

CHARACTERIZATION OF THE PARTICLE SIZE FRACTIONS OF BENTONITE CLAY FROM GINOVCI, REPUBLIC OF NORTH MACEDONIA

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A b s t r a c t: The examined bentonite clay contains montmorillonite as a basic mass. As an admixtures it contains quartz, illite and other minerals. The admixtures are inhomogeneously distributed in various particle size fractions. The subject of this research is concentration and separation of existing admixtures from bentonite clay (–0.063 mm), a commercial product of Bentomak. Wet sieve analysis and elutriation were applied as separation methods. In order to define the efficiency of the methods, a complex examination for characterization of the fractions was realized. Simultaneous review of the results of XRD, microscopic examinations and chemical analysis indicates that in the finest fraction (–0.032 mm) the concentration of the admixtures is smaller for 80% than in the other fractions.

Key words: bentonite clay, admixtures, particle size fractions, wet sieve analysis, elutriation

INTRODUCTION

Bentonite clays are widespread in the north-eastern region of the Republic of North Macedonia. Clay sediments were formed in Tertiary basins due to volcanic activity. Clay deposits were discovered in the Kratovo-Zletovo volcanic area, precisely in Slaviško Pole (Kriva Palanka region) and in the catchment area of the river of Pčinja (Kumanovo region). Bentonite clay deposits are located in the villages of Rankovce, Ginovci and Ljubanci (Figure 1).

The clay deposit in Rankovce is located in the western part of Slaviško Pole. Bentonite clay was formed by the process of alteration of volcanic tuffs, and exist in a Pliocene sediments. The deposit has three layers of bentonite. At the bottom is a layer about 6 m thick, with a basic tuff mass. Then there is a layer of montmorillonite clay, about 25 m thick. Above is a layer of bentonite about 11 m thick, which has several layers of sandy clay. The deposits in Ginovci and Ljubanci are contiguous, and are located east of Rankovce. Two layers of bentonite clay about 1 m thick are present in a Pliocene sediments (Spasovski et al., 2003; Stojanović, 2005).

Bentomak Mines exploit the clay deposits in Slaviško Pole. The company has a surface exploitation of fine dispersed bentonite, and manufacture

several types of bentonite products with high quality: alkali-activated bentonite, casting grade bentonite, drilling mud bentonite, injecting grade bentonite, bentonite grade used for pelletizing processes of the ore concentrates, and as filter (Figure 2).



Fig. 1. Geographical location of the deposits



Fig. 2. Bentomak production plant

In order to determine the composition, structure and properties, the bentonite clay mined from these deposits was the subject of many studies and examinations (Atkovska et al., 2016; Boševska 1980; Brezovska; 1996; Brezovska et al., 2004, 2005; Memedi et al., 2017).

MATERIALS AND METHODS

In this research the bentonite clay (-0.063 mm), a commercial product of Bentomak, was examined (Figure 3).



Fig. 3. Bentonite clay (-0.063 mm), a commercial product of Bentomak

For characterization of the bentonite clay, as well as the separated fractions were applied: che-

Montmorillonite has a dominant mass content in the bentonite clays. Also as admixtures there are quartz, tridymite, opal, calcedon, including hydro-micas, particularly in the teregenic form. The weakly expressed boundaries of montmorillonite crystals and higher negative electric charge of their layered crystal lattice provide a large ionic - exchange capacity. The presented mixtures have a negative impact on the properties of the bentonite clay. Therefore, there is a tendency to upgrade the technological process in manufacturing bentonite clay with minor quantities of admixtures. Bentonite clay separated of admixtures has a high profitable implementation (Guera et al., 2013; Khan et al., 2017; Moosavi 2017; Park et al., 2016).

The main goal of this research, is to separate admixtures from bentonite clay. Wet sieve analysis and elutriation were applied as methods for separation of the existing admixtures (Knežević, 2001; Nikolovski, 1995; Zafirovski et al., 1974; Alves et al., 2016; Barakan et al., 2019; Sarbatly et al., 2009; Bogoevski et al., 2016).

mical analysis, XRD analysis, DTA/TGA and mineralogical-petrographic examinations.

Chemical composition was defined by silicate chemical analysis (Zafirovski et al., 1974; Bogoevski et al., 2014; Reka et al., 2014; Pavlovski et al., 2011). Existing minerals were determined on a DRON X-ray diffractometer ($2\theta = 2-60^\circ$; $U_A = 38$ kV; $I_A = 18$ mA; $1^\circ/\text{min.}$; $\text{CuK}\alpha/\text{Ni}$). DTA/TGA were performed on a Perkin Elmer-7 Diamond instrument, in an air atmosphere with rate of heating $10^\circ/\text{min}$ from ambient to 1000°C . Mineralogical-petrographic examinations were realized on Carl Zeiss Jena optical microscope, as well on FEI Quanta 3D FEG electron microscope.

A set of standard sieves with perforation size of 0.063 mm, 0.050 mm, 0.040 mm and 0.032 mm were used for a wet sieve analysis on 50 g of bentonite clay. Elutriation was performed on a glass tube with diameter of 42 mm, discontinuously on two-stage with various flow of water in opposite direction. Elutriation 1 with water flow of $Q_1 = 180$ $\text{cm}^3/\text{min.}$, and Elutriation 2 with $Q_2 = 120$ $\text{cm}^3/\text{min.}$ A suspension of 10 g bentonite clay and 300 cm^3 of distilled water was prepared for elutriation. In order to prevent aggregation of clay particles, the suspension was heated up to 50°C and several drop of NH_4OH were added.

RESULTS AND DISCUSSION

The commercial product of bentonite clay was initially characterized. The average chemical composition is presented in Table 1.

Some deviations in the chemical and mineralogical composition of the clay are a consequence of natural variations in the deposits. According to XRD presented on Figure 4, the commercial product of bentonite clay contains montmorillonite, quartz, calcium carbonate and sodalite.

