

## CHARACTERIZATION OF DIATOMACEOUS EARTH FROM THE SLAVISHKO POLE LOCALITY IN THE REPUBLIC OF MACEDONIA

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**A b s t r a c t:** With the complex examination of raw material from a new deposit in Slavishko Pole in the region of Kratovo–Zletovo volcanic area, Republic of Macedonia, its physical, chemical and mineralogical-petrographical properties have been defined. It has been found that it is a dominantly amorphous material, sedimentary rock of the type silicite-diatomite, with biogenic or phytogenic origin. The raw material consists of approximately 72% SiO<sub>2</sub> and it can be classified as diatomite of the type 1 (for 70–80% SiO<sub>2</sub>) according to British Standard Specification (BS 1795:1976) [1]. Of the physical properties, more significant is porosity, which is higher than 60%. The raw material has a wide spectrum of possibilities for practical application: intensive absorbent for several types of liquids, regulator of physical and chemical properties of the soil, ionic substitution of heavy metals in soil, natural insecticide, clarifier in food industry etc. For determination of the chemical contents, properties and origin of the raw material the following methods have been used: silicate chemical analysis, XRD analysis, transmission optical microscopy and other methods.

**Key words:** diatomaceous earth; characterization; chemical analysis; XRD analysis; optical microscopy

### INTRODUCTION

Inorganic raw materials in the Republic of Macedonia have a wide spectrum of their potential application [9, 10]. Slavishko Pole, as a former lake of Pliocene age, has a potential of exploiting biogenic sediments of the type of diatomaceous

earth [2–8]. According to the defined properties, the raw material offer many possibilities for practical application in inorganic industry and agriculture.

### EXPERIMENTAL

#### *Geological and geographical setting*

The deposit of the raw material is situated in the furthest northeast of the Republic of Macedonia (Fig. 1), near Vetunica village, the municipality of Rankovce, as part of Slavishko Pole. The microlocality is situated in the boundary area of the Kratovo-Zletovo volcanic area with the Serbian-Macedonian massif.

#### *Mineralogical-petrographical examinations*

The mineralogical-petrographical examinations of the raw material have been realized by transmission optical microscopy and XRD analysis.



**Fig. 1.** Geographical map of the deposits

### Macroscopic properties

The raw material (Fig. 2.) is of white color and fine grained structure. It is crumbling easily in hand, at which fine grains is released. The raw material absorbs water easily, which indicates it is highly porous, and becomes of grayish-white color. It takes significant time to release the absorbed water and regain its primary white color. The raw material has no high volume mass, due to its high porosity. With heating to 100°C the material darkens, which indicates presence of organic material. The raw material is easily scratched with no significant resistance. The sample is layered, with color changes into creamy-white color, in thin layers of dimensions of 1 – 2 mm. Considering the abovementioned macroscopic properties, it can be presumed that the raw material represent a not strong bounded silicite sediment.

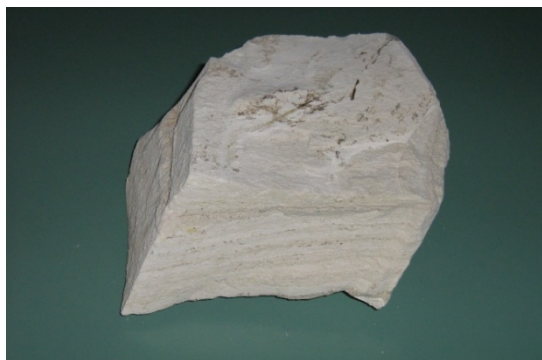


Fig. 2. Macroscopic figure of raw material.

### Microscopic examinations

The microscopic examinations have been realized by transmission optical method, on the microscope SM-POL, Letz-Wetzlar, Germany.

With the microscopic examination of the sample was determined that raw material is consisted of cryptocrystalline ground mass (over 95%). There can be found in several percent very fine rounded grains of quartz and feldspars with dimensions of 0.005 – 0.01 mm to 0.05 – 0.1 mm. Feldspars has polysynthetic twin lamells (Fig. 3) of acid plagioclases of albite-oligoclase type.

