

Supplementary Materials: Comparing Schwertmannite and Hydrobasaluminite Dissolution in Ammonium Oxalate (pH 3.0): Implications for Metal Speciation Studies by Sequential Extraction

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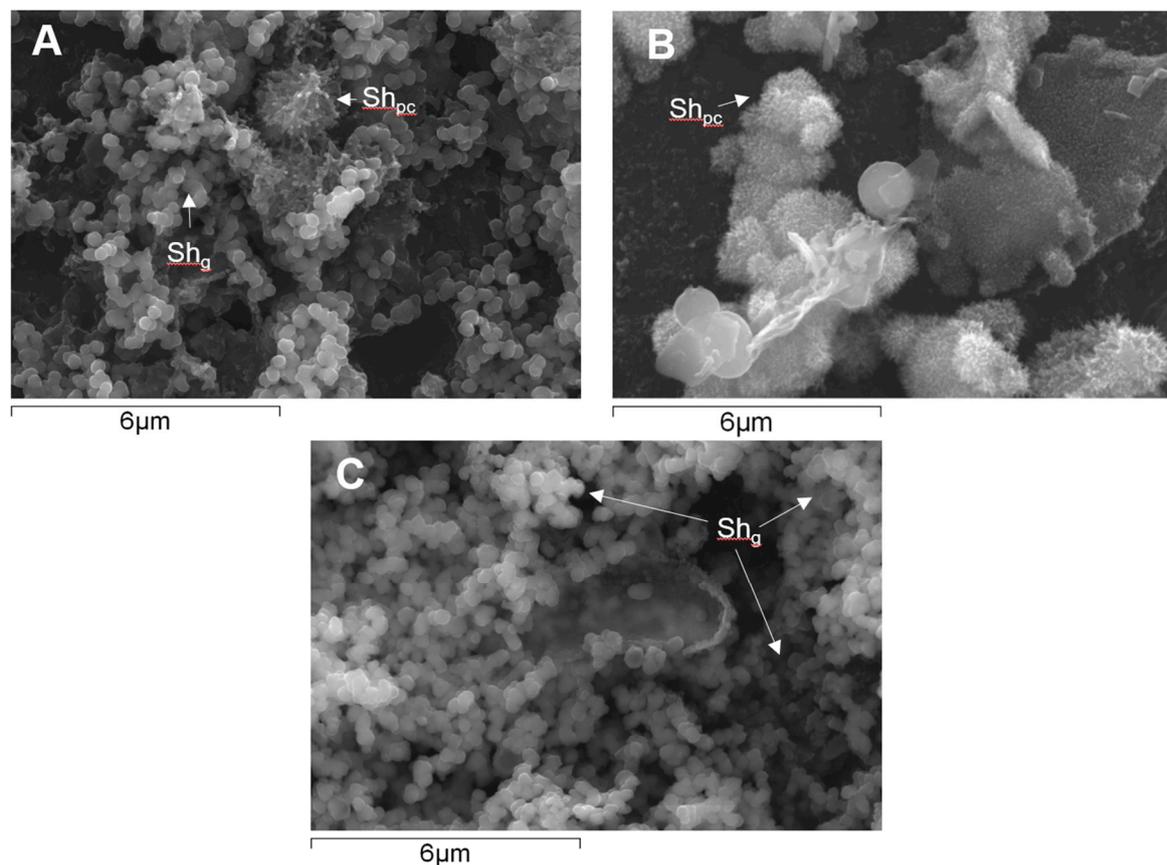


Figure S1. SEM pictures of different schwertmannite samples from the studied sites: (A) Natural schwertmannite formed by Fe(II) oxidation in Herrerías mine pit lake (note difference between *pin-cushion*-like (Sh_{pc}) and globular (Sh_g) schwertmannite); (B) Natural, *pin-cushion*-like schwertmannite formed by Fe(II) oxidation in acidic water sampled from a deeper layer in the same mine site; (C) Synthetic schwertmannite particles formed in the laboratory (note similarity of particle morphology and size with (A)).

