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GEOME 2
Manuscript received: November 5, 2020
Accepted: March 3, 2021

Geologica Macedonica, Vol. **35**, No. 1, pp. 5–14 (2021)
On print ISSN 0352 – 1206
On line ISSN 1857 – 8586
UDC: 552.581.08(497.7)
DOI: 10.46763/GEOL21351368005ar

Original scientific paper

DIATOMITE – EVALUATION OF PHYSICO-MECHANICAL, CHEMICAL, MINERALOGICAL AND THERMAL PROPERTIES

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A b s t r a c t: Diatomite is one of the most intensively examined raw materials in the materials industry with a broad range of various applications. The diatomite sample, collected from Vitačevo plateau in the vicinity of Kavadarci, was fully characterized by means of physical-mechanical, chemical, XRPD, SEM, TEM, DTA/TGA and IR techniques. The physical-mechanical features pointed out to soft, light, white to gray rock with shell-like structure exhibiting compressive strength from 4.65–4.88 MPa in dry form, whereas the total porosity ranges 70–72% and the density is 2.06–2.09 g/cm^3. The chemical analysis of the diatomite revealed that SiO_2 content exceeds 91%. The results from the X-ray powder diffraction indicate predominant amorphous SiO_2 phase associated with minor presence of crystalline quartz, muscovite, chlorites and plagioclase. The IR spectrum of the diatomite manifested characteristic bands for amorphous silica at 799 cm^{-1} and 1101 cm^{-1}. DTA/TGA results display great thermal stability of the sample remaining amorphous up to 1050ºC whereas the SEM analysis determined the morphology, surface characteristics and the nanometric pores in the raw material. Thus, the studied diatomite is classified as a natural nanomaterial that is suitable for broad application in various construction materials, refractory ceramics, special oxide ceramics, and also finds potential use in filtering, adsorbent, catalysts, food and pharmaceutical industries.

Key words: diatomite; amorphous silica; nanomaterial; thermal stability; phase analysis

INTRODUCTION

Republic of North Macedonia is very diverse in various non-metallic raw materials including diatomite, clayey diatomaceous earth, bentonite, dolomite, pumice, perlite, granite, quartzite, tuffs, etc., that have extensive usage and application (Spasovski and Spasovski., 2012; Makreski et al., 2009; Jovanovski et al., 2012; Memedi et al., 2016a, 2016b, 2016c, 2017; Reka et al., 2014, 2018, 2019; Pavlovski et al., 2011, 2018; Boškovski et al., 2015; Bogoevski et al., 2014; Cekova et al., 2013). Diatomite also known as kieselguhr is biogenic sedimentary rock composed of the opaline frustules of diatoms (Eldernawi et al., 2014; Smirnov et al., 2017; Ediz et al., 2010; Ilia et al., 2009). Diatomite remarkable properties comprises high porosity, low bulk density, permeable structure, chemical resistance, high purity, large specific surface area, high adsorption capacity that find use in the food industry

for filtration purposes (sugar syrup, water, fruit juices, etc.). In addition to the application in filtration, the diatomite finds various use as pozzolanic additive for cement, adsorbent, catalytic carriers, construction, environmental engineering, removal of textile dye, organic pollutants, heavy metals, glass industry, production of porous ceramics, humidity control materials, production of prolonged-release drug carriers (Vu et al., 2013; Bello et al., 2014; Yılmaz and Ediz, 2008; Janićijević et al., 2014; Chen et al., 2020; Akhtar et al, 2010; Reka et al., 2016, 2017; Manevich et al., 2012; Athar and Asilian, 2012; Inglethorpe, 1993; Fragoulis et al., 2005; Rahimov et al., 2014; Ibrahim and Selim, 2012; Loganina et al., 2014; Zheng et al., 2018; Paules et al., 2018; Semenkova et al., 2020; Belousov et al., 2020; Yatsenko et al., 2020; Goltsman et al., 2020a, 2020b). Diatomite may contain impurities such as iron, aluminum, magnesium, calcium, predominantly originating form clay minerals, micas and feldspars depending on the origin location (Reka et al., 2015; 2019b, 2019c). During the industrial preparation process, the impurities are removed utilizing air separators, cyclones and magnetic separators (Lamamra et al., 2020a, 2020b. 2021). The aim of this work was to continue with the characterization of the natural resources from North Macedonia with particular emphasis on diatomite from Vitačevo plateau (Kavadarci region, Figure 1).