In the cryptocrystalline ground mass can be found long (approximately 0.1 mm) sticks of alga Diatomeae (Figs. 4 and 5). These sticks have elongated forms which are transverse cut by longitude and are connected. Some of the sticks in the middle of their longitude are boundary by a symmetrical line, therefore these relics are reminiscent of silicite spicule of the type spongolite (Figs. 6 and 7).

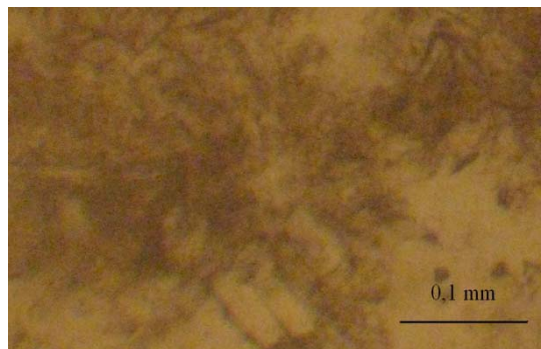


Fig. 3. Polysynthetic twin lamells of feldspars

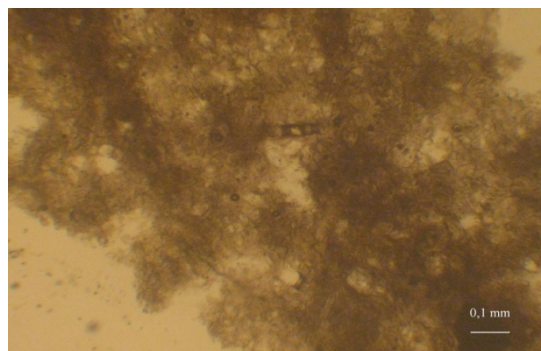


Fig. 4. Typical longitudinal section of fine grain Diatomeae (0,1 mm) incorporated into cryptocrystalline ground amorphous opal mass in which co-exist round grains of feldspars and quartz

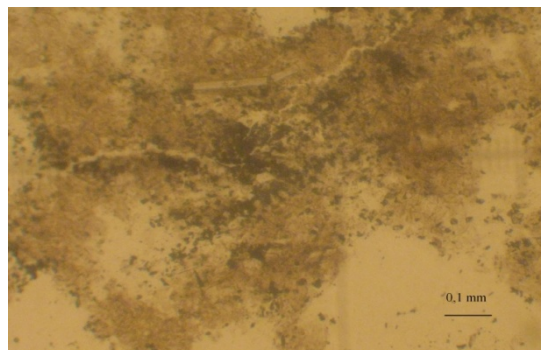


Fig. 5. Fine grain Diatomeae partially grained and incorporated in cryptocrystalline ground amorphous mass

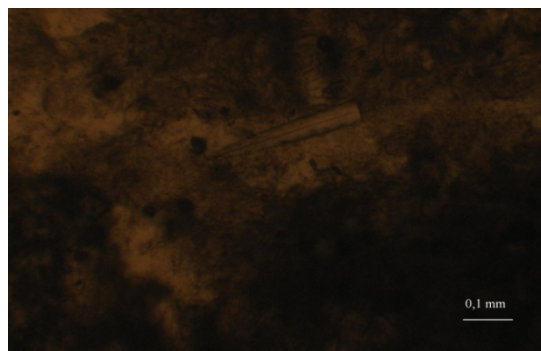


Fig. 6. *Spongolite spicule* incorporated in cryptocrystalline ground amorphous opal mass (N<sup>7</sup>)

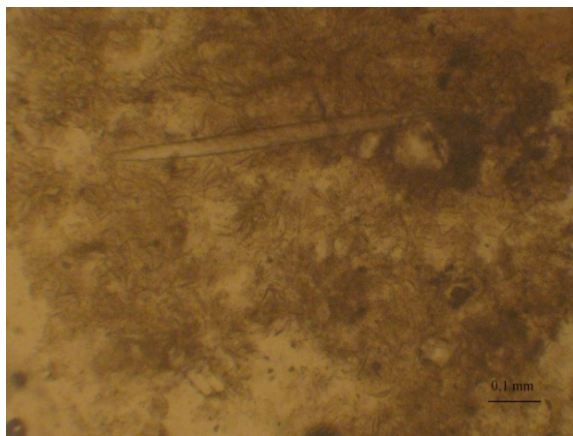


Fig. 7. *Spongolite spicule* incorporated in cryptocrystalline ground amorphous opal mass (N)

Identification of the microfossils in this sediment is carried by a comparative analysis of identical species, quite well known according to literature data [11].