DTA/TGA curves are presented on Figure 5. Characteristically for montmorillonite, DTA curve has two endothermic peaks. In correlation, TGA curve has an intensive loss of mass (hygroscopic water) up to 200°C, and loss of mass (structural water) at temperature range of 600–800°C.

Table 1

Average chemical composition of the commercial product of bentonite clay (mass %)

SiO ₂	54.56
Al ₂ O ₃	11.74
Fe ₂ O ₃	5.43
CaO	9.15
MgO	0.72
K ₂ O	0.90
Na ₂ O	1.97
SO ₃	0.50
l.w	14.28
Σ	99.25

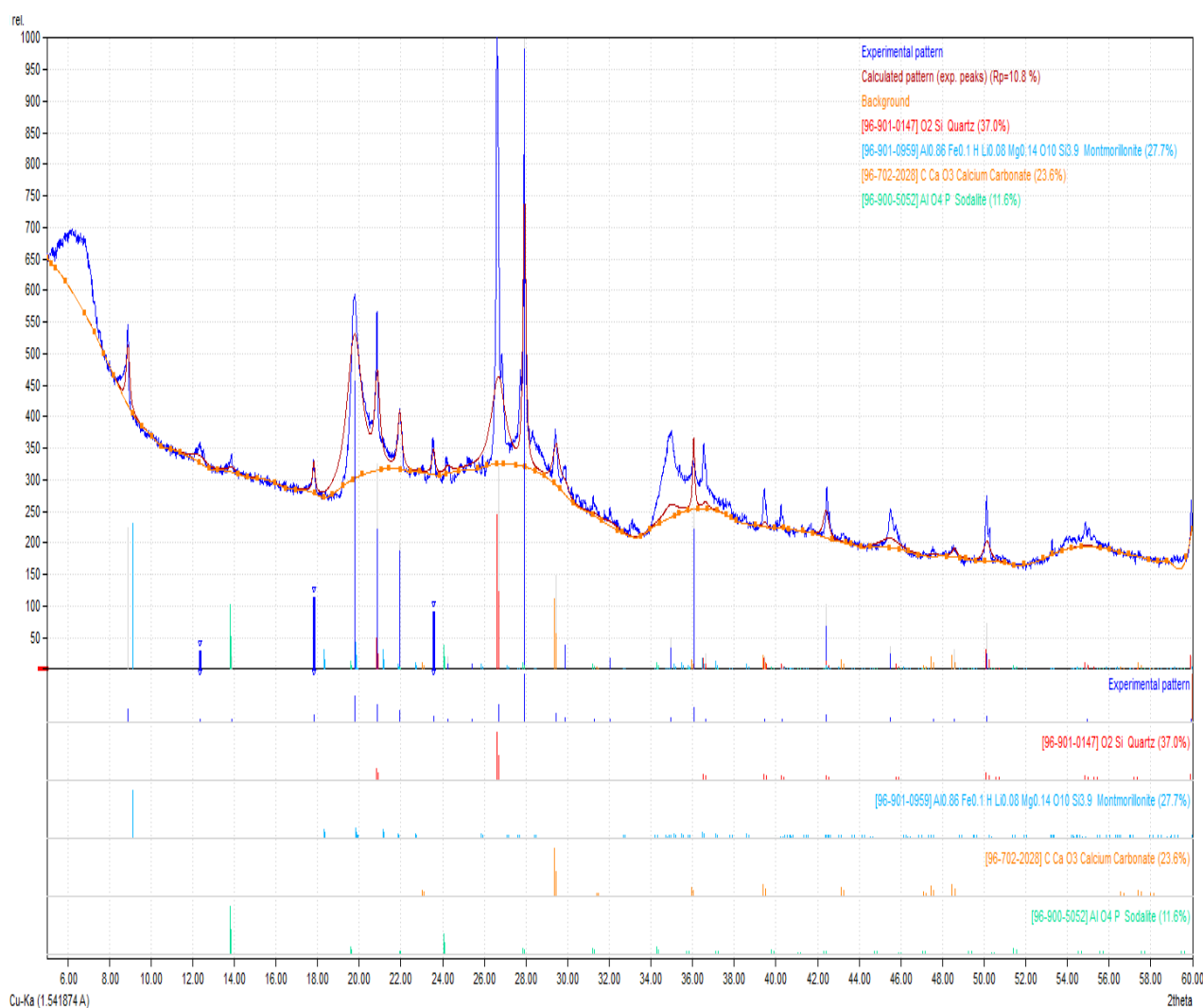


Fig. 4. XRD of the commercial product of bentonite clay

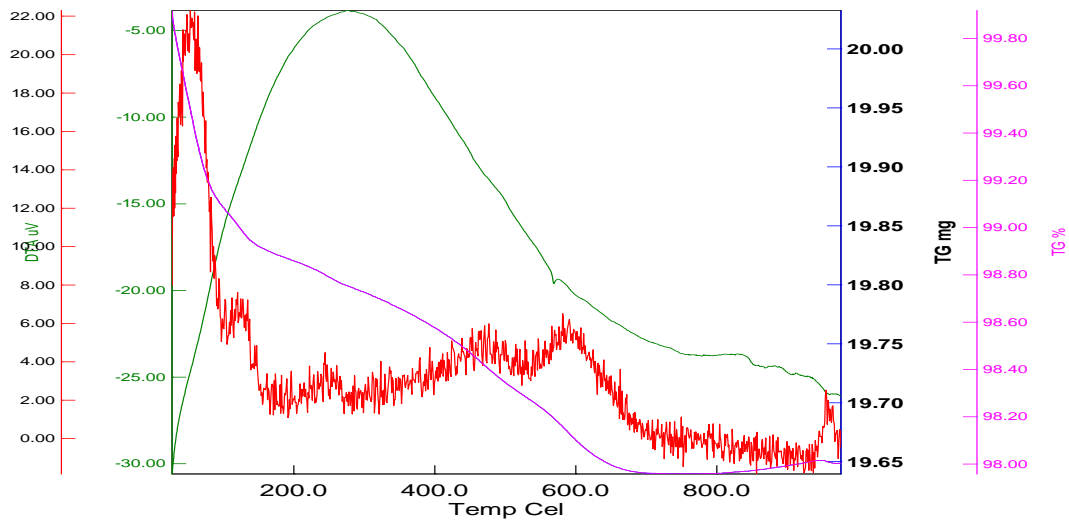


Fig. 5. DTA/TGA of the commercial product of bentonite clay

As a primary method for separation of the existing admixtures from bentonite clay, a wet sieve analysis was applied (Figure 6).