Fig. 1. Geographical map of North Macedonia that pinpoints the diatomite deposit in Vitačevo (plateau near Kavadarci).

MATERIALS AND METHODS

The consolidation time and the occurrence of volcanic rocks in the area were calculated as being between 6.0 and 1.8 m.y. using the K/Ar method (Boev et al., 1988), and this is consistent with their stratigraphic age.

The chemical composition of the diatomite was determined with the classical silicate analysis. The diatomite was fused in a mixture of carbonates, while the percentages of various oxides present in the raw material were determined with complexometric titration. The determination of the alkali metal oxides (Na_2O and K_2O) was performed by flame photometry using an Evans Electroselenium Ltd 410 instrument, while the presence of the trace elements was performed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Agilent 7500 cx).

The mineralogical characterization of the diatomite was carried out by an X-ray powder diffraction (XRPD), thermal analysis (TGA/DTA), scanning electron microscopy (SEM-EDX), transmission electron microscopy (TEM) and infrared spectroscopy (IR).

XRPD analysis was performed on Rigaku Ultima IV X-ray diffractometer equipped with D/teX high-speed 1-dimenzional detector using CuKα radiation (λ = 1.54178 Å) in 2θ range from 5 to 60°. The accelerating voltage and the current power were set to 40 kV and 40 mA, respectively.

DTA/TGA analyses of the diatomite were guided in air environment with Stanton Redcroft apparatus, under the following experimental conditions: temperature range from 20 to 1050°C; speed of heating set to 10°C/min; sample mass of 11 mg; and ceramic pot as a material carrier.

Scanning electron microscopy VEGA3 LMU coupled with energy dispersive X-ray spectroscopy (INCA Energy 250 Microanalysis System) was used to quantitative analyzing the material. The accelerating voltage of the SE detector was set to 20 kV.

Transmission electron microscopy on the natural diatomite was carried out on Hitachi H-7650 apparatus (120 kV automatic microscope).

The Perkin-Elmer FTIR system 2000 interferometer was engaged to record the IR spectra in 4000–500 cm^{-1} range using the KBr pellet method. The pellet was prepared by a mixture of 1 mg of the sample in 250 mg dried KBr loaded under pressure of 10 tons cm^{-2}.

The optical microscopy measurements were conducted on a transmission polarizing microscope SM-POL, Leitz, Wetzlar, Germany.

The diatomite specimen (one bag) was collected from Vitačevo region, Republic of North Macedonia.

RESULTS AND DISCUSSION

Physical-mechanical characteristics and chemical composition of diatomite

The tested diatomite sample (Figure 2) represents soft and loose white-colored material with homogeneous texture and shell-like fragility. The tested sample easily disintegrates by applying pressure while the fine particles gives the sensation of a scratch. The sample is characterized by a low bulk density of 0.59–0.61 g/cm^3. Total of 3 samples were tested whilst determining the bulk density.

Fig. 2. Excavation of the raw diatomite from the collection site

The diatomite expressed total porosity within 70–72 %, while its compressive strength spans from 4.65 to 4.88 MPa. The results of the physical-mechanical characteristics (Table 1) are given in intervals due to the zonal variations.

T a b l e 1

Physical-mechanical characteristics of diatomite from Vitačevo

Property	Determined value
Compressive strength	4.65–4.88 MPa
Bulk density	0.59–0.61 g/cm^3
Water absorption	55–57 %
Total porosity	70–72 %

The major and trace elements content in the diatomite was determined (Tables 2 and 3). The results obtained from the chemical composition of the diatomite confirmed the high purity of the material with a dominant presence of SiO_2 (91.84 %, Table 2). The content of 45 trace elements was rather low (<10 ppm) except for the earth alkaline Ba and Sr metals and V, Y, Zr with values between 20 and 50 ppm. The loss of ignition (LOI) is determined (5.37%) while heating the sample at 1000ºC for a period of 1 hour.

