

UNIVERSITY OF BELGRADE
TECHNICAL FACULTY BOR

**52nd International October Conference on
Mining and Metallurgy**



PROCEEDINGS

Edited by

Saša Stojadinović

and

Dejan Petrović

November 29th – 30th 2021

Bor, Serbia

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POSSIBILITY OF USING LIMESTONE FROM “PJEŠIVAČKI DO”- DANILOVGRAD DEPOSIT AS FILLER IN VARIOUS INDUSTRY BRANCHES

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Abstract

This paper presents results of investigations of the possibility of using “Pješivački Do”-Danilovgrad limestone 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 CaCO₃ content of 98.65%, as well as low MgCO₃ content of 0.95% and low silicate content (SiO₂ 0.23%). 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; in production of cattle feed; glass industry; production of mineral fertilizers and sugar industry. Due to the low degree of whiteness (87,6%) Pješivački Do” limestone cannot be used in pharmaceutical and cosmetics industry. Due to relatively high content of Cr (10 ppm) as heavy metal, content of S (0,19%), as well as biogenic elements P₂O₅ (0.030%), “Pješivački Do” limestone cannot be used in, foundry industry and metallurgy and for neutralization of acidic soils.

Keywords: limestone, filler, industrial use, standards.

1. INTRODUCTION

The limestone is a sedimentary rock comprising predominantly carbonate minerals (i.e. calcium carbonate, CaCO₃) in the quantity surpassing 50%. Calcite, aragonite and/or vaterite mineral phases are carbonates usually appearing in the limestone. However, the calcite [1-3] is the only crystal form with real significance. Being one of the most common minerals on Earth, the calcite (CaCO₃) occupies about 4% of the *Earth's crust* [4] mass. Calcite is an important resource and subject of investigations in various scientific fields: mineralogy, chemistry, physics, materials science [1, 5-7]. Republic of Montenegro has big reserves of limestone [3, 8]. Even though deposits are huge, limestone is mainly used in construction as construction stone, and to some extent as architectural stone [3, 8]. 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, 8]. 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, 8]. “Pješivački Do”-Danilovgrad deposit consists of carbonate sediments, mostly limestone ones, and less dolomitic sediments. Out of total reserves of 5,000,000 t of limestone, it is mainly used in construction as construction stone. The aim of the investigations presented in this paper was to determine the possibility of using this material, to obtain filler for application, in various industry branches.

2. EXPERIMENTAL

2.1 Materials and methods

Starting limestone sample used in investigations was from “Pješivački Do”-Danilovgrad 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. 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.

2.2 Investigation of physical properties of starting sample

Specific volumetric weight of the starting sample is $\gamma = 2,632\text{ g}/\text{cm}^3$, ggk of the sample is 16.67 mm, and $d_{50} = 6.98\text{ mm}$.

2.3 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.

2.3.1 Characterization of micronized product

Granulometric composition of the micronized products showed that ggk is 33 μm , and that the finest class -5.7 μm content is 58.84%, whiteness is 87.60%, oil absorption 13.90% and water absorption 15.50%.

2.3.2 Thermal (DT/TG) analysis

Results of thermal (DTA/TG) analysis of the micronized “Pješivački Do” limestone are presented as a diagram in Figure 1.

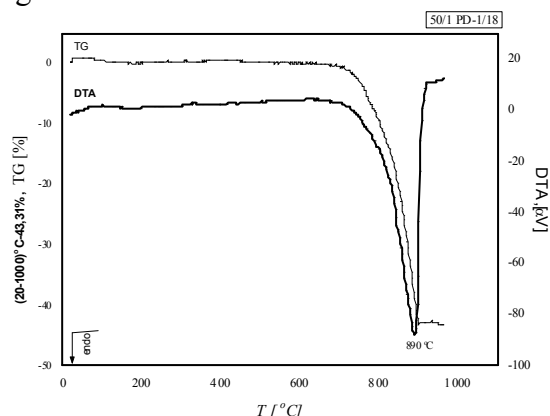


Figure 1. DTA/TG diagram of “Pješivački Do” limestone sample

DTA diagram (Figure 1.) shows endothermic peak with maximum at 890 $^{\circ}\text{C}$, which is attributed to phase transformation of calcite (CaCO_3) into CaO , which is followed by mass loss of 43.31% in temperature range from 650 $^{\circ}\text{C}$ to 900 $^{\circ}\text{C}$ (TG diagram, Figure 1.).

