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NEW FINDINGS FOR DIATOMITE (DIATOMACEOUS EARTH) BETWEEN THE VILLAGES OF MANASTIR AND BEŠIŠTE (MARIOVO)

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A b s t r a c t: Diatomaceous earth as porous, fine granular siliceous sedimentary rock, due to their specific physical-chemical properties, often attracted attention to the study of professional and scientific aspect. For diatomaceous earth from the Manastir–Bešište deposit in geological literature very little has been written, especially its mineralogical and chemical composition. In the paper will be present the latest research and results obtained on samples taken from the surface of the ground from the Manastir–Bešište deposit. The test is made in the laboratories of the Faculty of Natural and Technical Sciences with X-ray diffraction and skanning electron microscope. From the X-ray powder diagram are seen most widespread mineral phases quartz, illite, muscovite, pyroxene, and amorphous phase.

Key words: diatomaceous earth; Manastir; Bešište; skanning electron microscope; chemical composition; ore quality

INTRODUCTION

The deposit of diatomaceous earth between Manastir and Bešište is located 35 km southeast of Prilep and to get there is a relatively good car road that continues on to the village of Vitolište (Fig.1). The terrain where diatomite of Manastir–Bešište is located represents a plateau which gradually rises from Pulic on southeast toward Bešište, which is overlaped with calcareous tuff–limestone slab. On some places is eroded, so today there are remains and preserved only individual patches of calcareous tuff. Such is the case in the space between the Pulic and Premka. The terrain, seen from afar, has an amphitheater view, because it consists of various lithological members which are not equally susceptible to erosion. It can especially be seen between Pulic and Premka. The whole terrain is criss-crossed by several smaller rivers and temporary streams, most of which flow directly into the river of Crna Reka, which flows nearby.

The first data of the Manastir–Bešište diatomite are found in the work of Tuchan (1935). Later more data provide Maric (1938) and Tajder (1939) in petrographic studies from the Mariovo–Tikveš region and suggest that in lake sediments near the village of Manastir is located diatomite. The first mining investigative works in diatomite (diatomaceous earth) of Manastir–Bešište dating from 1951. From mineralogical-petrographic and chemical aspect, the deposit of diatomite from Monastery–Bešište is studied by Laskarev (1950), Petronijević (1952), Karamata (1956), Pantić (1956), Kitanov (1956), Stamboliev (1954), Jenko and Gjuzelkovski (1953), Gjuzelkovski (1955).

Fig. 1. Tectonic map of the Republic of Macedonia (Arsovski, 1997 [1])
I – Vardar zone; II – Pelagonian massif; III – Western Macedonian zone; IV – Krasta zone; V – Serbo-Macedonian massif, VI – Kraištide zone

In order for regional study of the deposit was carried out geological mapping, mining investigation works, deep drilling investigation and geoelectrical test measurements. Measurements were performed by the Department of Geophysics of the Institute of Geological Research of Serbia – Belgrade. Geoelectrical measurements were performed around borehole 3, as well as in the mine of Pulic and around Manastir. More recent geological, mineralogical-petrographic and chemical studies of the immediate vicinity of the deposit of diatomite from Manastir–Bešište are studies by Spasovski et al. [14–19].

METHODOLOGY

The location of Manastir–Bešište is explored using field and laboratory techniques. Field study provided an insight into the terrain, familiarization with its geological and structural-tectonic features as well as the collection of representative samples from the diatomaceous earth and definition of their chemical-mineralogical composition. Mineralogical composition of rocks was determined by XRD. Instrument XRD Shimadzu 6100 was used. It was used copper radiation CuKα = 1.54178 Ǻ, the voltage of the generator 40 kV, and the current was 30 mA, θ–2θ range (deg) 10 000–80 000, speed (deg/min) 2.00, pich (deg) 0.100, present time (sec) 0.12.

Chemical compositions of main mineral phases were determined by scanning electron microscopy model VEGA3 LM and EDS detector connected with INCA 250 EDS software.

GEOLOGICAL REVIEW

The entire terrain where occurs diatomite (diatomaceous earth) has almost the same geological composition. Basis on the ground is composed of old Paleozoic orthogneisses and granite-gneisses, intruded with pegmatite, aplite and quartz wires which composed the base of all other sedimentary rocks (Figs. 2, 3, 4, 5).

