

8th BALKAN MINING CONGRESS

PROCEEDINGS

September 28 – 30, 2022
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MINING INSTITUTE BELGRADE

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POSSIBILITY OF USING LIMESTONE FROM VUČIĆA BRIJEG – ULCINJ DEPOSIT AS FILLER IN VARIOUS INDUSTRY BRANCHES

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Abstract: This paper presents results of investigations of the possibility of using “Vučića Brijeg”-Ulcinj limestone (Republic Montenegro) as filler in various industry branches. Micronization methods, granulometric composition, oil and water absorption and degree of whiteness were investigated, and chemical and thermal analyses (DT/TG) were performed.

Physico-chemical properties of this limestone classify it among high quality carbonate raw materials with high CaCO_3 content of 97.87%, as well as MgO content of 1.04% (2,18% content of MgCO_3) and low silicate content (SiO_2 0.40%). Its quality satisfies requirements of standards on using of calcium carbonate as filler in industry of paints and coatings; paper industry; rubber and PVC industry; glass industry; production of mineral fertilizers; foundry industry; sugar industry and metallurgy. Due to the low degree of whiteness (81.65%) „Vučića Brijeg” limestone cannot be used in pharmaceutical and cosmetics industry. Due to relatively high content of heavy metals, Pb (55 ppm), Cu (10 ppm), Ni (23ppm) and Cd (5 ppm), “Vučića Brijeg”-Ulcinj limestone cannot be used, in production of cattle feed and for neutralization of acidic soils.

Key words: FILLER, INDUSTRIAL USE, LIMESTONE, STANDARDS

INTRODUCTION

Republic of Montenegro has big reserves of limestone in coastal area and in south of the territory [1]. Even though deposits are huge, limestone is mainly used

in construction as construction stone, and to some extent as architectural stone [2]. Since calcium carbonate as filler is much more expensive than construction stone, relevant institutions of Montenegro initiated investigations of the possibility of using limestone as filler [3]. On the basis of the obtained results it was evaluated whether it can be used as filler in accordance with standards (SRPS) in various industry branches [3-6]. “Vučića Brijeg”-Ulcinj deposit consists of carbonate sediments, mostly limestone ones, and less dolomitic sediments. Ore reserves are estimated at about 4,000,000 t of limestone [1]. The aim of investigations presented in this paper was to determine the possibility of using raw material as filler in various industry branches.

EXPERIMENTAL

Materials and methods

Starting limestone sample used in investigations was from “Vučića Brijeg“ - Ulcinj deposit. First, its specific volumetric weight (density) and granulometric composition were determined. Its density was measured by pycnometer with xylol as fluid, granulometric composition was determined by Tyler screen [7]. Granulometric composition of the micronized sample was determined by sieve size 63 μm , classification on CycloSizer and Bach elutriator. Limestone filler quality was determined by chemical analysis. Thermal (DT/TG) analysis of the sample was performed using Netzsch-Simultaneous Thermal Analysis- STA 409 EP device, with heating speed of $\Delta T = 10$ $^{\circ}\text{C}/\text{min}$, in temperature interval from 20 to 1000 $^{\circ}\text{C}$. Degree of whiteness was determined by whiteness meter, according to MgO 100% standard.

Investigation of physical properties of starting sample

Specific volumetric weight of the starting sample is $\gamma = 2,673$ g/cm^3 . The granulometric composition data, obtained by sieving, are shown in Figure 1, for the

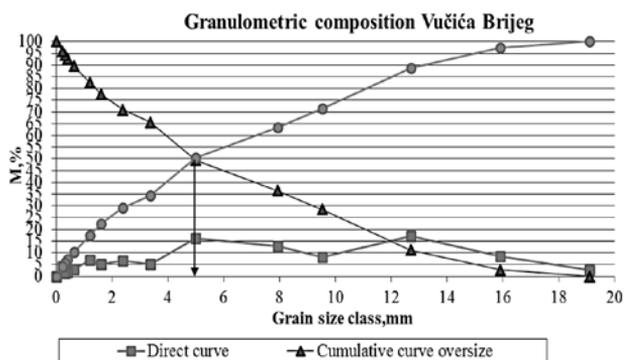


Figure 1 The curves of particle size-composition of the starting sample „Vučića Brijeg“ - Ulcinj [1]

limestone sample of “Vučića Brijeg«. From the intersection of cumulative curves average outflow and flow determined that the average diameter of the sample of limestone $d_{50} = 4.93$ mm, and the upper size limit of the sample was 15.092 mm.