The structure of the rock is crypto-crystalline to amorphous, and the texture is very low layered and massive to homogenous. Based on the above-mentioned, it points to the fact that the raw material is sedimentary rock of the type silicite-diatomite, with biogenic (phytogenic) origin. Beside the presence of opal of biogenic origin, it is not excluded that there might be presence of opal of inorganic origin.

The raw material could be treated also as a trepel, most probably of Pliocene age. It was formed by sedimentation of opals of biogenetic origin – former Pliocene alga *Diatomeae*, due during their existence extracted the silica and incorporated it in their skeletons. After extinction, these opal algae are sedimented on the lake bottom along with the mechanically incorporated fine grain of quartz and feldspars. Hypothetically it is not excluded the presence of a minimal admixture of clay (around or less than 1%) in these silicates.

#### XRD analysis

XDR analysis was realized on the DRON-UMI X-ray diffractometer ( $2\theta = 2^\circ - 60^\circ$ ; 30 kV; 20 mA;  $\text{CuK}\alpha/\text{Ni}$ ). The interpretation of XRD has been conducted on the basis of data from ICPDS.

According to the XRD the following can be concluded:

The specific shape of the diffractogram and the high level of basic line, suggest a high mass quantity of amorphous (opal) mass in the raw material.

From the XRD have been determined F – feldspars (K feldspar of the type microcline), as well as Na-Ca type of plagioclase of albite-oligoclase type. The raw material also contains minimal mass quantities of quartz (at the limit of sensitivity of the method).

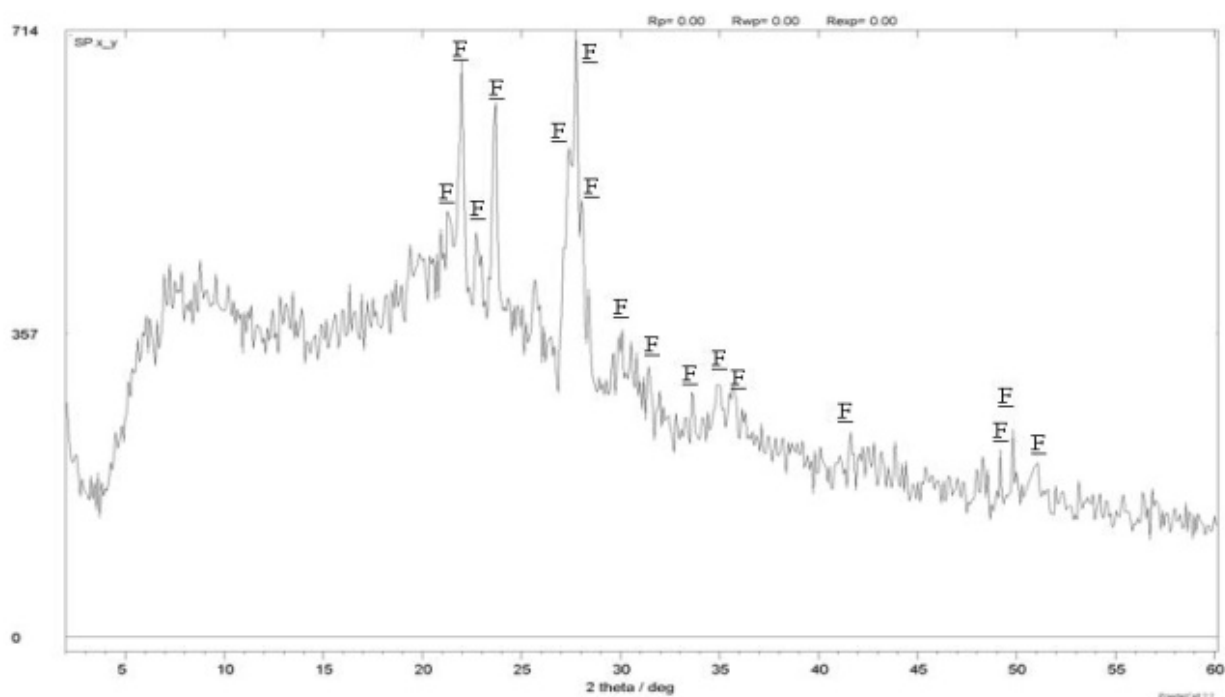


Fig. 8. XRD of the raw material (F – feldspars)