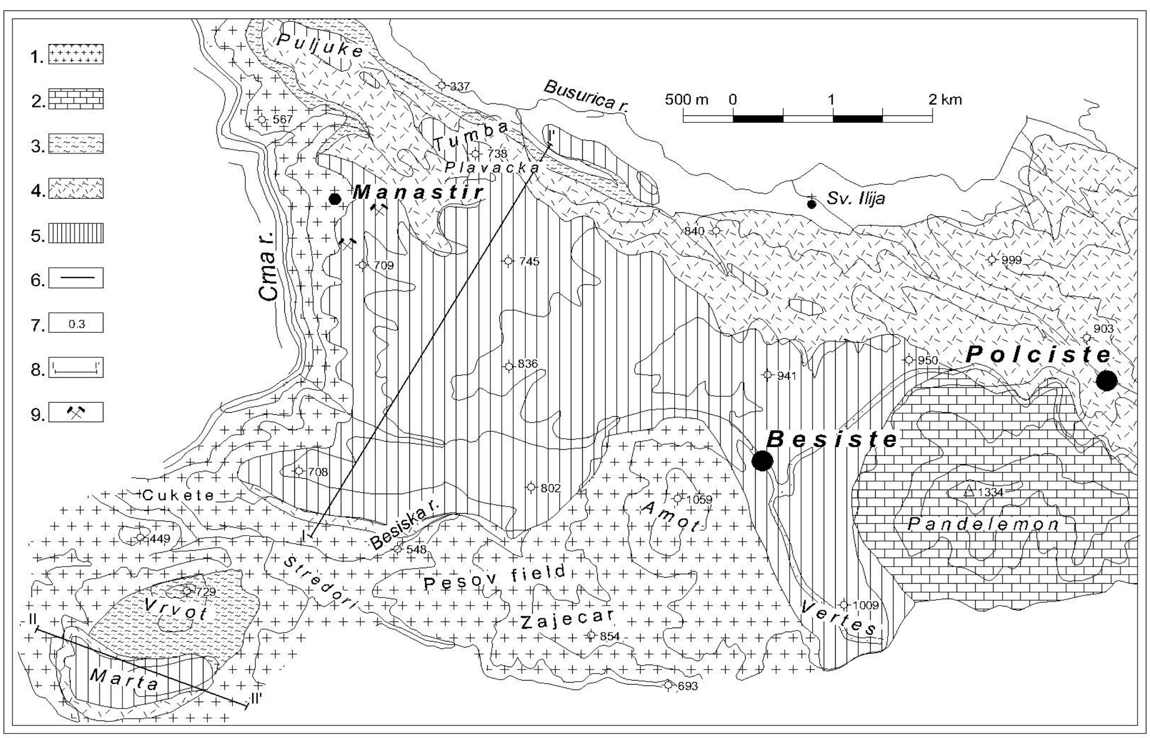

Fig. 2. Geological map of the deposit of diatomite (diatomaceous earth) Manastir–Bešište (Gjuzelkovski, 1955 [13])
1. Orthogneiss, gneiss-granite; 2. Marble; 3. Sands, gravels, cleys and conglomerates; 4. Andesite tuff; 5. Calcareous tuff; 6. Diatomite (diatomaceous earth); 7. Borehole; 8. Profile line; 9. Mine.

Through gneisses which represented expressive paleorelief transgressive and discordant lie Neogene sediments, presented with various sediments, such as: conglomerates, sandstones, sands, gravels, clays, diatomaceous earth and calcareous limestone. In these sediments are interstratified andenzite tuffs, fairly well stratified, suggesting that were created during the sedimentary phase. Superpozitional order of Neogene strata is almost synchronic for the entire field, and going from northwest to southeast, the number of lithologic members and the thickness of diatomaceous earth increase.

Frequent vertical lithological changes clearly point to the numerous variations of the Neogene lake level in which prevailed psamite rocks. These are determined by epirogenetic movements, which in the last stages are accompanied with volcanism, andenzite extrusive (Kravicki Kamen etc.). Individual members of the layers occur rhythmically.

The lowest member of Neogene are sands mixed with gravel and weakly bonded sandstones. Than is calcareous tuff, with thickness about 4–5 meters near Manastir and Premka, and on Pulik is lost. After it lie conglomerates composed of well rounded pieces of marble (from St. Pantelejmon), quartz and gneiss. Above them lies andezite tuff that creates bottom of the diatomite. As roof apear clay (in Premka and Pulik), sand and sandy clay (in Manastir) and above is calcareous tuff. With this member on Pulik is finished Neogene series, and on Premka and Manastir are lined up calcareous limestone, tuff and tuffose sand.

As cover of the whole field appears calcareous tuff, which earlier represents an entire plate, and later eroded so that in places has preserved certain patches of the former single plate. A similar lithological composition have Neogene sediments in Zović, also overlaped with calcareous limestone.

Calcareous limestone in the lower sections is more porous, and in the upper parts is more compact and looks like real limestone. It serves as a protective cover of diatomite, and on the places where it is eroded, and diatomite is eroded too. Such is the case on the terrain between Pulik and Premka where is preserved a small calcareous limestone cap under which is diatomite, and in the parts where calcareous tuff is not present, the ore is eroded and the bottom of the diatomaceous earth is uncovered – andesite tuff.

Over Manastir, with drilling is drilled the whole Neogene series to the base that is composed of gneisses, above them are sands and gravels, than calcareous tuffs. Than, comes a layer of clay which, in direction of adit no. 5, pinch out. Above the clay is andesite tuff with which is connected diatomite, and after it is layer of yellow limonitized sand, tuff sand and sandy clay above which is calcareous tuff.

Neogene sediments in which is found diatomite (diatomaceous earth) are of Upper Pliocene age, and some of them belong to Middle Pliocene

