CHEMICAL, SPECTRA-STRUCTURAL AND MICROSCOPY STUDY OF THE NATURAL TRIDYMITE FROM REPUBLIC OF MACEDONIA

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ABSTRACT:

The naturally occurring tridymite collected from Kavadarci region, Republic of Macedonia represents a pretty loose, soft, weak rock with white to greyish color that resembles diatomaceous earth. The chemical composition of the samples was determined by classical silicate analysis and the results revealed presence of the following oxides (wt%): SiO_2 (90.17), Al_2O_3 (1.79), Fe_2O_3 (0.328), MnO (0.0004), TiO_2 (0.055), CaO (0.132), MgO (0.20), K2O (0.52), Na_2O (0.25), and LOI (6.15). Results from the X-ray diffraction examination enabled to determine presence of predominant tridymite crystalline phase, associated with quartz and muscovite. The IR spectrum of the naturally occurring tridymite exhibits absorption bands at 791 cm^{-1} and 1100 cm^{-1} ascribed to the stretching Si–O–Si vibrations. The band at 3650 and 1640 cm^{-1} are assigned to the stretching and bending vibrations from the absorbed water, respectively. Characteristics hexagonal crystals of tridymite and globular forms present in the raw material were found from the SEM investigations.

Keywords: natural tridymite, XRPD, FTIR, SEM

EXPERIMENTAL:

The naturally occurring tridymite that is subject of research in this paper, is taken from vicinity of Alshar Mountain (Kavadarci, Republic of Macedonia).
The chemical composition of tridymite is determined with the classical silicate analysis, whereas the microelements are determined with ICP (ICP-MS Elan DRC II, PerkinElmer, USA). The results of the chemical composition are shown on Table 1. The mineralogical investigation was further analysed by an X-ray powder diffraction (XRPD), thermal analysis (TGA/DTA), scanning electron microscopy (SEM), optical microscopy and Infrared spectroscopy (IR).
XRPD analysis was performed on Rigaku Ultima IV X-ray diffractometer equipped with D/teX high-speed 1-dimnesional detector using CuKα radiation (λ = 1.54056 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 tridymite were performed in air environment with Stanton Redcroft apparatus, under the following experimental conditions: temperature range from 20–1100 °C; speed of heating set to 20 °C/min; sample mass of 67.389 mg; and ceramic pot as a material carrier.
The optical microscopy is performed with transmission polarizing microscope SM-POL, Leitz, Wetzlar, Germany.
Scanning electron microscopy (SEM) of tridymite was performed with energy dispersive X-ray spectroscopy (EDX) employing FEI Quanta 3D FEG dual beam microscope.
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.

Table 1. Chemical composition of naturally occurring tridymite

Oxides	Mass %
SiO_2	90.17
Al_2O_3	1.79
Fe_2O_3	0.328
MnO	0.004
TiO_2	0.055
CaO	0.132
MgO	0.208
K_2O	0.526
Na_2O	0.254
LOI	6.156
Total	99.63

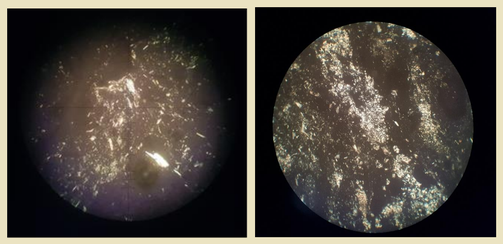

Figure 1. Optical microscopy of naturally occurring tridymite

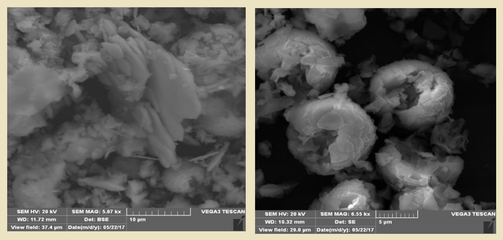

Figure 2. Scanning electron microscopy of naturally occurring tridymite

RESULTS AND DISCUSSION:

Macroscopic examination
The tested samples represent a pretty loose, soft, weak rock with white to greyish colour that resembles diatomaceous earth. The samples seem very light and can be easily disintegrated by applying pressure to it. In Fig. 1 is shown a macroscopic view of the naturally occurring tridymite.

Scanning electron microscopy
The results from the scanning electron microscopy (Fig 2) show the characteristics hexagonal crystals of tridymite (a) and globular forms present in the raw material (b).