The mass contents of the particle size fractions, are presented in Table 2.



Fig. 6. Various particle size fractions retained on sieves

Table 2

Wet sieve analysis of bentonite clay

Fraction (mm)	Mass content(%)
+0.063 mm	4.54
-0.063 +0.050 mm	6.44
-0.050 +0.040 mm	3.78
-0.040 +0.032 mm	5.10
-0.032 mm	80.14
Σ	100.00

Clay absorbs water and expanding, therefore for comparison purposes, a sieve analysis using alcohol was realized (Figure 7).

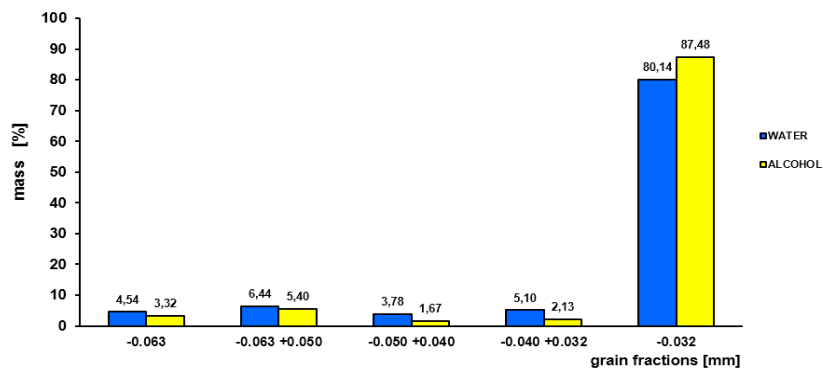


Fig. 7. Comparative histogram of sieve analysis

According to XRD on Figure 8, in the coarsest fraction (+0.063 mm) quartz, sodalite and phlogopite were determined (phlogopite is not determined in the commercial product of bentonite clay, because is below the detection limit).

The admixtures were also concentrated in the other coarse fractions (−0.063 +0.050 mm, −0.050 +0.040 mm and −0.040 +0.032 mm). In the finest fraction (−0.032 mm) from wet sieve analysis the concentration of admixtures is negligible, and contain clay as basic mass (Figure 9).