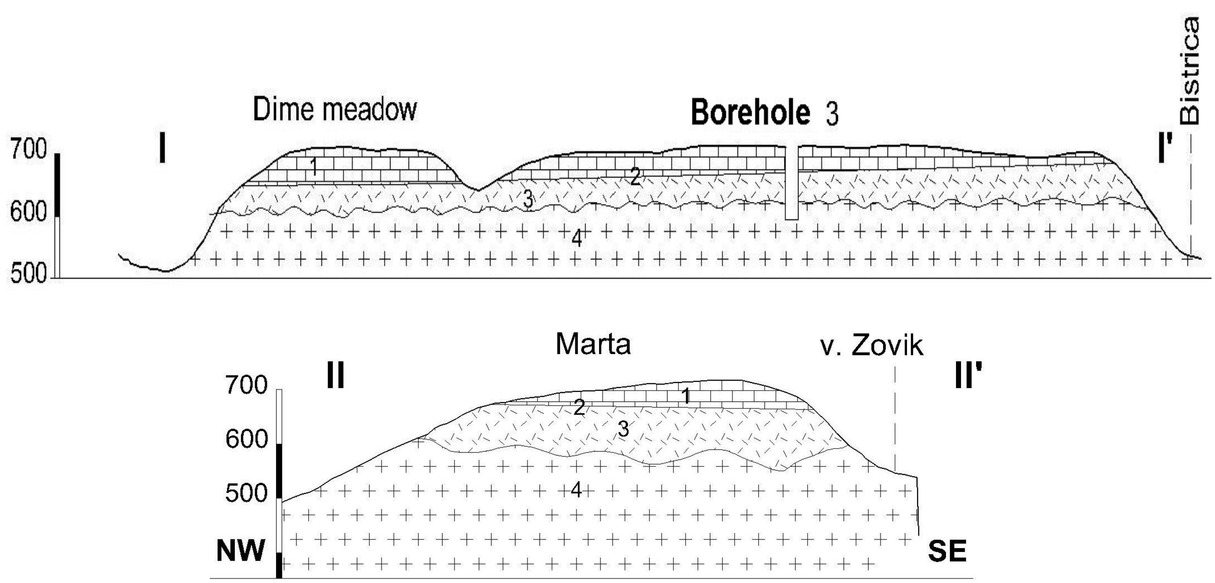

Fig. 3. Characteristic geological profiles
1. Marble; 2. Diatomite (diatomaceous earth); 3. Andesite tuff; 4. Orthogneiss, gneiss-granite

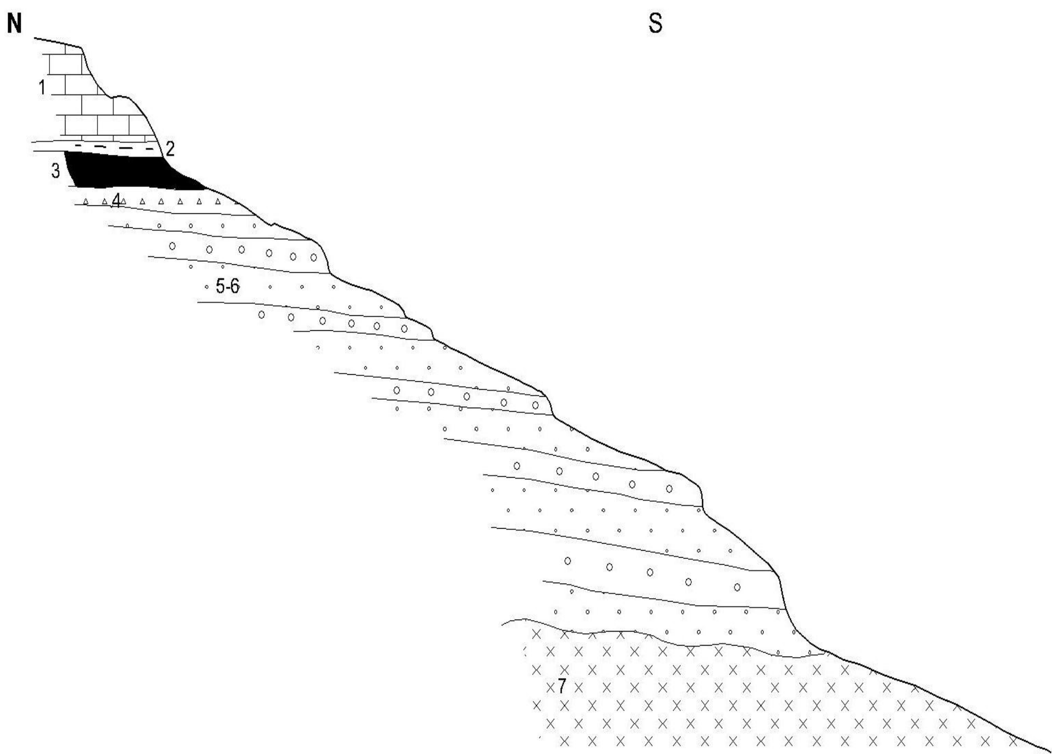

Fig 4. Geological profile Premka Pulik
1. Calcareous tuff; 2. Clay; 3. Diatomite; 4. Andesite tuff; 5. Sands and clays; 6. Sands, gravels, clays and conglomerates; 7. Orthogneiss, gneiss-granite

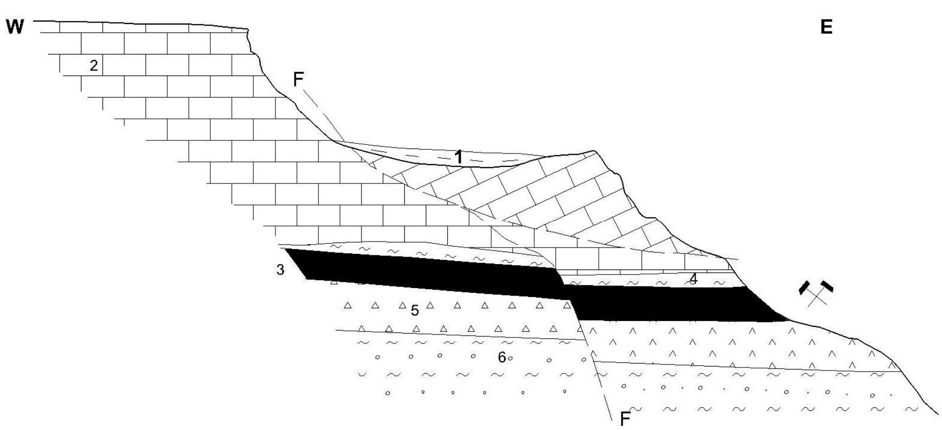

Fig. 5. Part of faultetd landslide Pulik
1. Humus, clay; 2. Calcareous tuff; 3. Diatomite; 4 Clay; 5. Andesite tuff; 6. Sands and clays; F. Faulted landslide

