CHEMICAL, MINERALOGICAL AND STRUCTURAL CHARACTERIZATION OF DIATOMITE FROM REPUBLIC OF MACEDONIA

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Abstract: For the characterization of diatomite from Vitachevo (Kavadarci, Republic of Macedonia), chemical, mineralogical and structural examinations were performed. The results of the chemical analysis of diatomite revealed a SiO_2 content exceeding 89%. The X-ray powder diffraction indicates presence of amorphous phase, as well as presence of crystalline phases quartz, muscovite and cristobalite. The results of the infra-red confirm the results of the XRPD analyses. The SEM examinations explain the high porosity of the material and the presence of pores with nano-metric dimensions ranging from 300-600 nm. Based on the performed analysis the raw material can be utilized for various uses.

Key words: diatomite, mineralogical characterization, structural examinations

INTRODUCTION

Inorganic non-metallic materials are suitable for various uses. Silicon dioxide, also known as silica, is widely spread in nature and it occurs in various forms (Callister & Rethwisch, 2010; Holleman & Wiberg, 2007; Kingery, 1960; Iler, 1978) Republic of Macedonia is rich in amorphous silica based crude materials, and these materials have a wide spectrum of potential use and application (Cekova et al., 2013; Cekova et al., 2014; Pavlovski et al., 2011; Reka et al., 2014). Diatomaceous earth, as well as other inorganic materials that contain amorphous SiO_2 such as trepel, are convenient and promising materials for the production of porous ceramics, refractory ceramics, special oxide ceramics, as well as widespread use as means for filtering, adsorbent, catalysts and other uses due to its natural porosity, low density and mineralogical composition (Reka et al., 2016; Reka et al., 2017). The raw material subject of this research is taken from Vitachevo (Kavadarci, Republic of Macedonia).

METHODS

The chemical composition of diatomite is determined with the classical silicate analysis. The characterization from the mineralogical point of view was performed by X-ray powder diffraction (XRPD), scanning electron microscopy (SEM) and infrared spectroscopy (IR). XRPD analysis was performed on Rigaku Ultima IV X-ray diffractometer equipped with D/teX high-speed 1-dimensional detector using CuKα radiation (λ = 1.54056 Å) in 2θ range from 10 to 60°. The accelerating voltage and the current power were set to 40 kV and 40 mA, respectively. The Perkin-Elmer FTIR system 2000 interferometer was employed to record the IR spectra in 4000–500 cm^{-1} range using the KBr pellet method. The spectral resolution was set to 4 cm^{-1} and the spectrum merged from 16 measurements. Scanning electron microscopy (SEM) of the products was performed with energy dispersive X-ray spectroscopy (EDX) employing FEI Quanta 3D FEG dual beam microscope.

RESULTS

The physical-mechanical analysis of diatomite showed that it represents a white to grey rock. The subject diatomite has a massive homogeneous texture and shell-like fragility. It represents a light, soft, weakly bound rock, and in dry condition exhibit compressive strength of 3.4–4.6 MPa. The rock's bulk mass and porosity are ranging from 0.55 to 0.60 g/cm^3 and 73–75%, respectively. Results of the XRPD analysis of the diatomite (Fig. 1) depicts amorphous behaviour of the sample manifested by the appearance of one complex “bump” widely positioned between 15 and 30° (2θ) with the maximum peaking in the 18–26.7° range. However there is presence of the following crystalline phases: quartz, muscovite and cristobalite. The IR spectrum of diatomite exhibits absorption bands at 800 cm^{-1} as result of the bending vibrations of Si-O-Si framework, whereas the band at 1103 cm^{-1} is as result of the stretching vibrations of Si-O-Al units. The band at 1636 cm^{-1} is due to bending vibrations from the absorbed water, while the band at 3435 cm^{-1} is due to the stretching vibration of the absorbed water molecules. The results from the scanning electron microscopy show presence of various skeletal shapes and their morphological characteristics, skeletons of microorganisms which have clearly visible pores and canals. Majority of the pores are open (and do not contain impurities) and are in the range 300-600 nm.