T a b l e 2

Major oxides of diatomite from Vitačevo

Oxides	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	MnO	P_2O_5	K_2O	Na_2O	LOI	Total
Mass %	91.84	1.64	0.28	0.03	0.32	0.14	0.007	0.04	0.12	0.09	5.37	99.87

T a b l e 3

Results of trace element analysis of diatomite from Vitačevo

Elements	ppm	Elements	ppm	Elements	ppm	Elements	ppm	Elements	ppm
Ag	<0.5	Cu	<10	In	<0.1	Rb	6.6	Tl	<0.5
As	<5.0	Dy	0.40	La	3.34	Sb	3.7	Tm	0.46
Ba	49.4	Er	0.29	Lu	0.051	Sc	<1	U	2.28
Be	1	Eu	0.07	Mo	<2.0	Sm	0.43	V	24.4
Bi	<0.1	Ga	<1	Nb	4.74	Sn	<1	W	<0.5
Ce	3.63	Gd	0.39	Ni	<20	Sr	16.6	Y	26.6
Co	<1.0	Ge	<0.5	Nd	2.45	Ta	0.024	Yb	0.31
Cr	<20	Hf	0.2	Pb	<5	Tb	0.06	Zn	<30
Cs	2.68	Ho	0.09	Pr	0.60	Th	0.66	Zr	25.2

X-ray powder diffraction analysis of diatomite

XRPD pattern of the diatomite from Vitačevo (Figure 3) revealed the characteristic halo peak positioned between 16 and 31° (2θ) resulting from the abundance of the amorphous opal. The obvious crystalline phases, present in significantly lower amounts, comprised silica (quartz peaks: 4.26Å or 20.80°; 3.34Å or 26.59°; 2.28Å or 39.42°; 1.81Å or 50.09°; 1.54Å or 59.96°), feldspars (plagioclase peaks, 3.76Å or 23.50°; 3.68Å or 24.14°; 3.21Å or 27.75°; 3.19Å or 27.94°; 3.13Å or 28.42°) and mica (muscovite peaks, 9.99Å or 8.84°; 4.04Å or 21.95°; 3.49 Å or 25.50°; 3.31 Å or 26.87°; 3.19Å or 27.89°; 3.03Å or 29.43°; 2.56Å or 34.89°; 1.98Å or 45.64°) (Anthony et al., 1995; Lafuente et al, 2015).

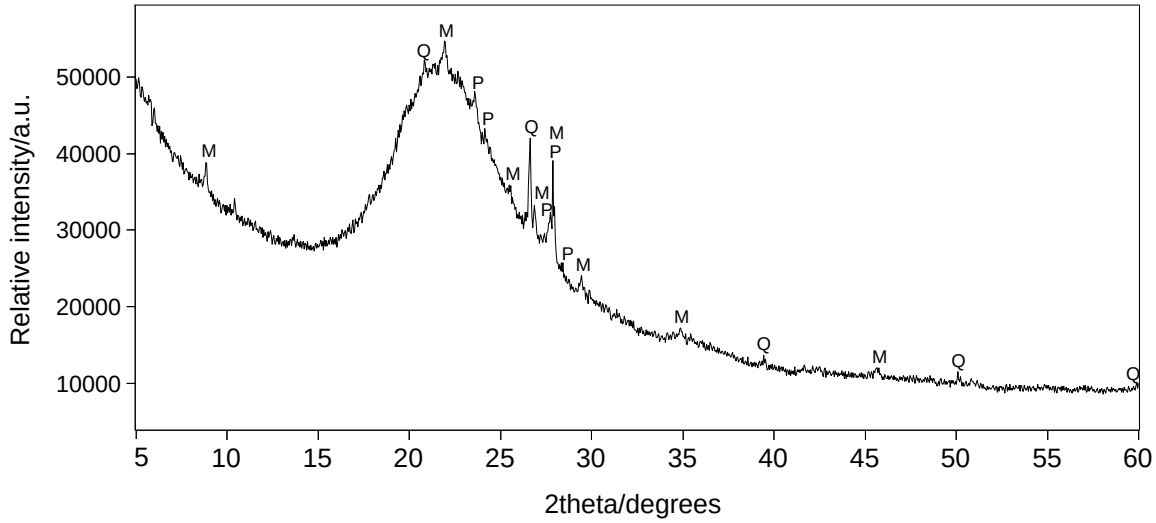
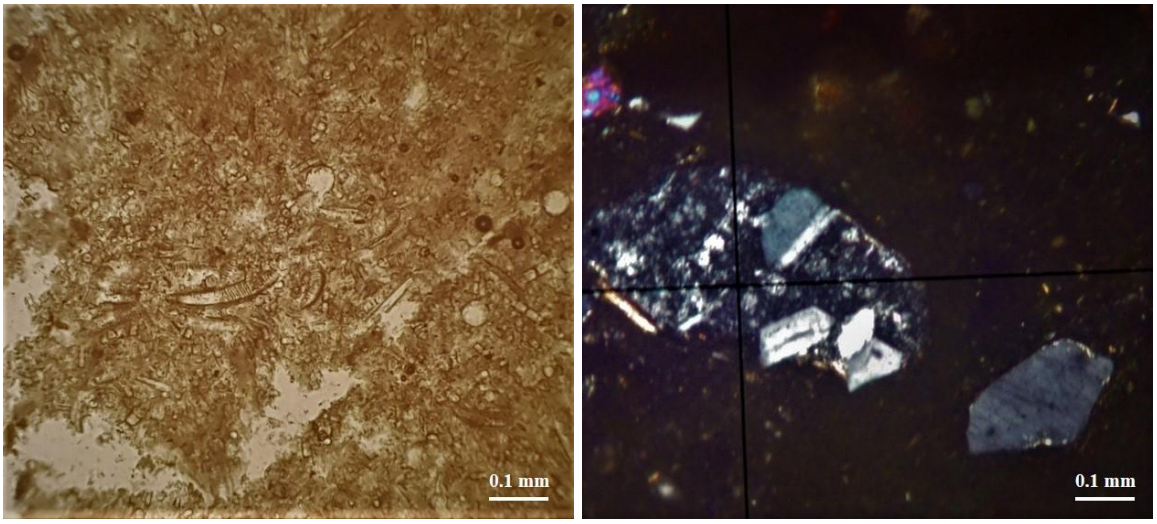