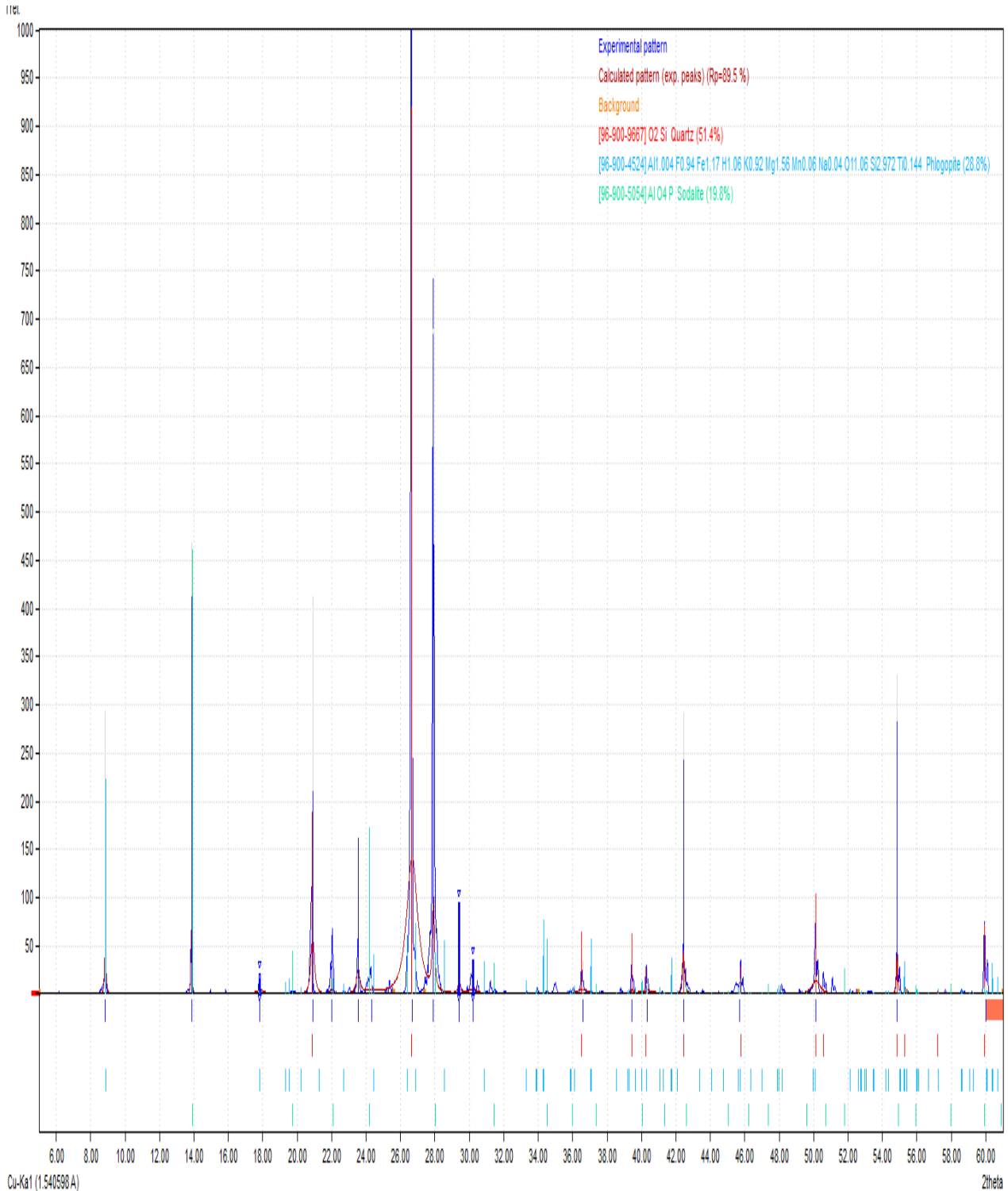


Fig. 8. XRD of the coarsest fraction (+0.063 mm) from wet sieve analysis

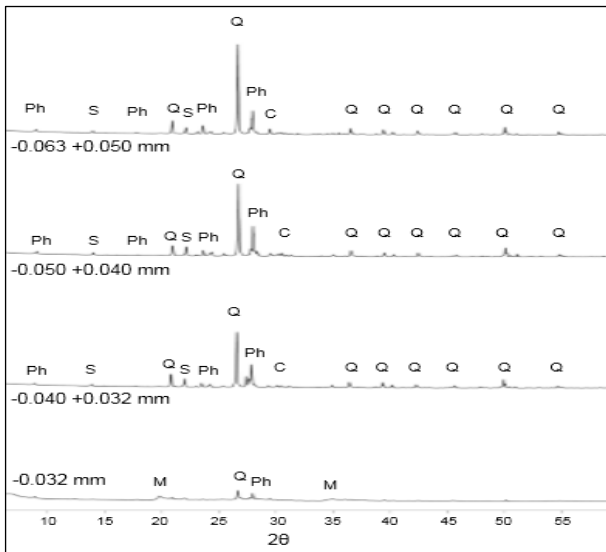


Fig. 9. XRD of various particle size fractions from wet sieve analysis (Q – quartz, S – sodalite, Ph – phlogopite, C – calcite, M – montmorillonite)

Microscopic examinations confirm the X-ray analysis. Coarse fractions from wet sieve analysis have dominant content of transparent clear quartz particles (Figures 10 – 13). Yellow is the particles of phlogopite, turquoise green particles of sodalite and milky white particles of calcite are also present in the coarse fractions. There is minor content of clay aggregates.

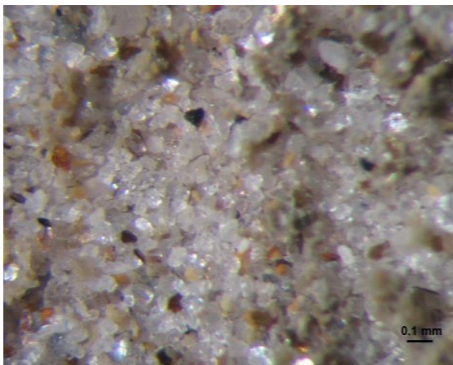


Fig. 10. Fraction +0.063 mm

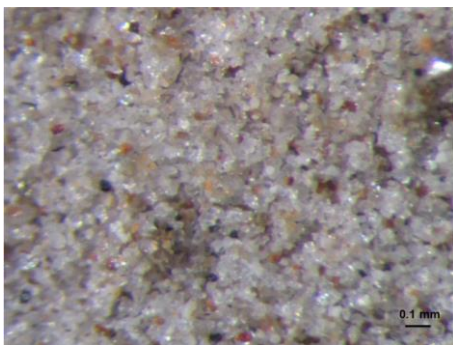


Fig. 11. Fraction -0.063 +0.050 mm

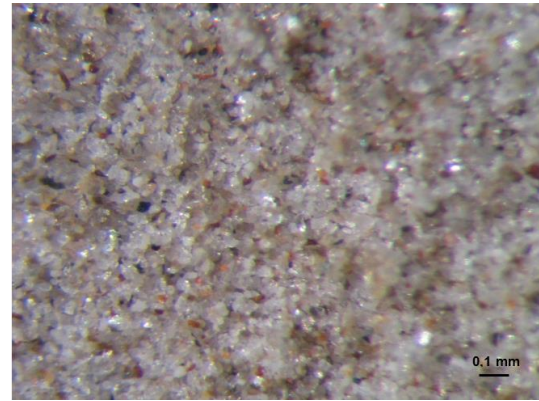


Fig. 12. Fraction -0.050 +0.040 mm

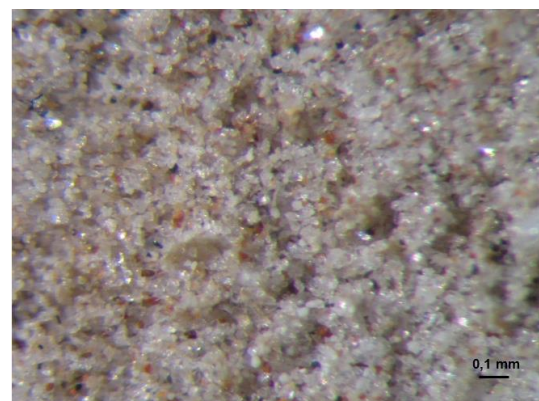


Fig. 13. Fraction -0.040 +0.032 mm

Opposite, the finest fraction (-0.032 mm) have absolute dominant content of clay particles, with a minor quantity of admixtures (Figure 14).