DISCUSSION

Based on the detailed examination of the diatomite from Vitachevo (Kavadarci, Republic of Macedonia), the crude diatomite represents a weakly bound, soft loose rock with a white to greyish white color; it has a low bulk mass and high porosity. The results obtained from the chemical composition of diatomite, indicate material with high purity, with a dominant presence of SiO_2 (89.15%), while the presence of the remaining oxides are as follows: Al_2O_3 (0.64 wt. %), Fe_2O_3 (0.23 wt. %), TiO_2 (0. 031 wt. %), CaO (0.20 wt. %), MgO (0.07 wt. %), MnO (0.005 wt. %), P_2O_5 (0.03 wt. %), K_2O (0.11 wt. %), Na_2O (0.10 wt. %) and LOI 9.10. The XRD analyses show that the material depicts amorphous behavior as well as presence of crystalline phases (quartz, muscovite and cristobalite). The infra-red spectra of the raw materials shows the characteristic absorption bands at 800 cm^{-1} as result of the bending vibrations of Si-O-Si framework, whereas the band at 1103 cm^{-1} is as result of the stretching vibrations of Si-O-Al units. The band at 1636 cm^{-1} is due to bending vibrations from the absorbed water, while the band at 3435 cm^{-1} is due to the stretching vibration of the absorbed water molecules. The scanning electron microscopy results shows presence of various skeletal shapes and their morphological characteristics, skeletons of microorganisms which have clearly visible pores and canals. Majority of the pores are open (and they do not contain impurities) and are in the range 300-600 nm.

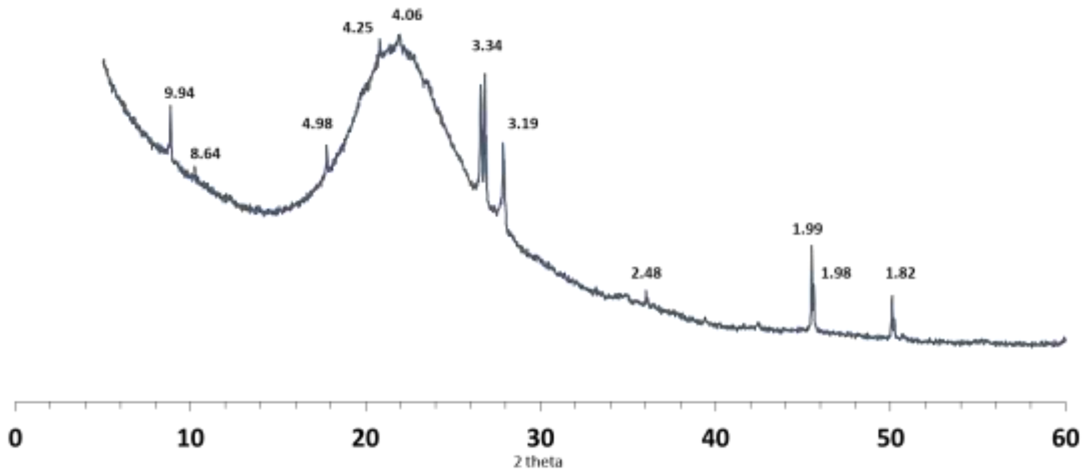

Figure 1. XRPD pattern of diatomite from Vitachevo (Kavadarci)

CONCLUSIONS

Based on the abovementioned results the following can be concluded: the raw material represents a white to grayish rock, porous, soft and weakly bound. A high quality diatomaceous earth, with high percent of SiO_2 (89%), and small amount of impurities: Al_2O_3 (0.64 wt. %), Fe_2O_3 (0.23 wt. %), TiO_2 (0. 031 wt. %), CaO (0.20 wt. %), MgO (0.07 wt. %), MnO (0.005 wt. %), P_2O_5 (0.03 wt. %), K_2O (0.11 wt. %), Na_2O (0.10 wt. %), while LOI is 9.10%. From the mineralogical point of view, the diatomaceous earth represents amorphous material with presence of crystalline phases (quartz, muscovite and cristobalite). The results from the scanning electron microscopy shows presence of various skeletal shapes and their morphological characteristics, skeletons of microorganisms which have clearly visible pores and canals. Majority of the pores are open (and they do not contain impurities) and are in the range 300-600 nm.

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