Fig. 3. XRPD pattern of diatomite (Q: quartz, M: muscovite, P: plagioclase)

Mineralogical-petrographic analysis

Based on the results obtained from the mineralogical-petrographic examinations (Figure 4), diatomite predominantly comprised crypto-crystalline opal matrix with numerous diatom frustules and various unidentified microfossils (most probably algae) in the flake formations and rod shape. Grains of monocrystal quartz and lithic fragments of chrystal-vitric tuff were also observed in the opaline phase.



a b

Fig. 4. Transmission optical microscopy of diatomite. **a)** (N–) – diatom frustules in the basic mass (amorphous matrix); **b)** (N+) – presence of quartz and a lithic fragment of crystal of vitric tuff in the basic mass of diatomite

Scanning electron microscopy results of diatomite

The scanning electron microscopy study of diatomite (Figure 5) has proven the biogenic origin of the material confirmed by the skeletal forms of diatoms with rod or flake shape. In addition, a manifestation of the 250–500 nm pores in the frustules was evidenced with majority of them open and free from impurities.

The EDX spectrum (Figure 6) enabled to quantitatively determine the chemical composition of the diatomite sample. The atomic ratio of oxygen and silicon is 1.88 (O: 65.24% and Si: 34.76%), approaching to the ideal molar O:Si ratio (2) in amorphous opal (SiO_2) confirming its major presence in the material.