2.3.3 Chemical analysis

Results of chemical analysis of the micronized limestone with contents of main components and damaging components are presented in Table 1 and Table 2.

Table 1. 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., %	55,24	98,65	43,41	0,95	0,053	0,026	0,23	0,012	0,064	<0,02	<0,030	43,80

Table 2. Chemical composition of damaging components of limestone sample

Comp.	Pb	Cu	Mn	S	Ni	Cr	Mo	Co	Cd	pH	Fe solu.	As	Hg
Cont., %	<10ppm	<0,5ppm	6,99ppm	0,19	<2ppm	10ppm	<50 ppm	<10ppm	<0,5ppm	9,35	0,0430	/	1,2ppm

Results of physico-chemical characterization of “Pješivački Do” limestone sample and the required filler quality (Standards) lead to conclusion that this limestone is of good quality. Namely, its CaCO₃ content is high- 98.65%, and MgCO₃(0.95%) and silicates (SiO₂0.23%) content low. However, an increased content of Cr (10ppm) as heavy metal in the sample was found.

3. 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.

3.1 Evaluation of “Pješivački Do” limestone filler quality based on chemical composition

According to the results presented above, limestone from “Pješivački Do” – Danilovgrad 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 the paper industry; it is among A, B quality, while for the higher quality C and the highest quality D class 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 the sugar industry, it is among the highest I class in accordance with market and standard requirements (6, SRPS B.B6.013); in the production of glass, due to the increased content of MgO (0.452%) and SiO₂ (0.23%) can be classified from II to V quality class, while due to the increased content of Al₂O₃ (0.026%) and Fe₂O₃ (0.053%) can be classified in IV and V class in accordance with the market requirements given by the standard (SRPS B.B6.020); in the production of cattle feed, it can be used in accordance with a defined standard (“Official Gazette of the Republic of Serbia 2/90, 20/00, 4/2010; 54/2017); it can be used for production of mineral fertilizers since its quality is in accordance with manufacturer’s requirements (Azotara Pančevo).

Limestone from “Pješivački Do” – Danilovgrad deposit cannot be used:

- in the pharmaceutical and cosmetic industry due to the low degree of whiteness in relation to the market requirements defined by the standard (SRPS B.B6.034); for neutralization of acidic soils, because of the increased content of P₂O₅ (0,030%) as a biogenic element, the content of which is very strictly defined (“Official Gazette of the Republic of Serbia” 60/00, 41/09, 84/2017), due to the increased content of S (0,19%), it does not meet market requirements and standards in the foundry industry (SRPS B.B6.012) or in metallurgy (SRPS B.B6.011).

3.2 Evaluation of “Pješivački Do” –Danilovgrad 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 μ m, B quality 97% of -20 μ m and 0.01% of + 44 μ m; for paper industry for both quality categories (A and B) the required fineness is 100% of -45 μ m, where for A quality 75% of -10 μ m, for B quality 80%; 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 “Pješivački Do” 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; for production of mineral fertilizers “Azotara”- Pančevo does not define size classes limestone should meet for this purpose.

Following industries demand larger sizes and coarse limestone:

- 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%.

4. CONCLUSION

Limestone from “Pješivački Do”- Danilovgrad deposit according to its physico-chemical properties belongs to high quality carbonate raw material with high content of CaCO₃ of 98.65%, and low content of MgCO₃ of 0.95% and silicates (SiO₂ 0.23%). It meets the requirements of standards for using calcium carbonates as fillers in industry of paints and coatings; paper industry; rubber and PVC industry; glass industry; production of mineral fertilizers; in the production of cattle feed; sugar industry. According to market demand and standards it belongs to high quality raw material in industry of paints and coatings, rubber and PVC, in the production of cattle feed, sugar industry, and for production of mineral fertilizers. However, for paper and glass production it does not comply with the standards for the highest quality classes. Due to the low degree of whiteness, and an increased content of: S (0.19%), heavy metal Cr (10 ppm), as well as the biogenic element P₂O₅ (0.03%), limestone “Pješivački Do” cannot be used in the pharmaceutical and cosmetic industry, for neutralization of acidic soils, in the foundry industry and in metallurgy. 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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