone as a filler for various branches of industry is determined by a number of parameters: physical (whiteness, granular composition, oil and water absorption ...) and chemical content of individual elements or components. The values of physical parameters and the content of the mentioned components or elements are strictly defined by the standards that apply to individual industries [18-25]. First of all, regarding the chemical composition of limestone, the most important is the content of the main component in limestone - CaO and CaCO₃ content, as well as the content of accompanying and harmful components such as: MgO, CO₂, SiO₂, K₂O, total S, Al₂O₃ and Fe₂O₃, LOI, as well as the content of heavy metals above all Pb, Zn, Cr, Mn, Ni, Cd, Fe, Cu, As, Ba [25].

2. EKSPERIMENTAL

Limestone deposit - Medjedje is located in the area of the eponymous village Međeđe, near Budos (Nikšić municipality). Limestone from the „Međeđe“ deposit represents the uplift and subsoil of white bauxite that occur in this area. Reserves of limestone as a technical - building stone are estimated at the level of 15.000.000 m³, or about 40.000.000 tons [20].

From the deposit „Međeđe“, 8 samples of limestone were taken, on which physical-chemical tests were performed, which give insight into the quality of this raw material as well as the most rational possibilities of its commercial application. The mentioned 8 samples of limestone from the deposit „Međeđe“ were physically and chemically analyzed and based on the obtained results, the possibility of valorization of limestone from this deposit was assessed in accordance with standards (SRPS) in various industries [19-26].

Figure 1 shows a map of geological exploration works. Sample M-1/18 is the limestone taken from the upper layers of the white bauxite core exploitation well, sample M-2/18 was taken from the bottom layers of the white bauxite core exploitation well, samples marked M-3/18 and M-4 / 18 were also taken from the core of exploration wells, while samples marked M-5/18, M-6/18, M-7/18 and M-8/18 were taken from excavations of fresh, undecomposed and undegraded material.

Determination of the physical properties of these eight limestone samples was performed in the Labora-

ratories ITNMS-Belgrade. Samples for chemical analysis were sent by associates of Geozavod - Podgorica. The chemical analysis was performed at the Veritas Laboratory in Vancouver, Canada.

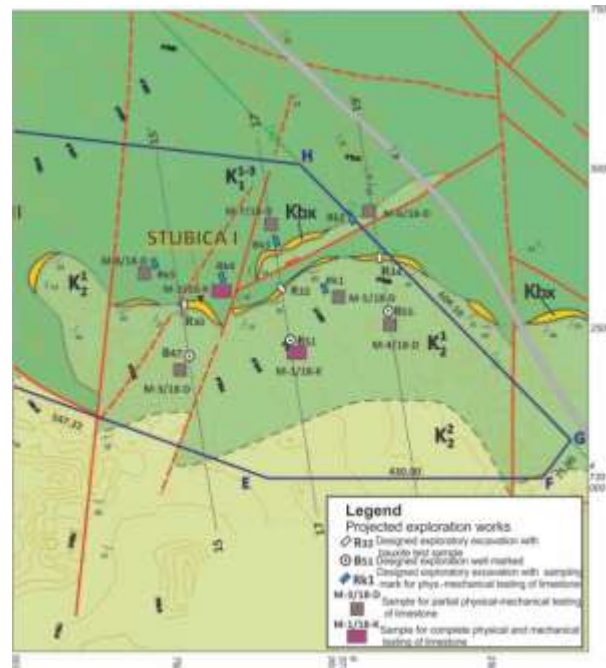


Figure 1 - Map of exploration geological works [20]

Sample preparation and sampling for laboratory processing

All eight samples were unpacked, then the primary sample was taken from each to determine the coarse moisture, the rest of each sample was homogenized and shortened by the quartering method and one half was sampled and the other half was saved as a reserve.

Determination of coarse moisture

All samples were dried at room temperature for a period of 24 h, and no coarse moisture was measured in any of them, i.e., all limestone samples delivered to the Institute were dry.

3. RESULTS

Particle size distribution

From the eight primary limestone samples, a composite sample with equal mass fractions was made to determine the granulometric composition. The granulometric composition of the limestone sample was determined by sieving on a Tyler series of sieves [27].