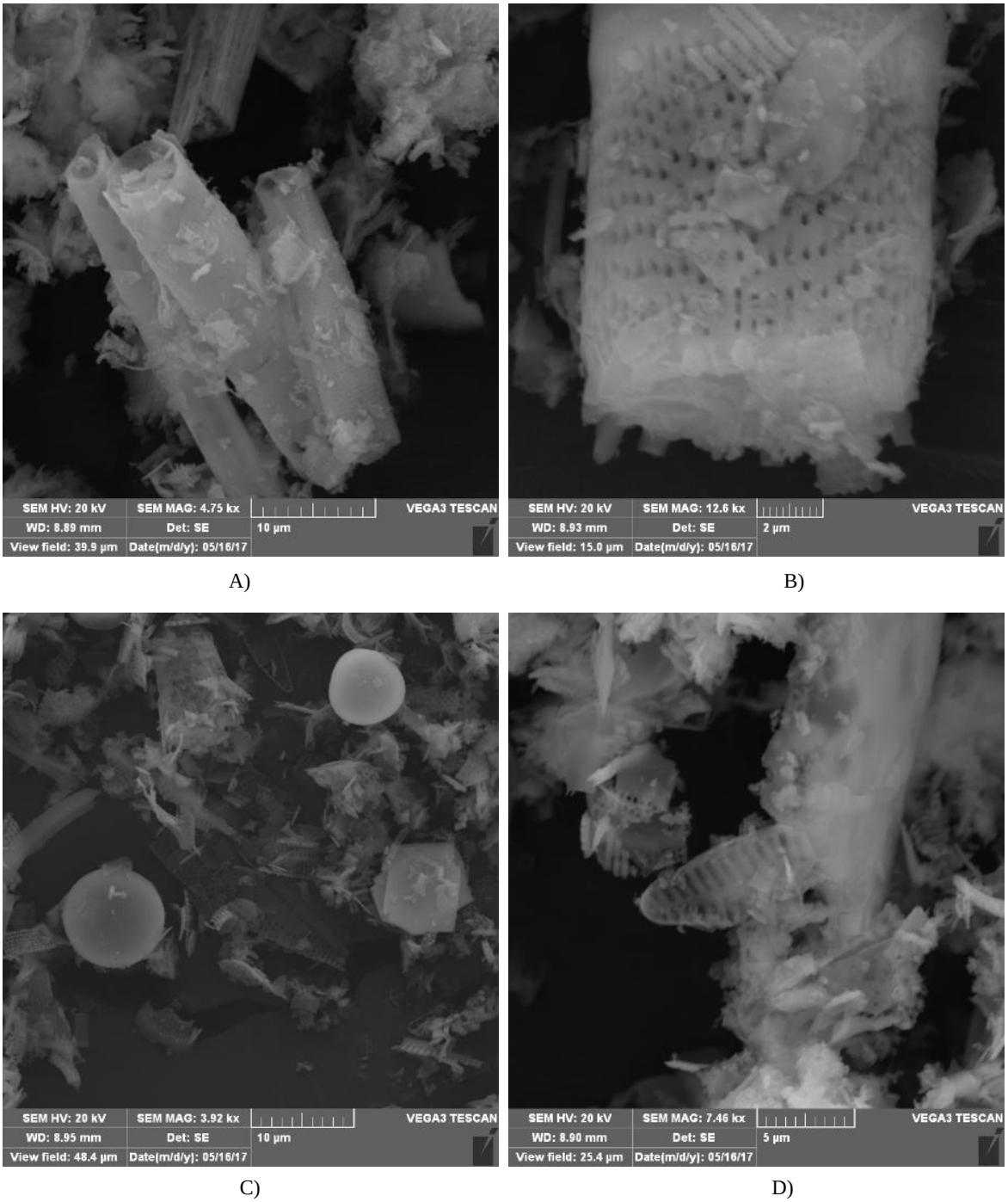


Fig. 5. SEM of diatomite. A) Rod-like diatoms with dimensions 25–35 microns length and 8–12 microns width. B) Close up view of rod-like diatoms. C) Various fragments of diatoms and lepispheres of silica. D) Fragments of diatoms and flake shape frustule in the middle

