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Formation and properties of inorganic Si-contaminant compounds

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Soil contamination with inorganic contaminants such as lead (Pb), copper (Cu) and cadmium (Cd) is a major environmental issue, especially concerning food and groundwater security. Various studies demonstrated positive effects of Si regarding resilience of some crops towards these inorganic contaminants. One reason could be a complexation reaction of Si and the metal cations. However, this process has not been systematically investigated yet. Thus, our research contributes to reducing the mobility of Cd, Cu and Pb in contaminated soils and to decreasing their transfer into aquifers or plants.

The main goal of this study is to elucidate the extent and the mechanisms of the interactions between Pb^{2+} , Cd^{2+} and Cu^{2+} and silicic acid, including the long-term kinetics, and to investigate whether the metals are bound by silicic acid. We carried out a series of precipitation experiments in aqueous solution at room temperature to understand these processes.

We used Tetraethoxysilane (TEOS) as Si source and $\text{Pb}(\text{NO}_3)_2$, $\text{Cd}(\text{NO}_3)_2$ and $\text{Cu}(\text{NO}_3)_2$ with an initial concentration of 10 mmol l^{-1} for synthesis. Selectivity of Si towards the metals was tested in an equimolar solution of all three salts and TEOS. Time-dependency of particle growth was examined at sixteen different dates using dynamic light scattering (DLS) and transmission electron microscopy (TEM). We measured the Si and metal concentrations in the dialyzed aliquots using microwave plasma-atomic emission spectrometry (MP-AES). Spectroscopic analysis of the dialyzed and freeze dried solid phase, was performed using FTIR and ^{29}Si -NMR spectroscopy.

DLS and TEM analyses showed that the metals had an accelerating effect on the polymerization reaction of silicic acid [$\text{Cu}^{2+} > (\text{Cu}^{2+}, \text{Pb}^{2+}, \text{Cd}^{2+}) > \text{Cd}^{2+} > \text{Pb}^{2+}$]. Particle growth followed initial formation of nanoparticles through homogenous nucleation. Particle growth in the control synthesis (TEOS in aqueous solution) stopped after 124 days at a size of 34 nm (Z-Average). Particles in the syntheses with the metals kept growing until the experiment was completed after 211 days. The final particle sizes depended on the metal present, reaching a final size of 260 nm (Cu), 96 nm (Pb) and 196 nm (Cd). Final concentrations of up to 15, 10 and $13 \mu\text{mol l}^{-1}$ of Cu, Pb and Cd, respectively, remained in the dialyzed aliquots. The Si concentrations in these aliquots increased continuously until an equilibrium was reached after 112 days at different concentrations (Cu, 7.3 mmol l^{-1} ; Pb, 6.9 mmol l^{-1} ; Cd, 4.8 mmol l^{-1}). The FTIR spectra showed a shift of the Si-O stretching vibration by 10 to 32 cm^{-1} towards lower wavenumbers, which could indicate an

incorporation of the metals in the polymeric network of the silicic acid. ^{29}Si -NMR relaxation experiments showed a shortening effect of Cu^{2+} -ions on the relaxation time of the Si nuclei. It appears that the proportion of the rapidly relaxing components decreases for the Si-atoms deep inside the silicate matrix. This indicates that the Cu centres are located predominantly at the huge surfaces (up to $667 \text{ m}^2 \text{ g}^{-1}$) of the Si matrix. Future extraction experiments will show how strong the metals are bound to the Si polymeric network.