
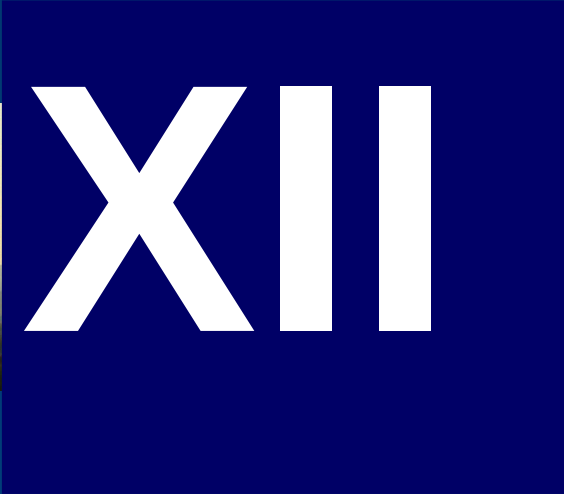


UNIVERSITY OF BELGRADE
TECHNICAL FACULTY IN BOR

PROCEEDINGS



XII

International Symposium on RECYCLING TECHNOLOGIES and SUSTAINABLE DEVELOPMENT

Editors:
Grozdana Bogdanović
Milan Trumić

Hotel Jezero, Bor Lake, Serbia
13 – 15 September 2017



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POSSIBILITY OF USING LIMESTONE FROM “GIGOVIĆI”-ULCINJ DEPOSIT AS FILLER IN VARIOUS INDUSTRY BRANCHES

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ABSTRACT – This paper presents results of investigations of the possibility of using “Gigovići”-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 relatively high CaCO_3 content of 98.16%, as well as low MgCO_3 content of 1.53% and low silicate content (SiO_2 0.30%). 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 (85.25%) “Gigovići” limestone cannot be used in pharmaceutical and cosmetics industry. Due to relatively high content of MgO (0.73%) and Fe (340ppm), as well as high content of heavy metals, Cu (18 ppm), Ni (24 ppm) and Cd (9 ppm), “Gigovići” limestone cannot be used, in production of cattle feed and for neutralization of acidic soils.

Keywords: limestone, filler, industrial use, standards, comminution and classification; recycling of industrial minerals

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

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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].

"Gigovići"-Ulcinj deposit consists of carbonate sediments, mostly limestone ones, and less dolomitic sediments. Ore reserves are estimated at about 5,000,000 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.

MATERIALS AND METHODS

Starting limestone sample used in investigations was from "Gigovići" - 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 $\Delta T = 10\text{ }^{\circ}\text{C}/\text{min}$, 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 $\gamma = 2,708\text{ g}/\text{cm}^3$.

Table 1. Granulometric-composition of the initial sample "Gigovići" - Ulcinj

Size class [mm]	M, %	$\downarrow \Sigma M, \%$	$\uparrow \Sigma M, \%$
- 19,1 + 15,9	3,68	3,68	100,00
- 15,9 + 12,7	9,48	13,16	96,32
- 12,7 + 9,52	17,23	30,39	86,84
- 9,52 + 7,93	7,88	38,27	69,61
- 7,93 + 5,0	13,83	52,10	61,73
- 5,0 + 3,36	14,99	67,09	47,90
- 3,36 + 2,38	4,74	71,83	32,91
- 2,38 + 1,6	5,98	77,81	28,17
- 1,6 + 1,19	4,91	82,72	22,19
- 1,19 + 0,63	6,63	89,35	17,28
- 0,63 + 0,4	2,93	92,28	10,65
- 0,4 + 0,3	1,69	93,97	7,72
- 0,300 + 0,200	1,62	95,59	6,03
- 0,200 + 0,000	4,41	100,00	4,41
Input	100,00	/	/

Based on data from the table is drawn a diagram of particle size distribution shown in Figure1, for samples of limestone "Gigovići". In Figure1, shows the direct curve of particle size distribution and cumulative curves and average sample of outflow and flow limestone deposits "Gigovići" - Ulcinj. From the intersection of cumulative curves average outflow and flow determined that the average diameter of the sample of limestone $d_{50}=8.11\text{mm}$, and upper size limit of the sample was $d_{95}=16.334\text{ mm}$.