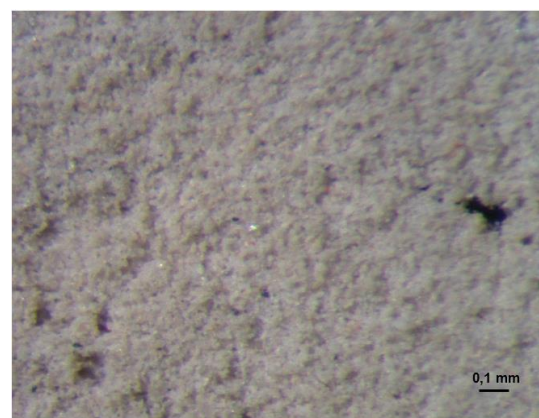
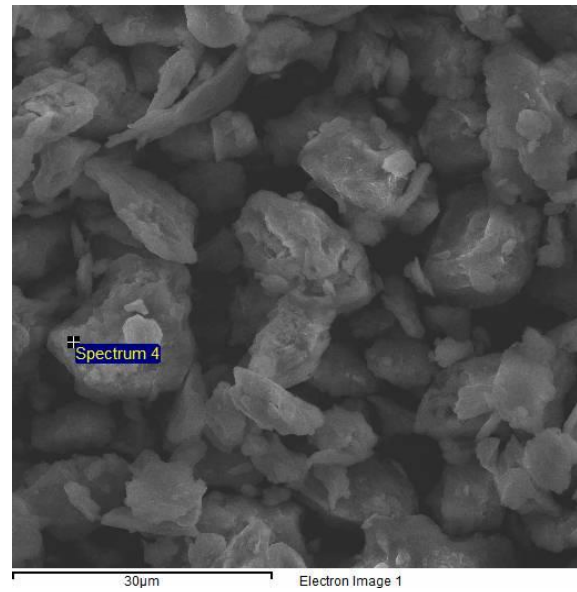
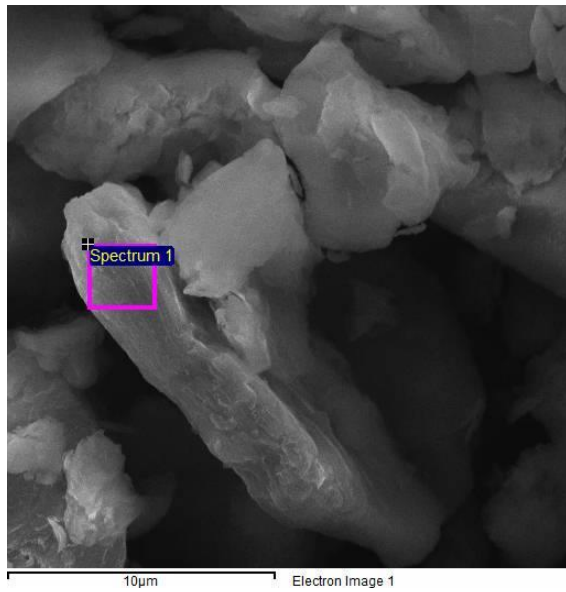


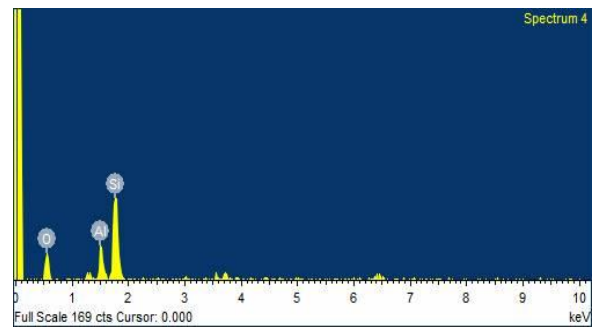
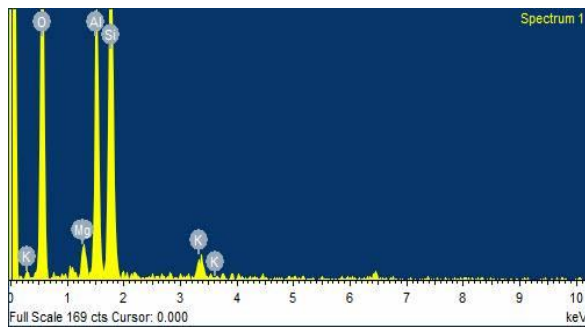
Fig. 14. Fraction -0.032 mm

Scanning electron microscopy was used to observe the particles morphology. Submicronic constituents of the coarse and fine particles are presented on Figure 15.



Element	Weight %	Atomic %
O	60.53	72.72
Mg	1.98	1.57
Al	13.40	9.54
Si	22.44	15.36
K	1.65	0.81
Total	100.00	100.00

Element	Weight %	Atomic %
O	49.02	62.61
Al	10.05	7.61
Si	40.93	29.78
Total	100.00	100.00



(a)

(b)

Fig. 15. SEM-EDS of particle, (a) fine and (b) coarse

Chemical composition of the various particle size fractions from wet sieve analysis was defined by silicate chemical analysis (Table 3). According to the microscopic examinations and XRD there are in significant variations in the coarse fractions from the wet sieve analysis, therefore it is defined average chemical composition of the composite sample from fractions $-0.063 +0.050$ mm, $-0.050 +0.040$ mm and $-0.040 +0.032$ mm. As a consequence of the concentration of the admixtures, the content of SiO_2 is higher in the coarse fractions (64.43% and 56.50%), compared to the fine fraction, which has a higher content of Al_2O_3 (16.41%) due to dominant mass of clay.

Table 3
Chemical composition of various fractions from wet sieve analysis (mass %)

	(+0,063 mm)	(-0,063 +0.032 mm)	(-0.032 mm)
SiO_2	64.43	56.50	51.37
Al_2O_3	9.21	12.20	16.41
Fe_2O_3	5.30	5.43	5.76
CaO	9.21	8.12	7.70
MgO	0.29	0.73	0.65
K_2O	0.90	0.58	0.66
Na_2O	2.19	1.66	0.50
SO_3	0.52	0.54	0.48
l.w	7.28	13.88	15.74
Σ	99.33	99.64	99.27

DTA/TGA curves of the coarsest (+0.063 mm) and the finest (−0.032 mm) fractions from wet sieve analysis are presented on Figures 16 and 17. TGA

curve of the finest fraction has a higher loss of mass, as a consequence of structural water decomposition of bentonite.