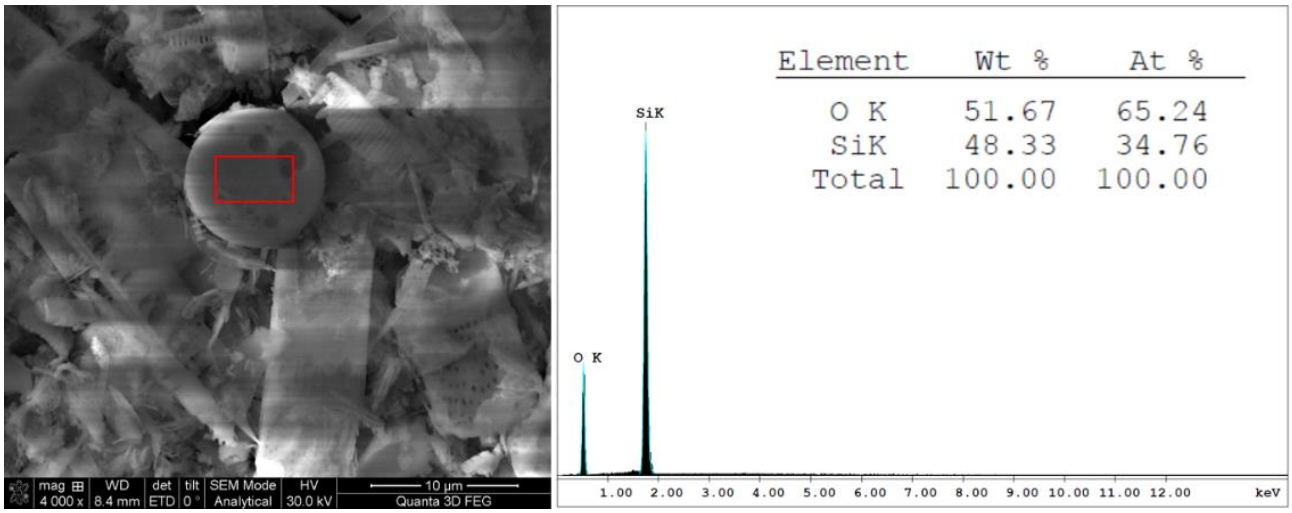


Fig. 6. Secondary electron image photos of diatomite

Transmission electron microscopy results of diatomite

The TEM observation of diatomite (Figure 7) nicely complement to the SEM results in evaluating the morphology and the pores shape and size. The diatomite particles exhibit smooth and clean surface with well-opened pores. Certain fragments of the frustules manifest ordered pores with diameters ranging 250–450 nm. Particles of impurities are occasionally found in the pores. TEM images show that the main composition of the diatomite rests as glassy mass, being the dominant amorphous phase in the sample.

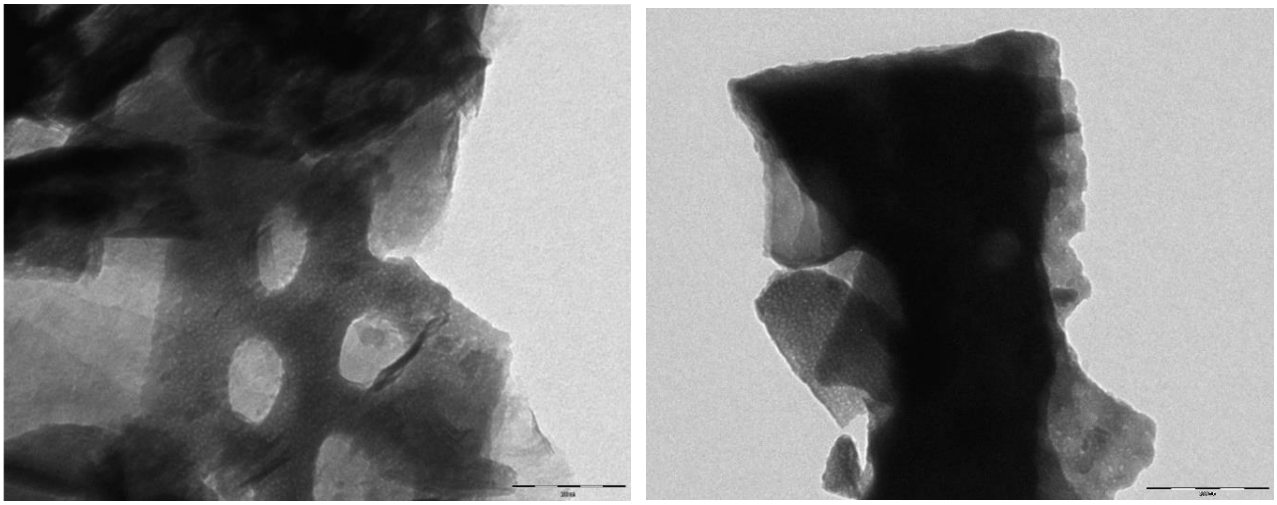


Fig. 7. TEM of diatomite show arrangement and details of the porous structure and the amorphous glassy mass

Thermal analysis of diatomite

The results from the thermo-gravimetric analysis of the diatomite (Figure 8) showed mass loss in two temperature intervals. The first interval spans from room temperature to 245°C where intensive mass loss of 6.5% was registered and attributed to the elimination of adsorbed water from the sample surface and from the diatomite pores. The second temperature interval (246–850°C) pointed on rather smaller mass loss (less than 1%) ascribed to the dehydration of the opaline phase and here (200–650°C) the process of morphological ordering of the amorphous silica took place. The DTA/TGA analysis showed no exothermic peak above 900°C which is a confirmation that the material remained amorphous.