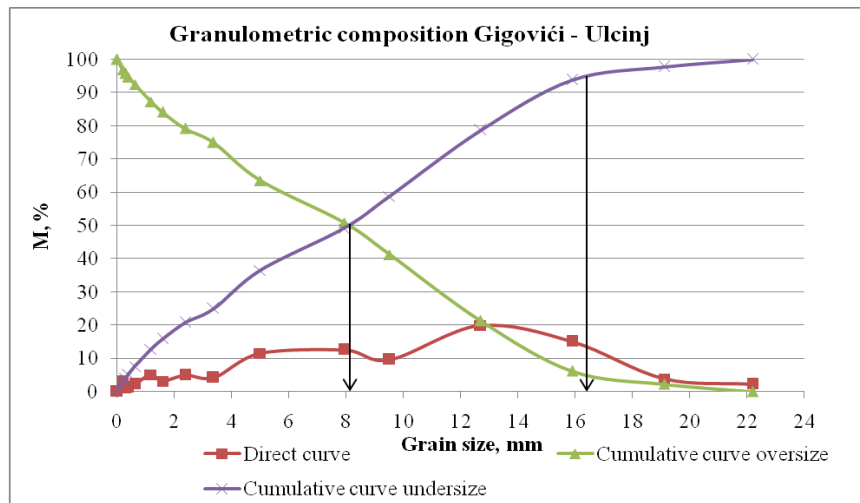


Figure 1. The curves of particle size-composition of the starting sample "Gigovići"-Ulcinj

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 "Gigovići"

Size class [μm]	M, %	$\downarrow \Sigma M, \%$	$\uparrow \Sigma M, \%$
+ 63	38,24	38,24	100,00
-63+44	5,17	43,41	61,76
-44+33	5,61	49,02	56,59
-33+23	4,99	54,01	50,98
-23+15	3,63	57,64	45,99
-15+11	3,51	61,15	42,36
-11+5,7	28,86	90,01	38,85
-5,7+0	9,99	100,00	9,99
Input	100,00	/	/

Granulometric composition of the micronized products showed that the finest class -5.7 μm content is around 10%.

Determining the degree of whiteness

Whiteness was assessed on three samples of the limestone from deposit "Gigovići", and the result is shown in Table 3.

Table 3. The degree of whiteness the limestone samples

No	mark of the sample	whiteness according MgO– 100%
1.	"Gigovići"-1	85.30
2	"Gigovići"-2	85.10
3	"Gigovići"-3	85.35
	Average value	85.25

Determination of absorption water and oil

In order to determine absorption water and oil are also used three samples of the limestone from deposit "Gigovići", and the results are shown in Tables 4 and 5.

Table 4. Absorption of the oil of samples of limestone

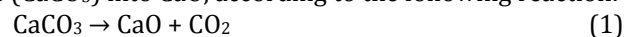
No.	mark of the sample	absorption of the oil, %
1.	"Gigovići"-1	13,82
2.	"Gigovići"-2	13,75
3.	"Gigovići"-3	13,77
	Average value	13,78

Table 5. Absorption of the water of samples of limestone

No	mark of the sample	absorption of the water, %
1.	"Gigovići"-1	18,81
2.	"Gigovići"-2	19,07
3.	"Gigovići"-3	19,13
	Average value	19,00

Thermal (DT/TG) analysis

Results of thermal (DTA/TG) analysis of the micronized sample "Gigovići" 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 871⁰C, which is attributed to phase transformation of calcite (CaCO_3) into CaO, according to the following reaction:



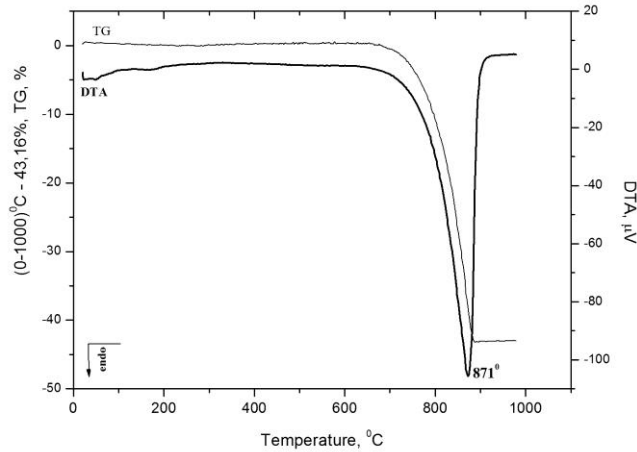


Figure 2. DTA/TG diagram of "Gigovići" limestone sample

This phase transformation is accompanied by weight loss of 43.16% (TG diagram, Figure 2) in the temperature range from 650°C to 900°C.