In diatomaceous earth macroscopically are distinguishable more species (Fig. 6): a) Gray ashy clay diatomaceous earth, with plated leaf habitus with limonite nodules and layers of 1 – 3 cm, the thickness of the layer of this kind varies from 0.7 to 1.4 m. b) Whitish pure diatomaceous earth shelly infraction with insignificant limonite nodules. The thickness of this is from 0.5 to 1.23 m. c) Whitish, leaf diatomaceous earth with limonite nodules and thin clay layers. This layer is the main representative of the deposit of diatomaceous earth. Lower border towards yellowish (d) or floor or diatomaceous earth, is vague and gradual. According roof (b) layer transition is somewhere gradual, and in places is sharp with thin layers of black clay of 1 cm. d) Yellowish diatomaceous earth differs from the above described, only with higher iron content. Yellow diatomaceous earth is major impurities of iron, and leaf like (c) and (d).

Apart from these species, in Manastir there is brown and black diatomaceous earth containing clay and organic matter, making it specifically more difficult from other species. There is a gradual transition from white diatomaceous earth in yellowish and brown, and this in black, which thickness increases considerably, reaching up to 12 meters. Mentioned types of diatomaceous earth in terms of moisture are quite variables. The appearance of ore in the gallery is gray, at sun becomes dried and become white ashy or yellowish, which to some extent depend on the impurities. Mentioned species can be removed and obtained in larger blocks.

Fig 6. The most common types of diatomaceous earth
a) Grey ashy diatomaceous earth with plated habitus. b) White diatomaceous earth with shelly defraction.
c) White sheeted diatomaceous earth. d) Yellow diatomaceous earth

ORE QUALITY AND CHEMICAL ANALYSES

White diatomaceous earth is clean and very reminiscent of chalk for writing. Black diatomaceous earth has clay, which is necessary for some products. The enclosed Table 1 clearly shows the chemical composition of the deposit.

Based on the made chemical analyses, the diatomaceous earth contains: SiO_2 78.05 – 88.35%, Al_2O_3 4.61 – 10.376%, CaO 0.62 – 0.81%, MgO 0.15 – 0.40% and Fe_2O_3 1.21 – 4.05% (Table 1). The damages of the diatomaceous earth samples directly affect of reducing the thermal conductivity of the mineral raw material, although diatomaceous earth contains a high percentage of SiO_2.

T a b l e 1

Chemical composition of diatomite (diatomaceous earth) from village of Manastir

Component	Pulik gallery		Premka–Belilo gallery	
	3	1	5	5
SiO_2	82.24	88.35	78.05	82.55
Al_2O_3	6.51	4.61	10.37	5.96
Fe_2O_3	3.73	1.21	4.05	4.05
CaO	0.81	0.68	0.62	0.72
MgO	0.34	0.26	0.40	0.15
Na_2O	0.28	Undetermined	0.19	0.13
K_2O	0.20	Undetermined	0.08	0.03
Loss on ignition	6.17	5.14	6.29	6.45

In addition to chemical examinations, mineral composition of the rock is determined with XRD method. Diffractogram is presented on Fig. 7

Based on the peak position and according the angle 2Θ is made identification of the minerals, and based on the peak intensities is concluded their relative quantity in the sample.

From the x-ray diagram shown on Fig. 7 can be seen that about amorphous phase most probably it is organic material, with little presence of quartz, illite, opal-A, pyroxene and muscovite.

The most intense registered maxima in the studied powder diagram were compared with the corresponding maxima in the diagrams of quartz-JCPDS 00 001 0649, illite-JCPDS 00 058 2016, opal-A-JCPDS 00 038 0448, muscovite-JCPDS 00 002 0058 and pyroxene-JCPDS 01 076 3329.

To determine the chemical composition of diatomaceous earth with skaning electron microscope separate representative samples were taken from the surface of the terrain. For testing a total of seven samples were taken, the results of the tests are shown in Tables 2, 3, 4, 5. Position of the analyzed samples is presented on Figs. 8, 9, 12, 13, 16, 17 and 20, and EDC spectrums are shown on Figs. 10, 11, 14, 15, 18, 19, 21.

Based on the obtained data for chemical composition given in Table 2, the samples 1 and 2 are defined as opal.

Based on the obtained data for chemical composition given in Table 3, the samples 3 and 4 are defined as illite.

