Influence of diatomite microstructure on its adsorption capacity for Pb(II)

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Since the contamination of ground and surface water with heavy metals is becoming a major concern, it is very important to find the suitable medium for their adsorption. As a low cost material, natural diatomite has been proposed as a potential adsorbent for the removal of heavy metals. Diatomite ($SiO_2 \cdot nH_2O$) is pall-colored, soft, lightweight sedimentary rock composed principally of silica microfossils of aquatic unicellular algae. It is a highly porous structure, chemically inert, with low density and high surface area [1]. Recently, the use of diatomite as possible additive for improvement of hydrogen storage properties of MgH_2 has been reported [2].

In the present work, remediation of lead containing solution using raw and diatomite modified by ball milling has been presented. Diatomite samples were processed by ball milling in a Turbula Type T2C at the standard milling frequency of 870 rpm, in the air atmosphere, using ball to powder ratio (BRP) 4, for different time intervals (from 1 do 5 hours). The effect of surface modification of diatomite by ball milling on Pb(II) adsorption and on filtration quality was investigated. The microstructure and surface properties of the raw diatomite and treated one have been characterized by scanning electron microscopy (SEM, JEOL JSM 6460LV and Oxford Instrument INCA-X-sight at 25kV) and atomic force microscopy (AFM, on Veeco MultiMode Quadrex IIIe in the tapping mode). Degree of Pb(II) metal adsorption was obtained using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP AES model Spectro CIROS vision, Kleve, Germany).

Fig.1 represents the micrographs of raw diatomite (Fig.1a) and treated one (Fig. 1b) with magnification in the order of 2000. Diatomite frustules are mainly divided into two categories; centric (discoid) and pennate (elongated to filiform). The length of the pennate shape is in the range form 10-20 μm, while the centric diatom has a radius of approximately 20 μm. It can be noticed from the scanning micrographs that diatomite has a highly porous structure which was the main reasons to select this material as a potential adsorbent for heavy metals. The porous texture of diatomite is not completely damaged after ball milling. Cylindraceous diatomite (centric) particles are still present in the sample, while rode-like (pennet) particles are partially pulverized during milling. AFM phase analysis of pure diatomite have shown very clear differentiation of sample phase composition. Sponge like structure with small pore sizes can be noticed too. The hexagonal structure of oxide unit cell is clearly visible. The average diameter of single hexagon is 60 nm. These cells are close, making a terrace structure of each hexagon with randomly distributed Si and Al oxides (Fig.2). In the Fig 3. AFM 3D surface topography of ball milled diatomite samples for 5 hours is presented. One can notice the major structural topography changes caused by milling. Hexagonal structure is disappeared and agglomerates with a sponge-like structure appear. Diatomite has become denser and compact. During the ball milling process diatomite structure has been partially destroyed and effective diameter of pore is decreased. Phase

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distribution has been changed also. This can suggest that the real chemical composition is changed during milling. Anyhow, XRD analysis (not shown in this paper) did not show the new phases, so one can conclude that changes are due to amorphization, which can lead to better adsorption properties of the sample. Similar results are obtained with SEM analysis. All samples have been subjected to sorption analysis. It was noticed that the quantity of adsorbed Pb(II) ions increase if the milling time rising and arrive to saturation at 4 hours. This can be ascribed to microstructural changes emerged after ball milling, such as appearance of mesoporous sponge-like structure. Similar results were obtained taking into account immobilization efficiency which increases from 22% for untreated samples to 81% to treated sample for 5h at BPR 4.

The presented results show the strong correlation between microstructure and immobilization efficiency and/or concentration of adsorbed Pb(II). Metal sorption capacity and immobilization efficiency of diatomite are considerably improved after mechanical modification. This improvement in diatomite performance is attributed to amorphization of the material.

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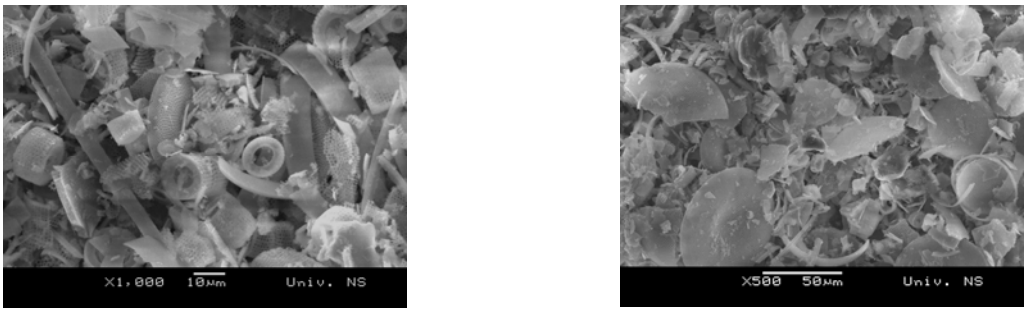

(a) (b)

Fig.1. SEM images of pure diatomite: (a) and mechanical treated (b)

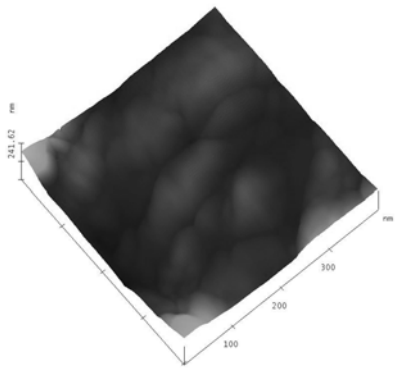

Fig. 2. AFM 3D surface topography of pure diatomite

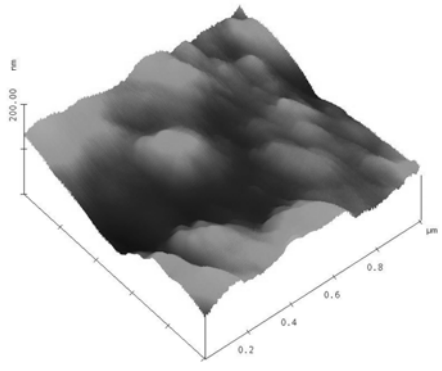

Fig. 3. AFM 3D surface topography of 5 hours milled diatomite