Technological investigations

For investigations of the possibility of using limestone as filler in various industry branches limestone was micronized, and thus obtained product were subjected to the following physico-chemical characterization:

-chemical analysis, thermal (DT/TG) analysis, determination of granulometric composition, degree of whiteness and absorption of oil and water.

Determining granulometric composition of micronized sample

Table 2. Granulometric composition of grinded sample Vučića Brijeg

Size class [μm]	M, %	$\downarrow\sum M, \%$	$\uparrow\sum, \%$
+ 63	33.61	33.61	100.00
-63+44	2.06	35.67	66.39
-44+33	5.57	41.24	64.33
-33+23	5.84	47.08	58.76
-23+15	3.92	51.00	52.92
-15+11	3.06	54.06	49.00
-11+5.7	29.40	83.46	45.94
-5.7+0	16.54	100.00	16.54
Feed	100.00	/	/

Granulometric composition of the micronized products showed that the finest class -5.7 μm content is around 16,5%.

Determining the degree of whiteness, oil and water absorption, and particle density

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 3 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 %.

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 3.

Specific weight was determined using standard method SRPS EN 1936:2009. All specific weights were determined on three samples and the data in the report are in fact the mean value, shown in Table 3.

Table 3. Physico-chemical characteristics of the limestone Vučića Brijeg

Sample mark	Whiteness according to MgO – 99.0 %	Oil absorption %	Water absorption %	Particle density g/cm ³
Vučića Brijeg-1	84.3	11.15	15.75	2.732
Vučića Brijeg -2	83.3	12.80	20.45	2.716
Vučića Brijeg -3	87.0	12.10	17.45	2.684
Average value	85.4	11.70	15.00	2.701

Thermal (DT/TG) analysis

Results of thermal (DTA/TG) analysis of the micronized sample “Vučića Brijeg” limestone are presented as a diagram in Figure 2.

In Figure 2 are presents the TG and DTA diagrams of the initial sample of limestone. DTA diagram (Figure 2.) shows endothermic peak with maximum at 868 °C, which is attributed to phase transformation of calcite (CaCO₃) into CaO, according to the following reaction:



This phase transformation is accompanied by weight loss of 43.32% (TG diagram, Figure 2) in the temperature range from 650 °C to 900 °C.

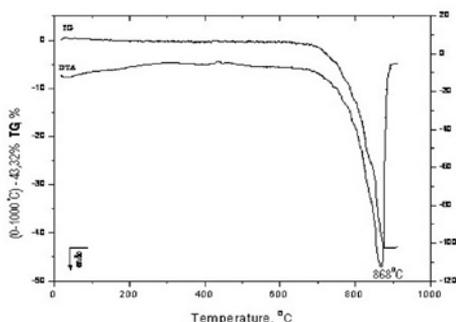


Figure 2. DTA/TG diagram of “Vučića Brijeg” limestone sample [1]

Chemical analysis

Results of chemical analysis of the micronized limestone “Vučića Brijeg” with contents of main components and damaging components are presented in Tables 4. and 5.

Table 4. Chemical composition of main components of limestone sample

Comp.	CaO	CaCO ₃	CO ₂	MgO	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	K ₂ O	Na ₂ O	TiO ₂	P ₂ O ₅	R ₂ O ₃	LOI
Content, %	54.84	97.87	43.38	1.04	0.031	0.085	0.40	0.0096	0.032	<0.02	<0.005	0.092	43.46

Table 5. Chemical composition of damaging components of limestone sample

Comp.	Cu	Mn	S	P	Ni	Cr	Mo	Sb	Pb	Cd	pH	Fe rast.	As	Hg
Content, %	13 ppm	10 ppm	<0.01 %	<0.005	23 ppm	3 ppm	<50 ppm	<25 ppm	55 ppm	5 ppm	9.14	0.024 %	/	/

Results of physico-chemical characterization of “Vučića Brijeg” limestone sample and the required filler quality (Standards) lead to conclusion that this limestone is of good quality. Namely, its CaCO₃ content is high- 97.87%, and MgCO₃ (2,18%) and silicates (SiO₂ 0.40%) content low. However, relatively high content of heavy metals was found, above all Pb (55 ppm), Ni (23 ppm), Cu (13 ppm) and Cd (5 ppm).

RESULTS AND DISCUSSION

Limestone filler quality for each industry branch is defined by appropriate standards or requirements of manufacturers who use limestone as raw material in their production cycle. Limestone quality requirements are defined as content of useful and damaging components, i.e. as chemical composition, as well as the necessary size class.