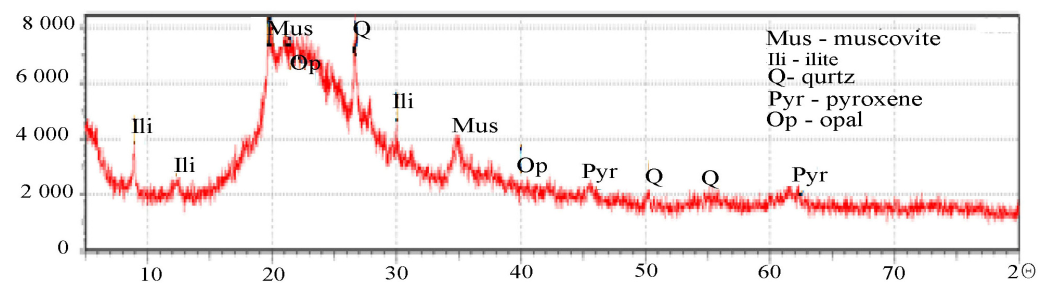

Fig 7. X-ray diffractogram of diatomaceous earth from the Manastir–Beišište deposit

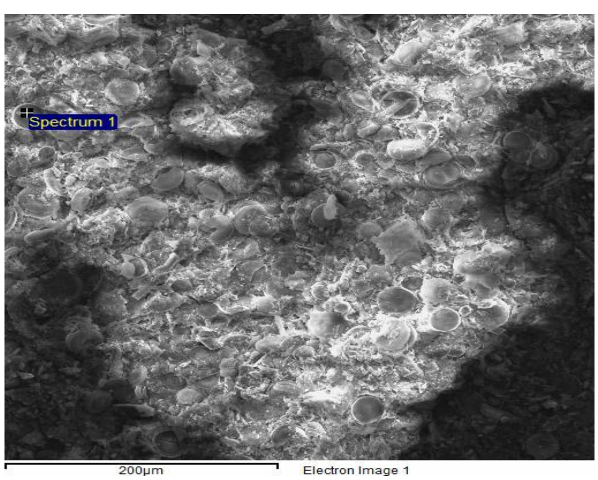

Fig. 8. SEM picture of diatomaceous earth, sample 1

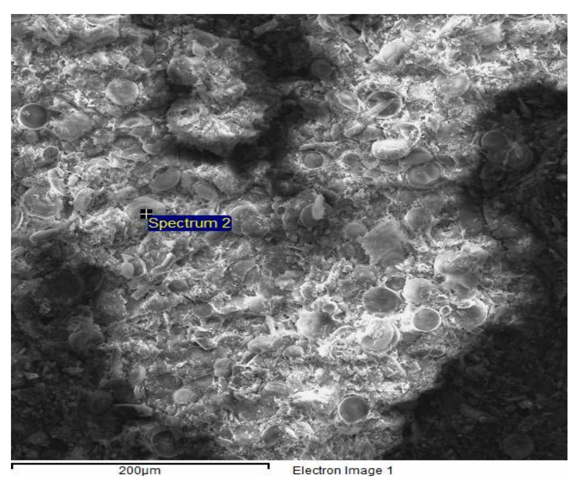

Fig. 9. SEM picture of diatomaceous earth, sample 2

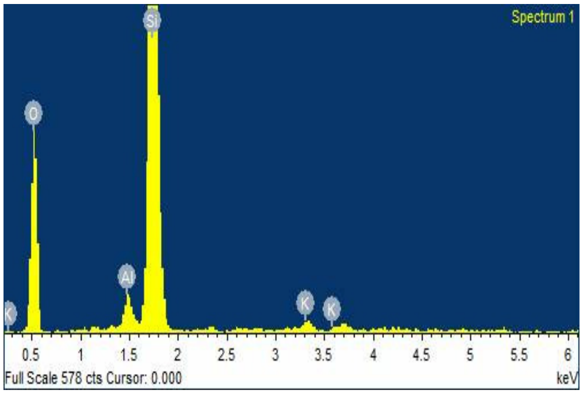

Fig. 10. EDC spectrum of diatomaceous earth, sample 1

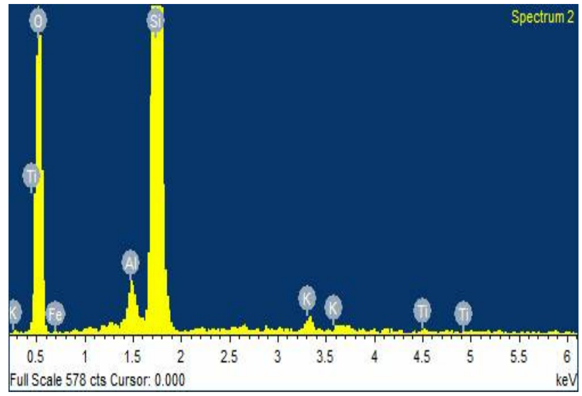

Fig. 11. EDC spectrum of diatomaceous earth, sample 2

T a b l e 2

Chemical composition of diatomaceous earth Pulik (%)

Element	Sample 1		Element	Sample 2	
	Weight	Atomic		Weight	Atomic
O K	52.81	66.36	O K	56.94	70.21
Al K	1.65	1.23	Al K	1.47	1.07
Si K	44.68	31.97	Si K	39.71	27.90
K K	0.86	0.44	K K	0.89	0.45
			Ti K	0.32	0.13
			Fe K	0.68	0.24
Total	100.00		Total	100.00	

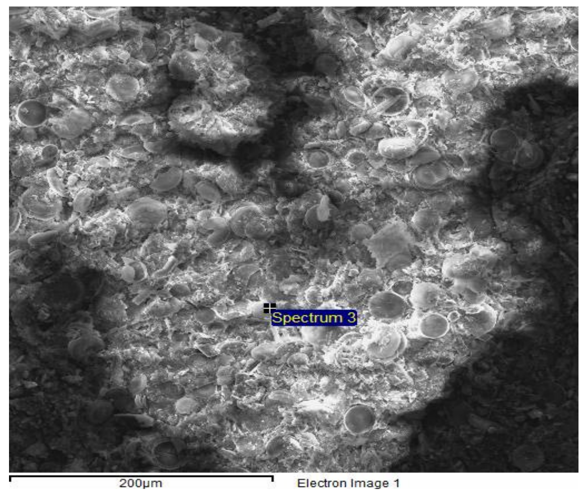

Fig. 12. SEM picture of diatomaceous earth, sample 3

T a b l e 3

Chemical composition of diatomaceous earth Pulik (%)