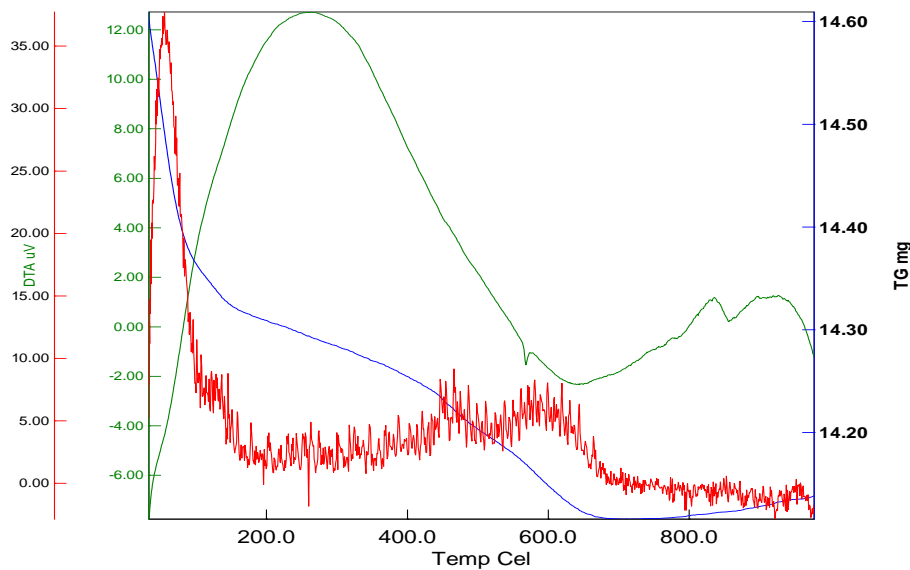


Fig. 16. DTA/TGA of the coarsest fraction (+0.063 mm) from wet sieve analysis

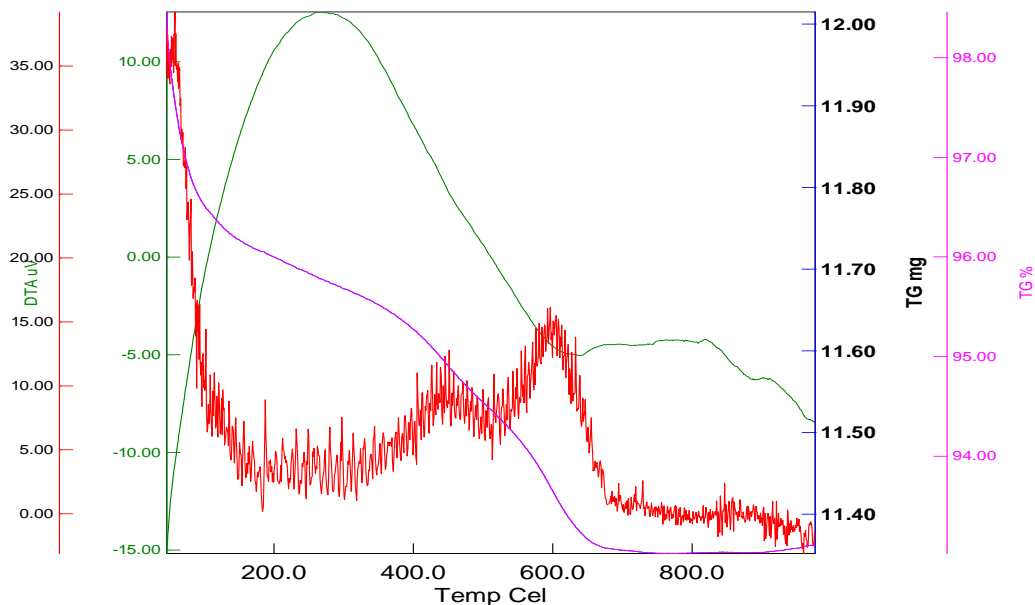


Fig. 17. DTA/TGA of the finest fraction (−0.032 mm) from wet sieve analysis

Elutriation was applied as a secondary method for separation of the existing admixtures from bentonite clay. Elutriation is a method for separation of particles based on their size, shape and density, using a gas or liquid flowing in the opposite direction from the sedimentation of the particles. This method is mainly used for separation of smaller particles.

Separation of the particles in the elutriation is based on the settling velocity of the particles. If settling velocity of the particle is higher than velocity of water which flow in opposite direction, then the particle will be settled and separate into a coarse fraction (Figure 18). Opposite, if settling velocity of the particle is lower than velocity of water which flow in opposite direction, then the particle will be

removed with water in over flow and will be separate into a fine fraction.



Fig. 18. Sedimentation of coarse particles

The prepared suspension of bentonite clay was classified by Elutriation 1 on coarse fraction settled in an Erlenmeyer flask, and fine fraction removed with water in overflow into a tank. Consecutively, the overflow from Elutriation 1 was classified by Elutriation 2 also on coarse and fine fraction (overflow). Therefore bentonite clay from the prepared suspension was classified by elutriation on three fractions: Elutriation 1 coarse fraction (Coarse fraction), Elutriation 2 coarse fraction (Medium fraction) and Elutriation 2 fine fraction (Fine fraction). The mass contents of classified fractions by Elutriation are presented in Table 4.

Table 4

Mass contents of separated fractions by elutriation

Fraction	Mass content (%)
Coarse fraction	2.57
Medium fraction	12.64
Fine fraction	84.79
Σ	100.00

Microscopic examinations (Figures 19–21) indicate that elutriation as a method has a less effective separation than the wet sieve analysis. Appropriate separation of the bentonite clay provides coarse and fine fractions. Opposite, medium fraction from elutriation (which is not insignificant with

12.64 mass %) is heterogeneous and contains particles of admixtures as well as clay aggregates.

