



Interaction of dissolved As(V) with gypsum: surface precipitation of guerinite, sainfeldite and $\text{NaCa}_2\text{H}(\text{AsO}_4)_2 \cdot 6\text{H}_2\text{O}$, a new arsenate.

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The interaction of Na_2HAsO_4 aqueous solutions with gypsum is studied, in order to check the effectiveness of this mineral in removing arsenic from water within a specific pH range. Sorption experiments were carried out at $25 \pm 0.1^\circ\text{C}$ and atmospheric pressure by reacting gypsum grains (1.0-1.5 mm) with Na_2HAsO_4 aqueous solutions in continuously stirred closed vessels during 50 days. Both pH and concentration were monitored at predetermined moments. Concentration of dissolved As was measured by ICP-OES, while Na and Ca were analysed via AAS. Representative individuals of the gypsum crystals were selected from the aqueous solution to verify the incorporation of arsenic-bearing phases on their surfaces via examination through SEM and powder and single crystal XRD. In all experiments an important decrease in arsenic concentration has been measured during the first 24 hrs, decreasing then towards an asymptotic value. The precipitate consists of aggregates of small ($\sim 30 \mu\text{m}$) crystals of guerinite with laminar morphologies that cover all the surface of the gypsum substrate after 24 hrs of reaction at high concentrations. A detailed observation of the precipitate shows crystals of guerinite and sainfeldite, with laminar and prismatic shapes, respectively. In the highest parent solution experiments a precipitate of crystals with an unknown diffraction pattern was initially formed. A suitable crystal ($\sim 0.15 \text{ mm}$) was carefully detached from the substrate surface and studied by single crystal XRD. The solved structure shows that this compound $[\text{Ca}_2\text{NaH}(\text{AsO}_4)_2 \cdot 6\text{H}_2\text{O}]$ crystallises in the triclinic system (P-1; $a=6.680 \text{ \AA}$, $b=8.223 \text{ \AA}$, $c=12.537 \text{ \AA}$, $\alpha = 73.464^\circ$, $\beta = 78.890^\circ$, $\gamma = 87.471^\circ$) with two formula per unit cell.