Element	Sample 3		Element	Sample 4	
	Weight	Atomic		Weight	Atomic
O K	58.16	71.53	O K	53.82	68.83
Na K	0.69	0.59	Al K	10.63	8.06
Mg K	0.67	0.54	Si K	24.74	18.02
Al K	4.75	3.46	K K	7.12	3.73
Si K	31.53	22.09	Fe K	3.69	1.35
K K	1.35	0.68	O K	53.82	68.83
Ca K	0.66	0.32			
Ti K	0.20	0.08			
Fe K	2.00	0.70			
Total	100.00		Total	100.00	

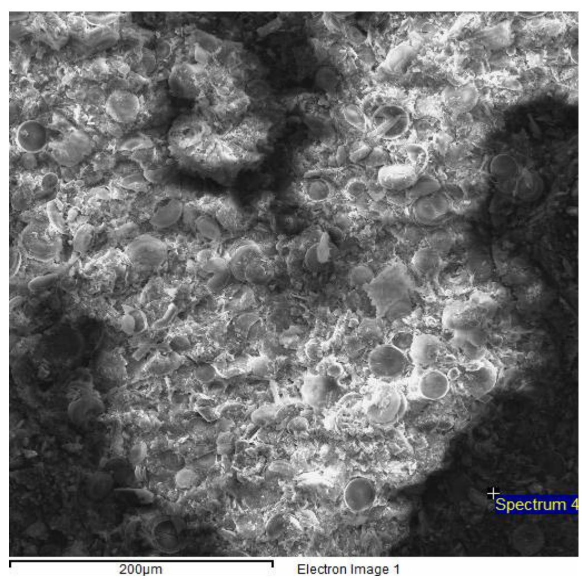

Fig. 13. SEM picture of diatomaceous earth, sample 4

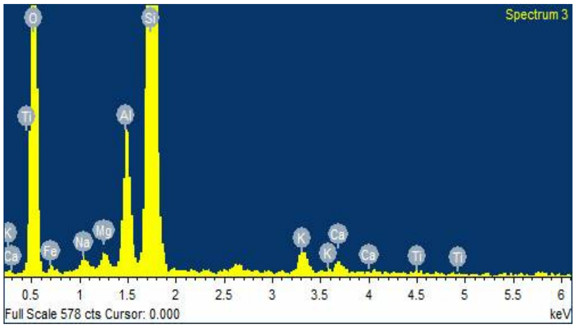

Fig. 14. EDC spectrum of diatomaceous earth, sample 3

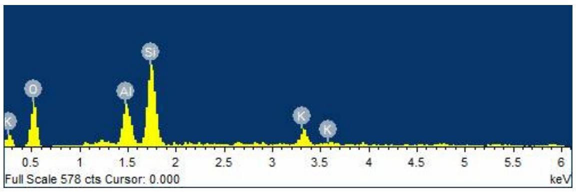

Fig. 15. EDC spectrum of diatomaceous earth, sample 4

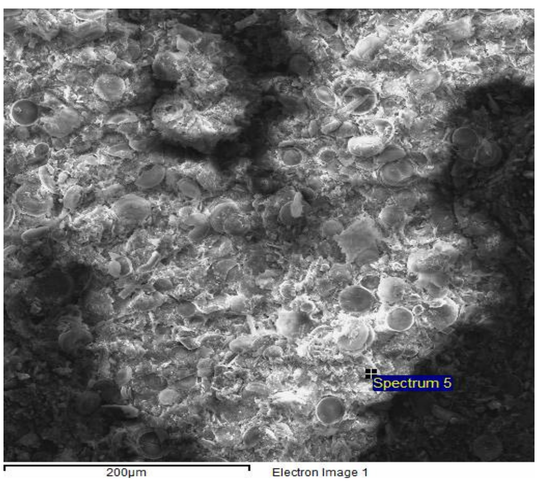

Fig. 16. SEM picture of diatomaceous earth, sample 5

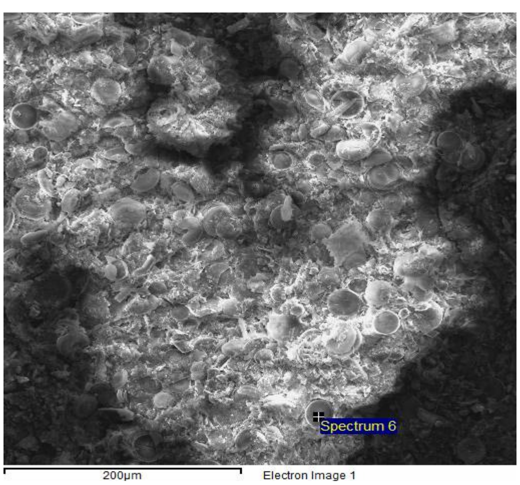

Fig. 17. SEM picture of diatomaceous earth, sample 6

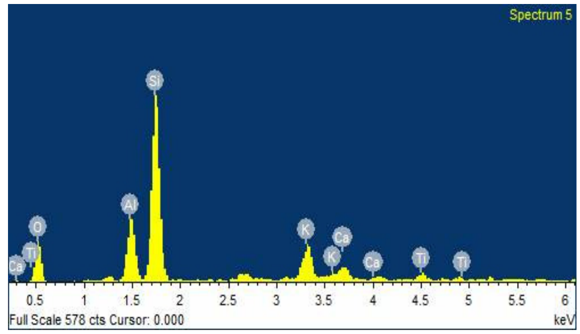

Fig. 18. EDC spectrum of diatomaceous earth, sample 5

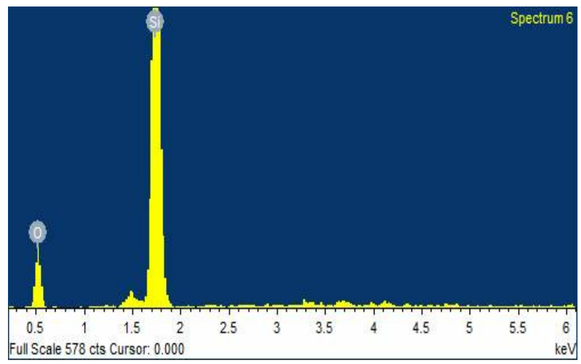

Fig. 19. EDC spectrum of diatomaceous earth, sample 6

Based on the obtained data for chemical composition given in Table 4 the sample 5 is defined such as pyroxene, while the sample 6 is quartz.

Based on the chemical composition given in Table 5 the sample 7 is defined such as pyroxene.

From microphotos (Figs. 8, 9, 12, 13, 16, 17, 20) is seen that diatomaceous earth represents multi phase material composed of porous, crystal and amorphous phases.

Diatomaceous earth from the Manastir–Bešište deposit contains significant contents of silicium, aluminium, calcium, potassium, sodium and iron (Tables 2, 3, 4, 5). With lower contents occur titanium and chlorine.