Evaluation of “Vučića Brijeg”-Ulcinj limestone filler quality based on chemical composition

According to the results presented above, limestone from “Vučića Brijeg” – Ulcinj deposit can be used in the following industries:

- In industry of paints and coatings; it is among high quality raw materials in accordance with market and standard requirements (SRPS EN ISO 3262-5:2009); in paper industry; it is among A quality, while for the highest B, C

i D quality classes its whiteness degree is not satisfying (SRPS B.B6.033); in rubber and PVC industry; it satisfies the highest quality standards and market requirements (SRPS B.B6.031); in foundry industry; it belongs to the highest class I in accordance with market requirements imposed by standard (SRPS B.B6.012); in sugar industry; it is among the II class (due to the increased MgO content) in accordance with market and standard requirements (SRPS B.B6.013); in metallurgy; it is in the highest class I in accordance with market requirements imposed by standards (SRPS B.B6.011); in production of glass; due to the increased Fe_2O_3 and MgO content it is in quality category IV and V in accordance with market requirements imposed by standards (SRPS B.B6.020);

Limestone from “Vučića Brijeg” – Ulcinj deposit cannot be used:

- In pharmaceutical and cosmetics industry because its low whiteness degree and increased content of heavy metals Pb and Cd relative to market requirements defined by standard (SRPS B.B6.034); for production of mineral fertilizers because of the increased MgO content, which is strictly defined by manufacturer’s requirements (Azotara Pančevo); in production of cattle feed because of the increased content of heavy metals Pb, Cu and Cd, which is very strictly defined for this use (“Official Gazette of the Republic of Serbia 2/90, 20/00, 4/2010; 54/2017); for neutralization of acidic soils; because of the increased content of MgO as biogenic element and heavy metals Pb, Ni and Cd, the contents of which are very strictly defined (“Official Gazette of the Republic of Serbia” 60/00, 41/09, 84/2017).

Evaluation of limestone filler quality based on users’ requirements for the necessary raw material size (fineness)

Some industries require finely micronized limestone, while others require raw material of larger particle size, sometimes even coarse. Following industries use ground and micronized limestone:

- For paints and coatings industry; A quality 99.5% of - 20 μm , B quality 97% of -20 μm and 0.01% of + 44 μm ; for paper industry for quality categorie A the required fineness is 100% of -45 μm , and 75% of -10 μm ; rubber and PVC industry requires for A and B quality raw material to be 99.5% of -45 μm , while for C and D quality upper limit limestone size is 45 μm ; for glass industry, since “Vučića Brijeg” limestone corresponds to quality IV and V according to its chemical composition, there is predefined granulometric composition for these quality classes, subdivided into six subclasses in size range from -1+0.1mm;

Following industries demand larger sizes and coarse limestone:

- For foundry industry, raw material should be size -50+30 mm, with class – 30 mm content up to 5%<; for sugar industry, limestone is to be classified into six subclasses in size range from -215+63mm, with maximum fine content in each subclass up to 8%; metallurgy uses limestone consisting of five subclasses in size range from -70+0.1mm.

CONCLUSION

Limestone from “Vučića Brijeg” - Ulcinj deposit according to its physico-chemical properties belongs to high quality carbonate raw material with high content of CaCO_3 of 97.87%, and low content of MgCO_3 of 2.18% and silicates (SiO_2 0.40%). It meets the requirements of standards for using calcium carbonates as fillers in industry of paints and coatings; paper industry, rubber and PVC industry; production of mineral fertilizers; foundry industry; sugar industry and metallurgy. According to market demand and standards it belongs to high quality raw material in industry of paints and coatings, rubber and PVC, foundry industry and metallurgy. However for paper and sugar industry and glass production it does not conform to standards. Because of increased MgO content “Vučića Brijeg” limestone cannot be used in fertilizers industry. Due to low whiteness degree and increased content of heavy metals Pb (55 ppm) and Cd (5 ppm) “Vučića Brijeg” limestone cannot be used in pharmaceutical and cosmetics industry. Due to high content of heavy metals Pb (55 ppm) Cu (13ppm) and Cd (5 ppm), “Vučića Brijeg” limestone cannot be used in production of cattle feed. Due to high content of heavy metals Pb (55 ppm), Ni (23 ppm) and Cd (5 ppm), as well as biogenic elements MgO (1.04%), “Vučića Brijeg” limestone cannot be used for neutralization of acidic soils

Obtaining of wide range of fillers for various industry branches would provide products which are more expensive per mass unit than products that have been used until now up to 10 times.

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