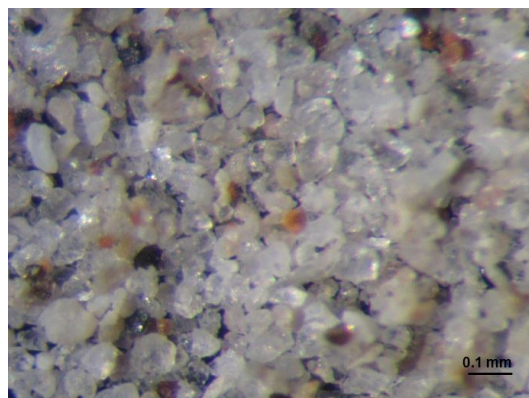


Fig. 19. Coarse fraction of elutriation

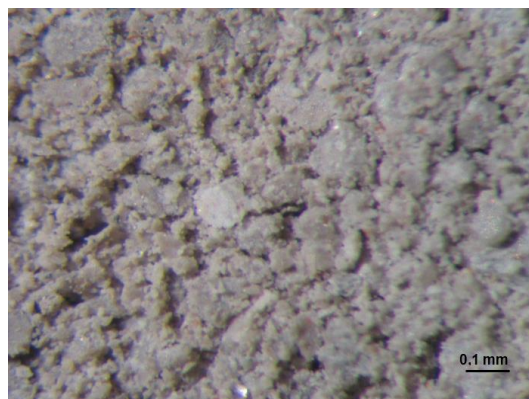


Fig. 20. Medium fraction of elutriation

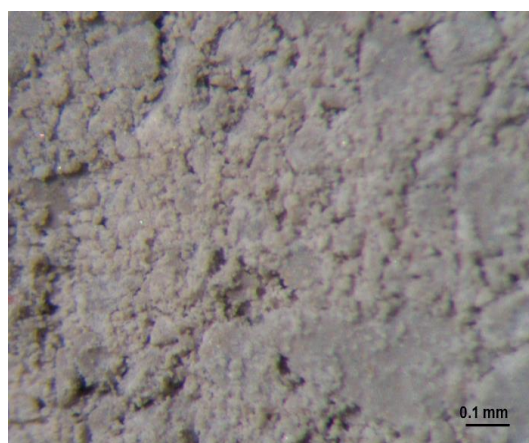


Fig. 21. Fine fraction of elutriation

XRD of various fractions from elutriation (Figure 22) correlate with the microscopic examinations.

The results of silicate chemical analysis of various fractions from elutriation are presented in Table 5.

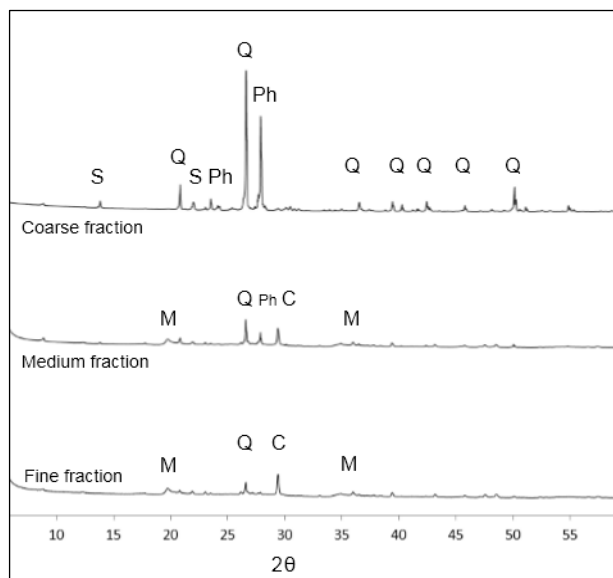


Fig. 22. XRD of various fractions from elutriation (Q – quartz, S – sodalite, Ph – phlogopite, C – calcite, M – montmorillonite)

Table 5

Chemical composition of various fractions from elutriation (mass %)

	Coarse fraction	Medium fraction	Fine fraction
SiO ₂	65.25	56.83	52.69
Al ₂ O ₃	9.03	12.12	15.55
Fe ₂ O ₃	4.64	5.07	5.57
CaO	9.32	8.28	7.85
MgO	0.29	0.73	0.62
K ₂ O	0.90	0.58	0.70
Na ₂ O	2.51	1.85	0.58
SO ₃	0.53	0.52	0.49
l.w	7.36	13.67	15.27
Σ	99.83	99.65	99.32

The settling velocity of particles (v) is defined according to Stokes law:

$$v = \frac{2 \cdot r^2 \cdot (\rho_p - \rho_f) \cdot g}{9 \cdot \mu} \text{ (cm/s),}$$

where:

- v is settling velocity (cm/s),
- r is radius of the particle (cm),
- ρ_p is density of the particle (g/cm³),
- ρ_f is density of the fluid (g/cm³),

g is acceleration of gravity (cm/s²) and
 μ is viscosity of the fluid (g/cm·s).

In Table 6 are presented the calculated settling velocity (mm/s) for particles with various dimensions of the existing minerals in the bentonite clay.

According to the defined flow of water (Elutriation 1 with $Q_1 = 180 \text{ cm}^3/\text{min}$, and Elutriation 2 with $Q_2 = 120 \text{ cm}^3/\text{min}$), and taking into consideration diameter $d = 42 \text{ mm}$, the velocities of water (V) in the tube were calculated:

$$V_1 = 2.166 \text{ mm/s}$$

$$V_2 = 1.444 \text{ mm/s.}$$

Therefore by Elutriation 1, where the velocity of water in the tube $V_1 = 2.166 \text{ mm/s}$, particles of the existing admixtures (quartz, phlogopite, sodalite, calcite) with dimensions larger than $50 \mu\text{m}$ were separated into the coarse fraction (Figure 19.). Then by Elutriation 2, where the velocity of water $V_2 = 1.444 \text{ mm/s}$, the particles of the existing admixtures with dimensions of $40\text{--}50 \mu\text{m}$, as well as the clay aggregates were separated into the medium fraction (Figure 20). Consecutively into a fine fraction from elutriation, removed in overflow, particles of clay with dimension smaller than $40 \mu\text{m}$ were separated (Figure 21).