The screen oversize together with the undersize of the last sieve were measured, the data were sorted and shown in Table 1. Based on the data from the table, a diagram of the granulometric composition shown in Figure 2 was drawn. Upon the results upper limit size is 9.06 mm while the mean grain diameter is 4.96 mm

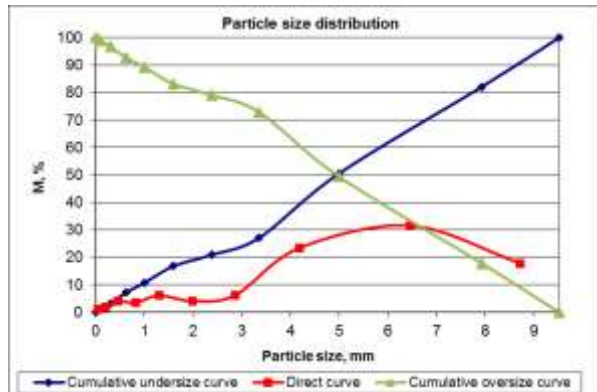


Figure 2 - Particle size distribution: $d_{95} = 9.06$ mm, $d_{50} = 4.96$ mm

Preparation of samples for technological testing

To examine the possibility of using limestone as a filler in various industries, all samples were micro-nized and then sent for the analyzes in order to deter-

mine: chemical composition, bulk density, white degree (whiteness), oil and water absorption, specific volume mass, granulometric composition and specific surface.

Determination of chemical composition

Chemical composition was determined by Mineral spot analysis using Laser Ablation ICP-MS. Based on the chemical analysis, it is noticed that the content of elementary components in these 8 samples of limestone varies and the obtained values deviate from each other to a noticeable extent.

For this reason, the average value of the content of these eight chemicals analyzes was taken to assess the possibility of using limestone from this deposit as a filler, as well as to design a technological scheme of processing that would obtain the mentioned products for different industries. The obtained values as well as the mean values are given in Table 2.

Table 1. Chemical composition of limestone samples from the "Medjedje" deposit (1-8)

Comp.	SAMPLE MARK								
	M-1/18	M-2/18	M-3/18	M-4/18	M-5/18	M-6/18	M-7/18	M-8/18	Mean value
	%	%	%	%	%	%	%	%	%
CaO	54.90%	55.80%	55.10	55.00	55.40	55.90	55.90	55.80	55.475
CaCO ₃	97.99	99.59	98.34	98.16	98.88	99.77	99.77	99.59	99.01
CO ₂	43.21	43.47	43.15	43.09	43.17	43.10	43.12	43.26	43.20
MgO	0.43	0.18	0.40	0.40	0.31	0.11	0.14	0.20	0.32
Fe ₂ O ₃	0.09	0.11	0.07	0.06	0.12	0.07	0.04	0.09	0.08
Al ₂ O ₃	0.16	<0.01	0.14	0.11	0.12	0.09	0.02	<0.01	0.08
SiO ₂	0.48	0.07	0.46	0.45	0.26	0.22	0.09	0.09	0.26
K ₂ O	0.08	0.03	0.08	0.07	0.05	0.04	0.03	0.03	0.051
Na ₂ O	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
TiO ₂	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
P ₂ O ₅	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
R ₂ O ₃	0.25	0.12	0.21	0.17	0.24	0.16	0.06	0.09	0.162
LOI	43.20	43.49	43.14	43.08	43.16	43.08	43.10	43.27	43.19
MnO	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
S	0.06	0.02	0.06	0.06	0.03	<0.02	<0.02	0.02	0.035
Cr ₂ O ₃	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cu, ppm	1.1	0.8	0.7	1.2	0.8	1.5	3.6	1.0	1.34
Ni, ppm	1.5	6.7	2.2	1.8	0.9	0.9	1.0	3.8	2.35
Co, ppm	<0.2	1	<0.2	<0.2	0.2	0.2	<0.2	0.7	0.34
Mo, ppm	2.3	9.7	1.3	0.9	1.3	1.6	1.4	7.1	3.2
Sb, ppm	0.1	0.6	0.1	<0.1	<0.1	0.1	0.2	0.4	0.21
Pb, ppm	0.5	0.3	0.4	0.6	0.4	0.7	1.7	0.6	0.65
Zn, ppm	3	2	3	3	4	2	2	3	2.75
Cd, ppm	0.1	<0.1	<0.1	0.1	<0.1	0.1	<0.1	0.1	0.08
Ba, ppm	4	<1	4	3	4	2	2	2	2.7
As, ppm	1.2	4.5	0.9	0.6	0.7	1.8	1.1	3.8	1.82
Hg, ppm	<0.01	<0.01	0.01	0.01	<0.01	<0.01	0.01	<0.01	0.002