According the mineralogical and chemical examinations, diatomaceous earth from the Manastir–Bešište deposit is of good quality and as a mineral resource may find application in: porcelain industry, food, chemical and pharmaceutical industries as a means of filtration as waterproof pads, etc.

T a b l e 4

Chemical composition of diatomaceous earth Pulik (%)

Element	Sample 5		Element	Sample 6	
	Weight	Atomic		Weight	Atomic
O K	37.07	54.50	O K	40.11	54.04
Al K	9.07	7.91	Si K	59.89	45.96
Si K	30.59	25.62			
K K	8.24	4.96			
Ca K	3.10	1.82			
Ti K	2.41	1.19			
Fe K	9.53	4.01			
Total	100.00		Total	100.00	

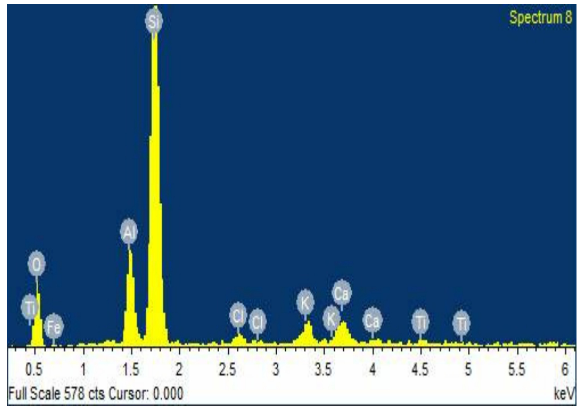

Fig. 21. EDC spectrum of diatomaceous earth, sample 7

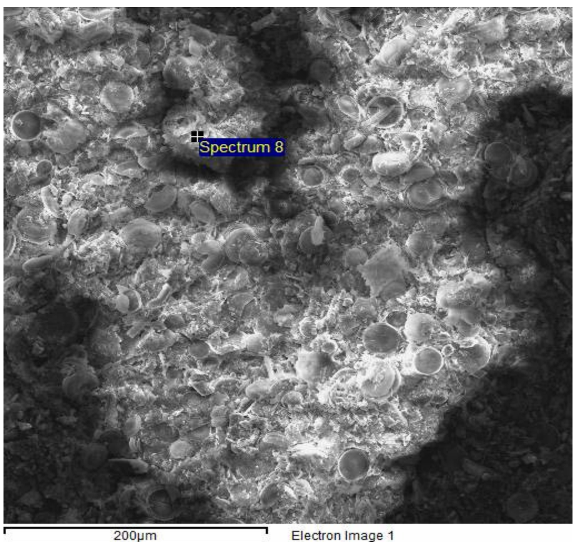

Fig. 20. SEM picture of diatomaceous earth, sample 7

T a b l e 5

Chemical composition of diatomaceous earth Pulik (%)

Element	Sample 7	
	Weight	Atomic
O K	35.72	51.70
Al K	8.35	7.16
Si K	39.94	32.93
Cl K	1.45	0.95
K K	3.12	1.85
Ca K	3.74	2.16
Ti K	0.80	0.39
Fe K	6.88	2.85
Totals	1 00	

GENESIS AND DESCRIPTION OF THE DEPOSIT

According to the position of the layers in the superpositional order of the Neogene series follows that diatomite is connected to a single horizon, above the first layer of tuff, going from floor layer to roof layer. It is likely that the deposit of the diatomite been affected by the volcanic phase, with their tuffs, increased the percentage of free silica acid in aqueous solution, and thus are given favorable conditions for the development of organisms with silica skeleton – diatomes. As is known, these are plankton organisms which multiply rapidly and form large masses which provided material for the creation of diatomite (diatomaceous earth). Important role in their proliferation, of the salts have nitrogen and phosphorus.

