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Angaben zur Veröffentlichung / Publication details:

Höllén, Daniel, P. Grunert, D. Klammer, and Martin Dietzel. 2011. "Hydrothermal alteration of diatomite for the fixation of heavy metal ions [Abstract]." *Mineralogical Magazine* 75: 1038. <https://doi.org/10.1180/S0026461X00006502>.

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Hydrothermal alteration of diatomite for the fixation of heavy metal ions

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The application of altered diatomite as a hierarchically structured material for ion fixation from aqueous solutions is based on macropores (~100 nm) from the intricate silica structure of diatomite, micropores (~0.3-0.4 nm) from neoformed zeolites and highly active surface sites from amorphous precursor phases. Such kind of materials can be obtained by hydrothermal synthesis.

A stock solution was prepared by dissolving 1 g of gibbsite ($\gamma\text{-Al(OH)}_3$) in 0.5 L of 1 M KOH and subsequent filtration (0.45 μm). 0.025 L of the stock solution reacted with 0.5 g diatomite ("Thiele") in a Teflon coated autoclave at 50 to 150°C. After distinct reaction times from 6 up to 1500 h reaction products were separated from the solutions by filtration. Solutions were analysed for composition and solids for composition and structure. Ion fixation capacities of the reaction products were studied in aqueous solutions containing 0.5 mM of Cu^{2+} , Pb^{2+} and Zn^{2+} at pH 5.1 and 25°C for 72 h.

Initially, the diatoms are converted partly into spherical amorphous nanoparticles (20-200 nm). After 24 to 192 h mainly merlinoite and minor amounts of chabazite were formed. Proportions depend on temperature and reaction time. At a reaction time between 96 and 192 h a quasi steady state was reached with ≈ 30 wt.% amorphous phase, ≈ 60 wt.% merlinoite and 10 wt.% accessory phases (relictic quartz, mica as well as newly formed chabazite). The heavy metal ion fixation capacity of the pristine diatomite is low. Interestingly, the reaction product containing the amorphous precursor phase non-selectively reduced ≥ 99 % of the primarily dissolved heavy metals (specific surface area = 38 m^2/g). The final reaction products (15 m^2/g) reduced heavy metal concentrations less efficiently (~ 90 %), but selectively. Challenging tasks for the ongoing study are to tailor the reaction products with regard to metal fixation and hierarchical pore structures for waste and drinking water purification.