*Physico-chemical characteristics***Table 2.** Physico-chemical characteristics of the limestone Medjedja

Sample mark	Bulk density, kg/m ³	Whiteness according to MgO – 99.0 %	Oil absorption, %	Water absorption, %	Particle density, kg/m ³	Specific surface, m ² /g
M-1/18	1.1322	84.3	11.15	15.75	2.732	2.332
M-2/18	1.0650	83.3	12.80	20.45	2.716	2.423
M-3/18	1.0985	87.0	12.10	17.45	2.684	2.622
M-4/18	1.1465	85.4	11.70	15.00	2.701	2.154
M-5/18	1.1720	85.6	11.70	15.50	2.682	1.945
M-6/18	1.0615	89.1	13.00	16.55	2.709	2.044
M-7/18	1.1540	89.6	13.40	17.15	2.684	1.794
M-8/18	1.1370	86.7	12.25	15.45	2.672	1.988

Determination of bulk density of micronized limestone samples

Bulk density was determined on micronized limestone samples. Determination of bulk density was performed with three different measurements on each sample. The values shown in Table 2 represent the mean values of the measurements.

Determining the whiteness

Whiteness was determined using Leukometer Carl Zeiss Jena using documented method DM -10/46. Due to its accuracy, the whiteness was determined so that three measurements were performed for each sample.

The test results are presented in Table 2 and represent the mean value of the three measurements performed. The whiteness is determined according to the standard for MgO whose degree of whiteness is 99.0 %.

Determination of oil and water absorption

Adsorption of oil and water was determined using documented method DM-10/70 and DM-10/71, respectively. The results of determining the absorption of oil and water were determined for each sample of limestone with three measurements and the presented results represent the mean value of these measurements. The obtained values of oil and water absorption for all limestone samples are shown in Table 2.

Determination of particle density

Specific weight was determined using standard method SRPS EN 1936:2009. All specific weights determined on three samples and the data presented in the report is in fact the mean value.

The values of the specific bulk density of micronized limestone samples are shown in Table 2.

Determination of specific surface area of limestone samples

The specific surface area of limestone samples according to bulk density was determined on a device for determining the granulometric composition of Sympatec Helos BR (H1506) Sucell 2; HELOS. When determining the granulometric composition, this device also determines the specific surface area in relation to the bulk density. By measuring the bulk density, it is possible to calculate the specific area per unit mass for each sample. The specific surface area of micronized limestone samples was calculated according to the formula:

$$Sm = \frac{S_v}{m_v}, \left[\frac{m^2}{g} \right].$$

The values of the specific surface area of micronized limestone samples are given in Table 2.

Determination of granulometric composition of micronized limestone samples

From the eight primary micronized limestone samples, a composite sample with equal mass fractions was made to determine the granulometric composition. Granulometric compositions of primary micronized limestone samples and intermediate micronized samples were determined on a Sympatec Helos BR (H1506) Sucell 2; HELOS, which works on the principle of laser diffraction (refraction) of laser beams on micronized particles of limestone samples.

Determination of granulometric composition was performed in the measuring range from 0.1 μm to 875 μm. The granulometric composition of the mean micronized limestone sample is given in the form of the diagram in Figure 4, and tabulated in Figure 5.