According to the method of the appearance and given the existence of only a single layer of diatomite (diatomaceous earth), it is undoubtedly that it is word about lake sediments about which is typical such a superposition of layers.

Geochemical examinations (Rankama and Sakama, 1952) for the conditions of sedimentation, especially for pH value, Karamata (1956) for the similar diatomite from the village of Gradec (Kriva Palanka) concluded that must be basic ground near the lake environment in which is deposited diatomite, i.e. terrain with increased pH value. Such an environment facilitates dissolution of the silicon and water enriched by silicon, which acts useful for the development of the diatomite, to such an extent, the skeleton of the extinct diatomes gave substance to create the entire deposit. That it is possible in a relatively short period of time, speaks in favor and the fact of the rapid multiplication of the diatomite.

To get to the enrichment of the silica in the water and higher pH value depends on the ground of the lake. In this case with most similar deposits in the former Yugoslavia, diatomaceous earth is tied to andenzite tuffs, in the sediments ground or the nearby volcanic phase, which acted immediately before deposition of diatomaceous earth, and partly at the same time with it.

The deposit is of sedimentary origin and has the shape of a simple plate. It is almost horizontal, which thickness increases going from the former edge of the Neogene lake basin to the middle of it. As a protective cover of the ore of all places is calcareous limestone. It toward the ore is in logical connection, i. e. where there is calcareous tuff, appears ore. If it is stripped of its protective cover, there is eroded.

The thickness of ore layer and the extent of the excavation vary in the other localities.

In Pulik (the most northwest location) according to the current level of investigation can be seen all mentioned types of ore, described earlier, except black. The maximum thickness of the ore for all species in this locality is 5.10 m, and the mean value is 3.80 m.

In locallity Premka maximum thickness of the ore is 3.75 m and the average is 2.90 m. To the east and south-east diatomaceous soil contains more clay, and than completely fails in pure clay.

Once, these two localities in terms of ore made up a whole, but due to erosion of the big parties of sediments between them to andesite tuff now ape divided separately and preserved as a relic is just a small patch of the diatomaceous earth.

In the Manastir locality, which is located above the village, beside the other types, appears brown and black diatomaceous earth which color comes from the presence of different materials. The thickness of the black ore increased, with gradual transitions to white-grey and black. Maximal thickness of all types together is 12 m.

In Zović, Neogene sediments lithologically are similar as in Manastir, but with significantly smaller thickness (about 3 m). In the lower parts is leaf-like, and in upper is white and very pure.

Apart from these localities, there are appearances of the diatomaceous earth south of the elevation 70, and Čuka. In Čuka, ore is white, maximum thickness of the diatomaceous earth is detected 14 meters, which is the maximum thickness observed in the area of Manastir–Bešište.

CONCLUSION

Diatomite (diatomaceous earth) in Manastir–Bešište and Zović occurs in a single layer in the form of plate, which is tied for the andesite tuff in the floor layer.

Based on the conducted chemical analyses, diatomaceous earth contains: SiO_2 78.05 – 88.35%, Al_2O_3 4.61 – 10.376%, CaO 0.62 – 0.81%, MgO 0.15 – 0.40% and Fe_2O_3 1.21 – 4.05 %.

The following minerals were identified: quartz, muscovite, illite, pyroxene and amorphous phase.

From microphotos is seen that diatomaceous earth represents multiphase material composed of porous, crystal and amorphous phases.

Diatomaceous earth from the Manastir–Bešište deposit contains significant contents of silicium, aluminium, calcium, potassium, sodium and iron. With lower contents occur titanium and chlorine.

According the mineralogical and chemical examinations, diatomaceous earth from the Manastir–Bešište deposit is of good quality and as a mineral resource may find application in: porcelain industry, food, chemical and pharmaceutical Industries as a means of filtration as waterproof pads, etc.

The deposit is of sedimentary origin and has the shape of a simple plate. It is almost horizontal, which thickness increases going from the former edge of the Neogene lake basin to the middle of it.

The diatomite (diatomaceous earth) deposit between the Manastir and Bešište villages is the largest of that type in the former Yugoslavia, which reserves will increased with further geological and mining investigations.

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Р е з и м е

НОВИ СОЗНАНИЈА ЗА ДИЈАТОМИТОТ (ДИЈАТОМЕЈСКА ЗЕМЈА) ПОМЕЃУ СЕЛАТА МАНАСТИР И БЕШИШТЕ (МАРИОВО)

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Клучни зборови: дијатомејска замја; Манастир; Бешиште; сканинг-електронски микроскпоп; хемиски состав; квалитет на рудата

Диајтомејската земја како порозна, фино гранулирана силикатна седиментна карпа поради своите специфични физичко-хемиски својства често привлекувала внимание за проучување од стручен и научен аспект. За дијатомејската земја од наоѓалиштето Манастир – Бешиште во геолошката литература многу малку е пишувано, особено за нејзиниот минералошки и хемиски состав. Во трудот се прикажани најновите испитувања и добиените резултати добиени од примероци земени од површината на теренот од наоѓалиштето Манмастир–Бешиште. Овие испитување се направени во лабораториите на Факултетот за природни и технички науки со методот на рендгенска дифракција и со сканинг-електронска микроскопија. Од рендгенскиот дијаграм на прав се гледа дека во испитуваниот примерок се застапени следниве минерални фази: кварц, илит, мусковит, пироксен и аморфна фаза.