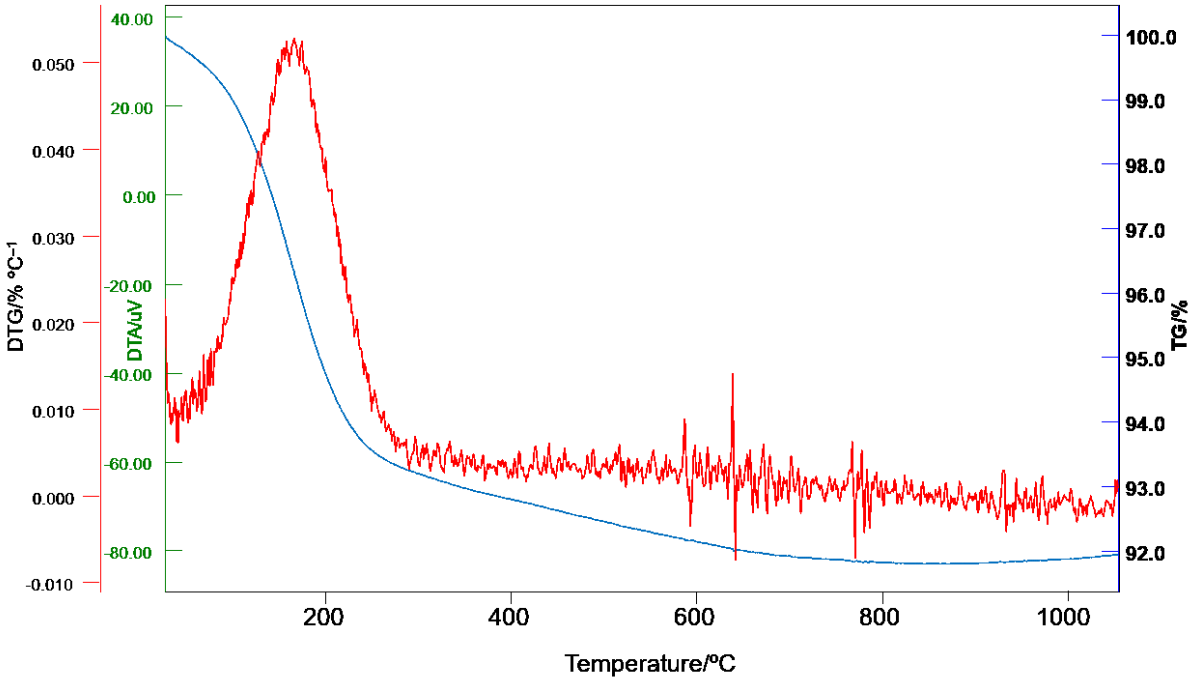


Fig. 8. DTA/TGA of diatomite

FTIR spectroscopy of the diatomite

The IR spectra (Figure 9) of a diatomite displayed sharp absorption band at 799 cm^{-1} that is assigned to the bending vibrations of the Si–O–Si framework (Ilia et al, 2009). Moreover, the strongest band with maxima centered at 1101 cm^{-1} is related to the stretching Si–O–Al vibrations, whereas the shoulder on its higher-wavenumber side (1235 cm^{-1}) was prescribed to the antisymmetric vibration of Si–O mode (extension-compression of Si–O distance) (Chen et al., 2020). The remaining bands in the spectrum exhibit lower intensity and are related to the presence of water molecules. Thus, band at 1638 cm^{-1} is attributed to the bending vibrations of the absorbed water, while the wide band around at 3444 cm^{-1} is due to the stretching vibrations of the absorbed water molecules in the sample. The shoulder around 3644 cm^{-1} originates from the OH stretchings within the structure of the present clay minerals in the sample (Chukanov, 2014; Krupskaya et al., 2019; Belousov et al., 2019b).