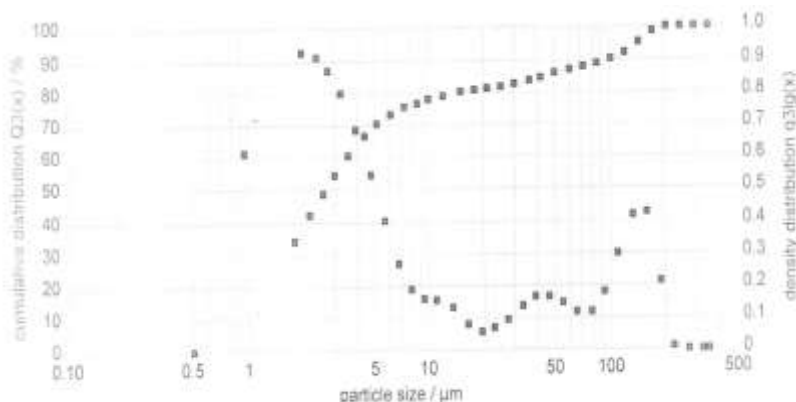


Figure 3 - Granulometric composition of micronized limestone sample

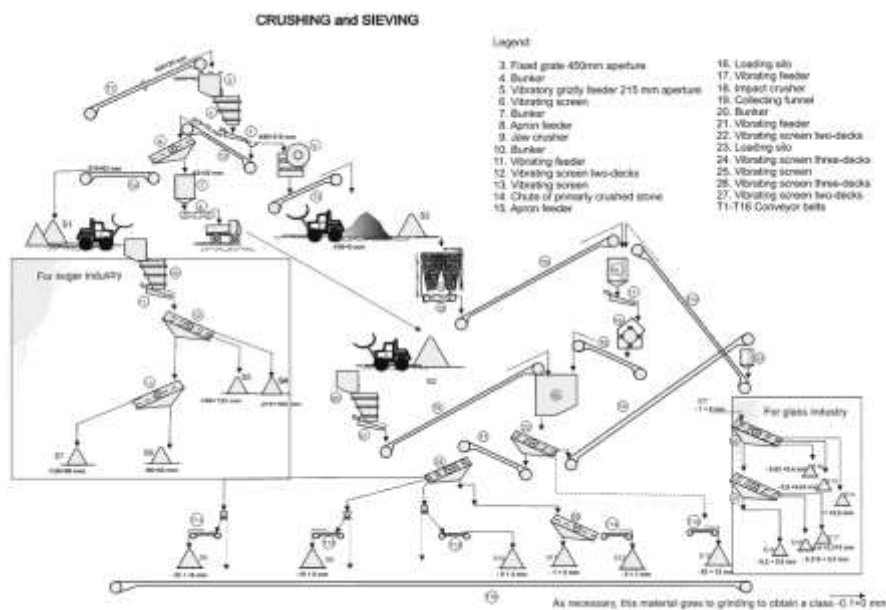


Figure 4 - Technological scheme of crushing and sieving of limestone of the deposit „Međeđa“ - Nikšić [19]

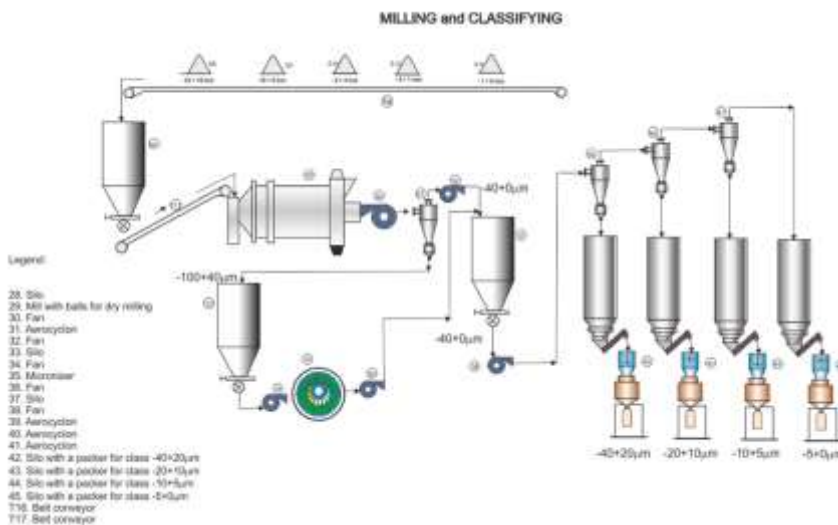


Figure 5 - Technological scheme of milling and classification of limestone of the deposit „Medjedje“ - Nikšić [19]

4. DISCUSSION

After considering the results of chemical analysis and the obtained chemical composition, it can be stated that Medjedja limestone has a high content of CaO or CaCO₃ (on average the content of CaCO₃ is 99.01 %), with a relatively low content of MgO-0.32 %, low content of SiO₂ -0.26 %, low content of alkali and R₂O₃, and also low content of heavy metals, which is very favorable for limestone. The only serious defect of Medjedja limestone is low whiteness, which does not exceed 89.6% in any of the eight samples. With this chemical composition and other important physico-chemical properties, Medjedja limestone can be used in the following branches of industry as a filler:

