American Mineralogist, Volume 104, pages 1323–1335, 2019

Identification of the occurrence of minor elements in the structure of diatomaceous opal using FIB and TEM-EDS

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Abstract

The occurrence of minor elements in the structure of biogenic diatomaceous opal-A is an important issue because it is closely related to biogeochemical processes driven by the precipitation, sedimentation, and storage of diatoms, as well as to the properties and applications of diatomite, which is the sedimentary rock composed of diatomaceous opal-A. However, to date, there is no direct microscopic evidence for the existence of minor elements, such as Al, Fe, and Mg, in the structure of diatomaceous opal-A, because such evidence requires observation of the internal structure of frustules to exclude the disturbance of impurity minerals, which is technically challenging using conventional techniques. In this work, transmission electron microscopy (TEM) and scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDS) mapping analysis were performed on diatomaceous opal-A from three typical diatomite specimens that were pretreated using focused ion beam (FIB) thinning. This technique produces a slice of a diatom frustule for direct TEM observation of the internal structure of the diatomaceous opal-A. The results of this work clearly indicate that minor elements, such as Al, Fe, Ca, and Mg, conclusively exist within the siliceous framework of diatomaceous opal-A. The contents of these minor elements are at atomic ratio levels of 1 (minor element)/10 000 (Si) – 1/100, regardless of the genus of the diatoms. The occurrence of minor elements in the internal structure is likely through biological uptake during biosynthesis by living diatoms. Moreover, surface coatings composed of aluminosilicates on diatom frustules are common, and the contents of elements such as Al and Fe are tens or hundreds of times higher in the coatings than in the internal siliceous structure of diatomaceous opal-A. The discovery of the incorporation of the above-mentioned minor elements in the diatomaceous opal-A structure, both in the internal Si-O framework and on the surface, updates the knowledge about the properties of diatomite.

Keywords: Diatomaceous opal-A, diatomite, focused ion beam (FIB), transmission electron microscopy (TEM), minor elements