X-ray powder analysis of the naturally occurring tridymite
Results of the XRPD analysis of the tridymite (Fig. 3) depicts crystalline behaviour of the sample manifested by the appearance of peaks characteristic for tridymite (d 4.11 at 21.60°, d 4.31 at 20.54° and d 2.50 at 35.81 (2θ). Evident is presence of muscovite as well as presence of the amorphous phase distributed between 18 and 30° (2θ), which is as result of the amorphous phase present in the raw material.

Infra-red analysis of the naturally occurring tridymite
The IR spectrum of the naturally occurring tridymite (Fig. 4) exhibits absorption bands at 791 cm^{-1} and 1100 cm^{-1} are due to the stretching vibrations of Si-O-Si band. The band at 1640 cm^{-1} is due to stretching and bending vibrations from the absorbed water, while the band at 3650 cm^{-1} is due to the combination of OH stretching. The infra-red spectrum of natural occurring tridymite is shown in Fig. 3.

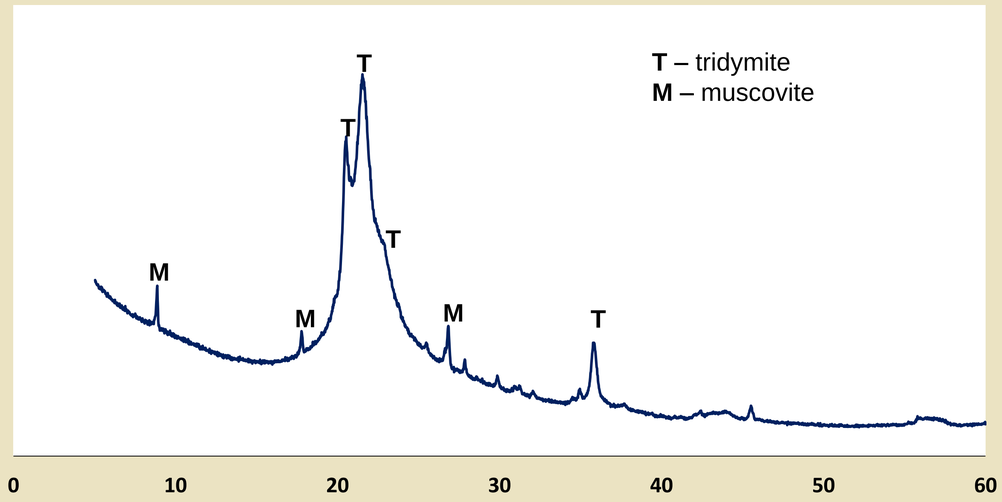

Figure 3. XRPD of naturally occurring tridymite

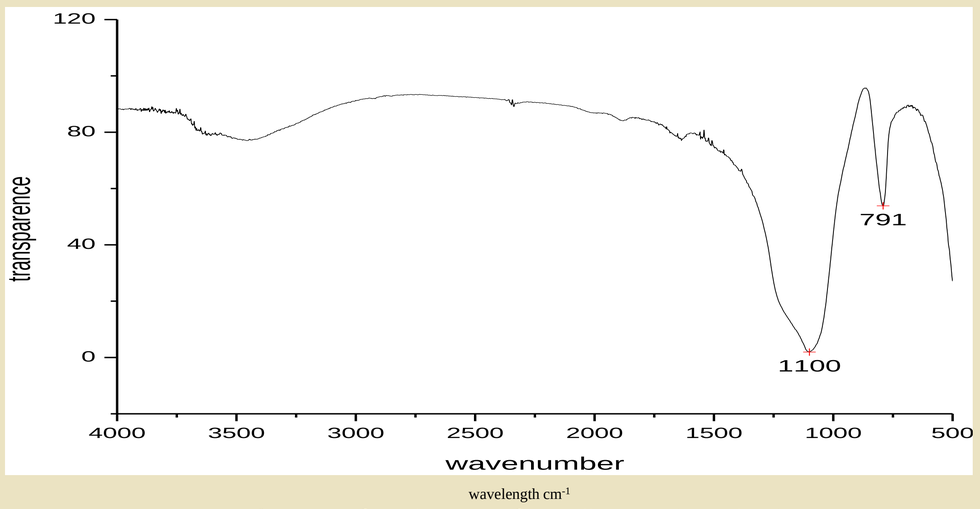

Figure 4. Infra-red spectra of naturally occurring tridymite

CONCLUSION:

Based on the above-mentioned results it can be concluded that the raw material, especially from the x-ray results it represents a tridymitic raw material with premises of muscovite. The results form the scanning microscopy show presence of hexagonal crystals (which crystals are characteristic for tridymite) as well as globular forms (which are most likely opal).