- In the paint and varnish industry where according to the standards (SRPS EN ISO 3262-5:2009) and market requirements it meets the highest A class quality
- Partly in the paper industry, where according to the standard (SRPS B.B6.033), its chemical composition could be classified in the highest quality group of products, however, despite the high content of CaCO₃ (over 99 %), low whiteness determines that it can be used as filler in lower quality A, B and C quality classes, while for the highest quality D quality class the whiteness of this limestone is not satisfactory.
- The required quality of calcium carbonate for use in the rubber and PVC industry as filler is also determined by the standard (SRPS B.B6.031). This limestone according to chemical composition of manganese (Mn) certainly meets B and D quality classes which demand that the content of Mn be up to 400 ppm. As for the requirements for better quality A and C class, it is envisaged, among other things, that the content of Mn be up to 50 ppm. According to a chemical analysis conducted by Bureau Veritas in Canada (given in Table 1), the Mn content was determined via MnO and it is in all eight samples less than 0.01 % MnO, i.e., less than 77.45 ppm Mn, so for these two classes it is necessary to perform a more precise chemical analysis and determine whether the Mn content is less than 50 ppm. As for the Fe content which is 800 ppm (chemical composition, Table 1), it determines that this limestone can be used as a filler for poorer B and D quality classes in the rubber and PVC industry, where it is according to the standard *JUS B. B6.031* allowed Fe content up to 1000 ppm, while for higher quality A and C quality classes in the same industries the allowed Fe content is max 300 ppm.
- The quality of limestone for the foundry and foundry industry is also defined by the standard

(SRPS B.B6.012). According to this standard, limestone Medjedja with all its parameters and chemical composition, corresponds to the I quality class of calcium carbonate for this purpose. In several samples, the S content was questioned, which is defined for the I quality class standard (SRPS B.B6.012) and should not exceed 0.03 %, while the average calculated value of S content for all eight limestone samples (Table 1) is at the level of 0.035 %.

- The required quality of calcium carbonate for use in the sugar industry is defined by the standard (SRPS B.B6.013). According to all parameters, „Medeđa“ limestone could belong to the I class of calcium carbonate quality for this purpose, but its slightly lower CO₂ content (43.20 %, Table 1) determines that it belongs to the II quality class for this industry, where it is planned that min. the CO₂ content will be 43.34 %.
- The required quality of calcium carbonate for use in metallurgical purposes is defined by the standard (SRPS B.B6.011). „Medeđa“ limestone according to the content of all important components for this purpose (CaO, MgO, SiO₂, R₂O₃ and P) belongs to the I quality class, while the content of S is similar to the one commented on the required quality of calcium carbonate for the foundry industry. Namely, the S content for metallurgy for all quality classes (I, II and III) is strictly defined by the standard (SRPS B.B6.012) and should not exceed 0.03 % for I and II class, while for Class III, max. the allowed S content is 0.4 %, and the average calculated value of the S content for all eight limestone samples (Table 1) is at the level of 0.035 %.
- The required quality of calcium carbonate for glass production is defined by the standard (SRPS B.B6.020). „Medeđa“ limestone according to the content of all essential components for this purpose (CaO, MgO, SiO₂, Al₂O₃) could belong to the I quality class, however, slightly increased Fe₂O₃ content of 0.08 % (Table 1), classifies this raw material for these purposes in III quality class.
- The required quality of calcium carbonate, which is prescribed by the manufacturer (Azotara Pančevo) for use in the production of mineral fertilizers, classifies „Medeđa“ limestone as a good raw material that can be used as a filler for these purposes.
- The required quality of calcium carbonate for use in the animal feed industry is strictly defined by various regulations and legal regulations *Official Gazette of the Republic of Serbia* 2/90, 20/00, 4/2010; 54/2017. According to these requirements, Medjedja limestone in accordance with the Pb

content (Table 1) can be used for the preparation of feed mixtures and nutrients as well as phosphate mineral nutrients. As for the content of Cu, which is strictly regulated by regulations, Medjedja limestone can be used as a filler for the preparation of feed mixtures for calves up to 6 weeks of age, sheep, pigs up to 16 weeks of age and other categories of pigs and other domestic animals. Regardless of the strictly prescribed contents of heavy metals Mn, Zn and Cd for these purposes, „Međeda“ limestone with good quality and low content of these elements qualifies without problems for this application.