The settling velocity primarily depend of specific mass (density) and size (dimension) of the particle. Particles of various minerals of a wide range of dimensions can be separated into specific fractions (as the medium fraction of elutriation, Figure 20), reducing the separation efficiency of elutriation. Aggregation of the clay particles (Figure 23) resulting with certain deviations in the settling velocity, also has a negative impact on the separation efficiency of the elutriation.

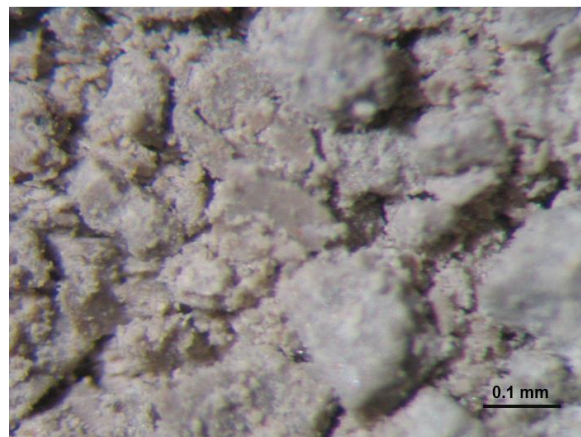


Fig. 23. Aggregated clay particles

Table 6

Settling velocity (mm/s) of particles for various minerals

Particle size (μm)	Settling velocity (mm/s)				
	Montmorillonite	Quartz	Phlogopite	Calcite	Sodalite
2	0.003	0.004	0.004	0.004	0.003
4	0.012	0.014	0.016	0.015	0.011
6	0.026	0.032	0.035	0.033	0.026
8	0.047	0.058	0.063	0.059	0.045
10	0.074	0.090	0.098	0.093	0.071
12	0.106	0.129	0.141	0.133	0.102
14	0.144	0.176	0.192	0.182	0.139
16	0.188	0.230	0.251	0.237	0.181
18	0.238	0.291	0.318	0.300	0.230
20	0.294	0.360	0.392	0.371	0.283
22	0.356	0.435	0.475	0.448	0.343
24	0.424	0.518	0.565	0.534	0.408
26	0.497	0.608	0.663	0.626	0.479
28	0.577	0.705	0.769	0.726	0.555
30	0.662	0.809	0.883	0.834	0.638
32	0.753	0.921	1.005	0.949	0.726
34	0.851	1.040	1.134	1.071	0.819
36	0.954	1.165	1.271	1.201	0.918
38	1.062	1.299	1.417	1.338	1.023
40	1.177	1.439	1.570	1.482	1.134
42	1.298	1.586	1.730	1.634	1.250
44	1.424	1.741	1.899	1.794	1.372
46	1.557	1.903	2.076	1.960	1.499
48	1.695	2.072	2.260	2.135	1.632
50	1.839	2.248	2.453	2.316	1.771
52	1.989	2.432	2.653	2.505	1.916
54	2.145	2.622	2.861	2.702	2.066
56	2.307	2.820	3.076	2.906	2.222
58	2.475	3.025	3.300	3.117	2.383
60	2.649	3.237	3.532	3.335	2.551
62	2.828	3.457	3.771	3.561	2.723
64	3.014	3.683	4.018	3.795	2.902
66	3.205	3.917	4.273	4.036	3.086
68	3.402	4.158	4.536	4.284	3.276
70	3.605	4.406	4.807	4.540	3.472
72	3.814	4.662	5.086	4.803	3.673
74	4.029	4.924	5.372	5.074	3.880
76	4.250	5.194	5.666	5.351	4.092
78	4.476	5.471	5.968	5.637	4.311
80	4.709	5.755	6.278	5.930	4.534

CONCLUSION

One of the commercial products from Bentomak, bentonite clay (–0.063 mm) contains montmorillonite as basic mass, and quartz, calcite, sodalite, phlogopite as admixtures. Wet sieve analysis and elutriation were applied as methods for separation of admixtures.

A higher degree of separation provides a wet sieve analysis. Mineralogical-petrographic examinations confirm that the existing admixtures are separated into coarse fractions. The coarse fractions dominantly contain admixtures and minor quantities of clay aggregates. Additionally the clay was concentrated in the finest fraction (–0.032 mm). Therefore

the finest fraction separated from admixtures has wide a range of high profitable practical applications.

Elutriation as a method has a lower degree of separation. The medium fraction from elutriation has a heterogeneous composition of admixtures and clay. The main reason for incompleting separation in the elutriation process is the aggregation and agglomeration of the fine clay particles on the surface of the coarse particles of the admixtures. The formed aggregates and agglomerates have a certain variability in the velocity of sedimentation, which resulted with sedimentation in various fractions.

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Резиме

КАРАКТЕРИЗАЦИЈА НА ГРАНУЛОМЕТРИСКИТЕ ФРАКЦИИ ОД БЕНТОНИТСКАТА ГЛИНА ОД ГИНОВЦИ, РЕПУБЛИКА СЕВЕРНА МАКЕДОНИЈА

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Клучни зборови: бентонитска глина; примеси; гранулометриски фракции; мокра гранулометриска анализа; елутрација.

Испитуваната бентонитска глина како основна маса содржи монтморилонит. Како примеси се јавуваат кварц, илити и други минерали. Примесите се нехомогено дистрибуирани во различните гранулометриски фракции. Целта на ова истражување е концентрација и сепарација на примесите од бентонитската глина (-0.063 mm), комерцијален производ на Бентомак. Како методи за сепарација беа при-

менети мокра гранулометриска анализа и елутријација. За да се дефинира ефикасноста на методите, беа направени комплексни испитувања за карактеризација на фракциите. Симултаниот преглед на резултатите од рентгенската, микроскопската и хемиската анализа покажа дека во најфината фракција (-0.032 mm) концентрацијата на примесите е помала за 80% во однос на другите фракции.