Chemical analysis

Results of chemical analysis of the micronized limestone with contents of main components and damaging components are presented in Tables 6. and 7.

Table 6. Chemical composition of main components of limestone sample

Comp.	CaO	CaCO ₃	CO ₂	MgCO ₃	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	K ₂ O	Na ₂ O	TiO ₂	P ₂ O ₅	LOI
Cont., %	55,00	98,16	43,55	0,730	0,055	0,023	0,30	0,0064	0,022	<0,02	<0,005	43,80

Table 7. Chemical composition of damaging components of limestone sample

Comp.	Cu	Mn	S	P	Ni	Cr	Mo	Sb	Pb	Cd	pH	Fe solu.	As	Hg
Cont., %	18ppm	36ppm	<0,01	<0,025	24ppm	3ppm	<50 ppm	<25 ppm	65 ppm	9ppm	9,26	0,0340	/	/

Results of physico-chemical characterization of "Gigovići" limestone sample and the required filler quality (Standards) lead to conclusion that this limestone is of good quality. Namely, its CaCO₃ content is high- 98.16%, and MgCO₃ (1.53%) and silicates (SiO₂ 0.3%) content low. However, relatively high content of heavy metals was found, above all Cu (18 ppm), Ni (24 ppm) and Cd (9 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 "Gigovići" limestone filler quality based on chemical composition

According to the results presented above, limestone from "Gigovići" – 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 B.B6.032);
- in paper industry; it is among A, B and C quality, while for the highest D quality 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; due to the increased MgO content it belongs to the highest class 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 MgO, SiO₂ and Fe₂O₃ contents it is in quality category IV and V in accordance with market requirements imposed by standards (SRPS B.B6.020);
- for production of mineral fertilizers because of the increased MgO content, which is strictly defined by manufacturer's requirements (Azotara Pančevo).

Limestone from "Gigovići" – Ulcinj deposit cannot be used:

- in pharmaceutical and cosmetics industry because its low whiteness degree relative to market requirements defined by standard (SRPS B.B6.034);
- in production of cattle feed because of the increased content of heavy metals Pb and Cd, which is very strictly defined for this use ("Official Gazette of the Republic of Serbia" 31/78, 6/81, 2/90, 20/00);
- for neutralization of acidic soils; because of the increased content of biogenic elements MgO, Fe and Cu, and heavy metals, Pb, Ni, and Cd, the contents of which are very strictly defined ("Official Gazette of the Republic of Serbia" 60/2000).

Evaluation of "Gigovići" – Ulcinj 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 - 20µm, B quality 97% of -20µm and 0.01% of + 44µm;

- for paper industry for all quality categories (A, B and C) the required fineness is 100% of -45 μ m, where for A quality 75% of -10 μ m, for B quality 80%, and for C quality the required fineness is 95% of -10 μ m and 90% of -2 μ m;
- rubber and PVC industry requires for A and B quality raw material to be 99.5% of -45 μ m, while for C and D quality upper limit limestone size is 45 μ m;
- for glass industry, since "Gigovići" 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 "Gigovići" - Ulcinj deposit according to its physico-chemical properties belongs to high quality carbonate raw material with high CaCO₃ content of 98.16%, and low MgCO₃ content of 1.53% and silicates (SiO₂ 0.3%). 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, sugar production, foundry industry and metallurgy. However, for paper industry and production of glass, this limestone does not comply with the highest standards.

Due to the low degree of whiteness, and increased content of heavy element (Pb 65ppm) "Gigovići" limestone cannot be used in pharmaceutical and cosmetics industry. Due to high content of biogenic elements MgO (0.73%), Fe (340 ppm) and Cu (18ppm), as well as heavy metals Pb (65ppm), Ni (24 ppm) and Cd (9 ppm), "Gigovići" limestone cannot be used in production of cattle feed and for neutralization of acidic soils.

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