- The required quality of calcium carbonate for the neutralization of acid soils is strictly defined by the regulations in terms of both nutrients and heavy metals *Official Gazette of the Republic of Serbia* 60/00, 41/09, 84/2017). Regarding the nutrients such as CaCO₃ (CaO), MgO, P₂O₅, Zn, Cu and Co, Medjedja limestone has the satisfactory quality for this use. The content of K₂O, Fe and Mn was questioned. The content of K₂O is prescribed by the regulations up to a maximum of 0.009 %, while in „Međeda“ limestone the value of 0.051 % is calculated. The content of 276 ppm is prescribed for Fe, while 800 ppm is present in the limestone of „Međeda“ and the content of Mn is not determined precisely enough because the chemical analysis determined that it has less than 77.4 ppm, and it is prescribed that for this purpose the Mn content can be up to 44 ppm. In terms of harmful heavy metals Cd, Ni, Cr and Pb, „Međeda“ limestone satisfies in terms of Pb, while it is questionable in terms of Ni, Cd where it is slightly above the required values, and in terms of Cr, it is not determined precisely enough, because chemical analysis found that it has less than 68.42 ppm, and it is prescribed that for this purpose the Cr content may be up to 8 ppm.

5. CONCLUSION

Based on laboratory and technological tests on samples of limestone „Međeda“ - Niksic, it can be stated that by using mineral processing procedures can be obtained micronized samples that according to the analysis (coarse moisture, particle size distribution, bulk and particle density, whiteness, oil and water absorption, granulometric composition of micronized samples and specific surface area) can be a good basis to obtain fillers for various industries.

The limestone of the Medjedje deposit has a high content of CaCO₃ of 99.01 % and relatively low contents of MgO of 0.32 %, SiO₂ of 0.26 %, alkali and R₂O₃, with also low content of heavy metals, which is very favorable for limestones. In addition to these

physical and chemical properties, this limestone can be used in the following industries: paints and varnishes, paper, rubber and PVC, foundry and foundry industry, sugar industry, metallurgical purposes, glass production, animal feed production and mineral fertilizer production. A serious defect of „Međeda“ limestone is the low degree of whiteness, which does not exceed 89.6 %, which prevents its use in the pharmaceutical and chemical industries, while the relatively high content of K₂O, Fe and Mn prevents its use in calcification of acid soils.

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REZIME

TEHNOLOŠKA ŠEMA PROIZVODNJE KREČNJAČKOG PUNILA SA LEŽIŠTA "MEĐEDA" - NIKŠIĆ

U ovom radu su prikazani rezultati ispitivanja mogućnosti primene krečnjaka iz ležišta Međeđe-Nikšić u različitim granama industrije. Prilikom ispitivanja krečnjaka određena je vlaga, hemijski sastav, granulometrijski sastav, nasipna masa, specifična zapreminska masa, stepen beline, upijanje ulja i vode, granulometrijski sastav mikronizovanih uzoraka i specifična površina. Sva ispitivanja su obavljena na 8 uzoraka uzetih iz različitih delova ležišta a u radu su prikazane srednje vrednosti dobijenih rezultata. Na osnovu fizičko-hemijskih karakteristika može se konstatovati da krečnjak iz ovog ležišta ima visok sadržaj CaO i CaCO₃ od 99,01%, relativno nizak sadržaj MgO od 0,32%, nizak sadržaj SiO₂ od 0,26%, alkalija i R₂O₃, kao i nizak sadržaj teških metala, što je veoma povoljno za primenu krečnjak. Sa ovim fizičko-hemijskim karakteristikama, ispitivani krečnjak se može koristiti u sledećim granama industrije: boja i lakova, papira, guma i PVC, livarstvo i livnička industrija, industrija šećera, metalurgija, proizvodnja stakla, proizvodnja stočne hrane i proizvodnja mineralnih đubriva. Ozbiljna mana krečnjaka iz ležišta Međeđe je nizak stepen beline, koji ne prelazi 89,6%, što onemogućava njegovu upotrebu u farmaceutskoj i hemijskoj industriji, dok relativno visok sadržaj K₂O, Fe i Mn sprečava njegovu primenu za kalcifikaciju kiselih zemljišta.

Cljučne reči: krečnjak, punilo, industrijska upotreba, standardi, usitnjavanje i klasifikacija