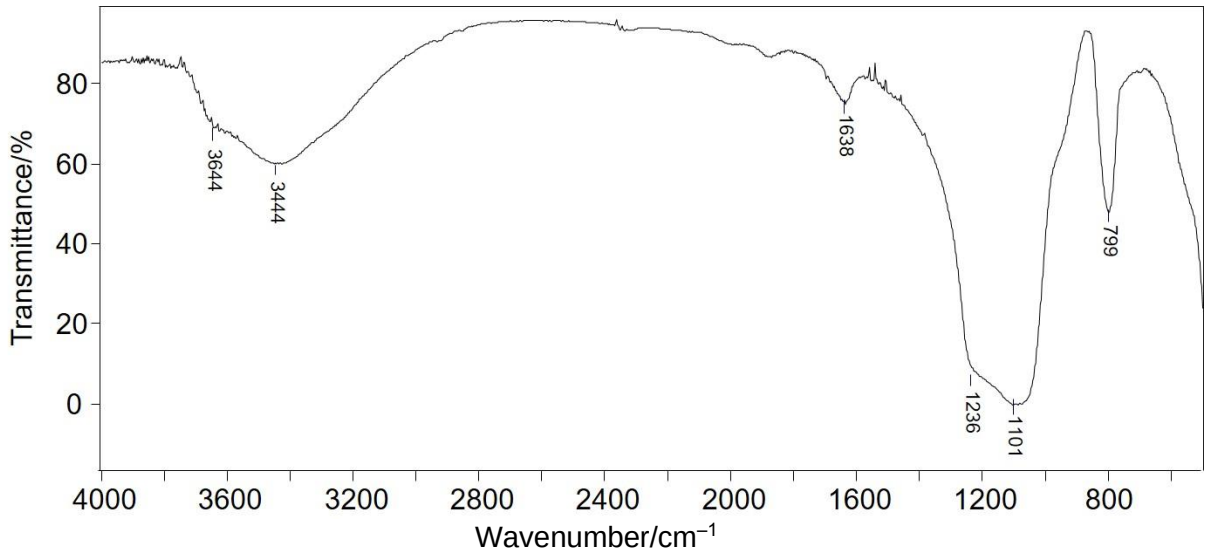


Fig. 9. IR spectrum of diatomite

CONCLUSIONS

The chemical composition of diatomite from Vitačevo region confirmed its high quality due to the high percent of SiO_2 (91.84 %). XRPD revealed the predominantly amorphous behavior with the additional presence of minor content of crystalline quartz, plagioclase and muscovite. SEM showed presence of various skeletal shapes of microorganisms with clearly visible pores and canals. Majority of the pores are open, mostly free from impurities with their size ranging from 250 to 550 nm. These results are complementary to the TEM findings that morphology of the frustules and the original geometry of the pores are well preserved. The observed diatomite features, associated by the great thermal stability of the sample that remained amorphous at temperature of 1050ºC, comply to the demanding specifications of various industrial applications for this natural nanomaterial in catalyst industry, filtering aid as well as in the design of various mesoporous materials, refractory and oxide ceramics. The obvious low level of crystalline silica makes this diatomite a potential materials for use in the food industry as well.

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Р е з и м е

ДИЈАТОМИТ – ЕВАЛУАЦИЈА НА ФИЗИЧКО-МЕХАНИЧКИТЕ, ХЕМИСКИТЕ, МИНЕРАЛОШКИТЕ И ТЕРМИЧКИТЕ ОСОБИНИ

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Клучни зборови: дијатомит; аморфна силика; наноматеријал; термичка стабилност; фазна анализа

Дијатомитот е една од најинтензивно испитуваните суровини во индустријата на материјали со широк спектар на примена. Примерокот од дијатомит земен од висорамнината Витачево во околината на Кавадарци беше целосно карактеризиран со примена на физичко-механичките, хемиските, XRPD, SEM, TEM, DTA/TGA и IR техники. Физичко-механичките карактеристики укажуваат на мека, лесна, бела до сива карпа со структура на школка, со јачина на притисок од 4,65–4,88 MPa во сува состојба, додека вкупната порозност е во опсег од 70 до 72%, а густината од 2,06 до 2,09 g/cm^3. Хемиската анализа на дијатомитот покажува дека содржината на SiO_2 надминува 91%. Резултатите од рендгенската анализа укажуваат на предоминантно аморфна фаза на SiO_2 асоцирана со минимално присуство на кристален кварц, мусковит, хлорити и плагиокласи. IR-спектарот на дијатомитот покажува карактеристични ленти на аморфна силика на 799 cm^{-1} и 1101 cm^{-1}. Резултатите од DTA/TGA покажуваат голема термичка стабилност на примерокот кој останува аморфен до 1050ºC, додека SEM-анализата ги детерминира морфологијата, површинските карактеристики и нанометриските пори во суровината. Затоа испитуваниот дијатомит е класифициран како природен наноматеријал кој е погоден за широка примена како градежен материјал, огноотпорна керамика, специјална оксидна керамика, а исто така наоѓа потенцијална примена во процесите на филтрација, атсорпција, катализа, како и во прехранбената и фармацевтската индустрија.