Between  $2\theta = 7-13^\circ$ , the diffractogram is hard to determine due to low intensity. In this interval there are XRD peaks of some natural zeolites. This suggests that with these examinations zeolites are not exactly determined, so it could be stated that they are only hypothetical.

#### Physical-chemical properties

A classical silicate chemical analysis has been applied for determination of the chemical composition of the raw material. The results have been presented in Table 1.

Table 1

#### Chemical composition of the raw material

SiO <sub>2</sub>	72.07
Al <sub>2</sub> O <sub>3</sub>	12.09
Fe <sub>2</sub> O <sub>3</sub>	1.00
CaO	2.95
MgO	1.41
K <sub>2</sub> O	1.90
Na <sub>2</sub> O	2.10
SO <sub>3</sub>	tr.
l.w	5.76
$\Sigma$	99.28

The following physical properties have been determined:

- specific mass,
- volume mass,
- water absorption,
- porosity (open, closed, total),
- pressure strength.

The results of the physical examination are presented in Table 2.

Table 2

#### Physical properties of the raw material

Specific mass (g/cm <sup>3</sup> )	2.355
Volume mass (g/cm <sup>3</sup> )	0.928
Water absorption (%)	50.20
Open	46,59
Porosity (%) Closed	13.99
Total	60.58
Pressure strength (MPa)	6.23–7.62

The pressure strength is given in an interval, due to various values because the raw material has a variable oriented layer structure.

## CONCLUSION

According to the examinations of the natural raw material from the Slavishko Pole locality and the presented results, it can be concluded that it is a sedimentary silicite rock with dominant biogenic origin of the diatomite type. As a significant admixture of inorganic origin, are feldspars in several variations, as well as minimal quantities of cryptocrystalline quartz. The raw material of structural aspect is dominantly amorphous. According to data of physical properties as well as according to the

chemical and mineralogical composition, the raw material can be applied in process of soil treatment from the aspect of regulator of porosity and regulator of the accumulative water capacity. From the present alkaline and earth alkaline mineral, the raw material has a potential capacity for cationic substitution. The material would be exceptionally efficient natural insecticide, with application of separated granulometric fractions.

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## Резиме

## КАРАКТЕРИЗАЦИЈА НА ДИЈАТОМЕЈСКА ЗЕМЈА ОД ЛОКАЛИТЕТОТ СЛАВИШКО ПОЛЕ ВО РЕПУБЛИКА МАКЕДОНИЈА

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**Клучни зборови:** дијатомејска земја; карактеризација; примена; XRD; оптичка микроскопија; хемиска анализа

Со комплексните испитувања на суровина од ново лежиште во Славишко Поле, во регионот на кратовско-злетовската вулканска област, Р. Македонија, дефинирани се нејзините физичко-хемиски и минералшко-петрографски карактеристики. Утврдено е дека се работи за доминантно аморфна суровина – карпа, која претставува силицит од типот на дијатомит и има биогено-седиментно или фитогено потекло. Суровината содржи приближно 72% SiO<sub>2</sub> и може да се класифицира како дијатомит од типот 1 (70–80% SiO<sub>2</sub>), според British Standard Specification (BS 1795:1976) [1]. Од физичките карактеристики позначајна е порозноста, која надминува 60%.

Суровината со дадените карактеристики има широко поле на можности за практична примена, и тоа во повеќе области, како, на пример, интензивен апсорбент за повеќе видови течности, регулатор на почвените физичко-хемиски карактеристики, јонски изменувач за тешки метали во почва, природен инсектицид, избиструвач во прехранбената индустрија итн. За детерминација на составот, карактеристиките и потеклото на суровината се употребени следните методи: силикатна хемиска анализа, XRD-анализа, оптичка трансмисиона микроскопија и други методи, потребни за физичко-хемиската карактеризација.