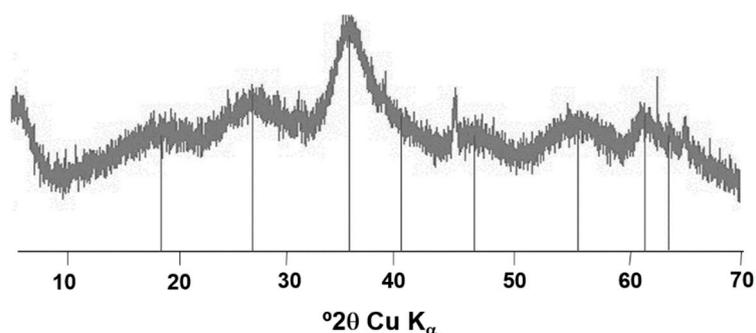


Figure S2. XRD pattern obtained for synthetic schwertmannite obtained by neutralization of acidic, Fe(III)-rich water from La Zarza mine, and later used for the dissolution experiments in ammonium oxalate at pH 3.0. The vertical lines indicate the positions of the reflections typical of this mineral.

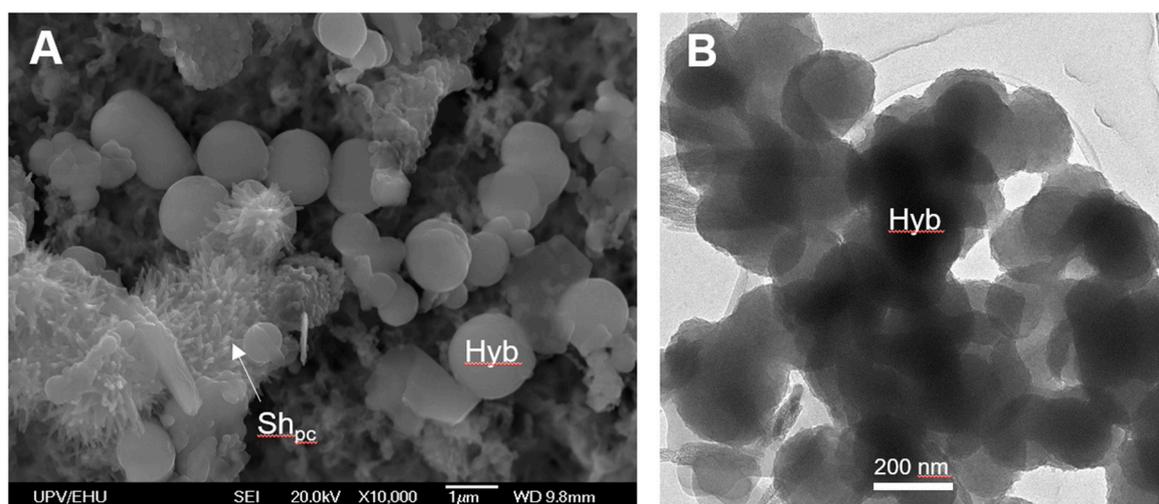


Figure S3. (A) SEM image of globular hydrobasaluminite particles (Hyb) formed naturally in Herrerías mine pit lake at depths of 45 m; this mineral coexists with *pin-cushion*-like schwertmannite (reprinted from [1], with kind permission by the Spanish Mineralogical Society); (B) TEM picture of hydrobasaluminite globules formed by Al(III) precipitation in water from Herrerías mine.

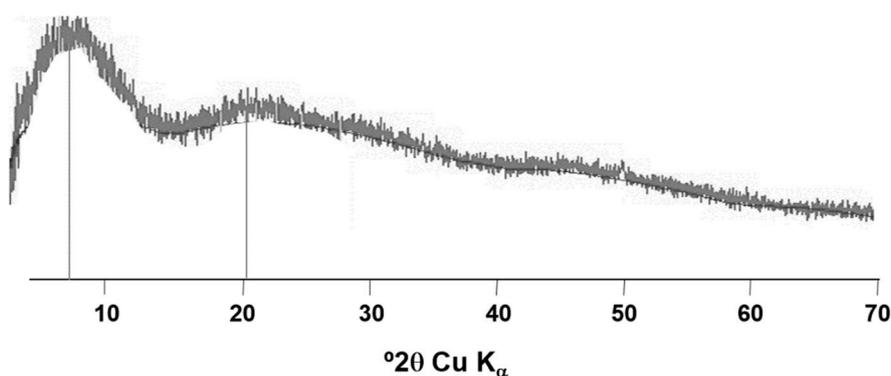
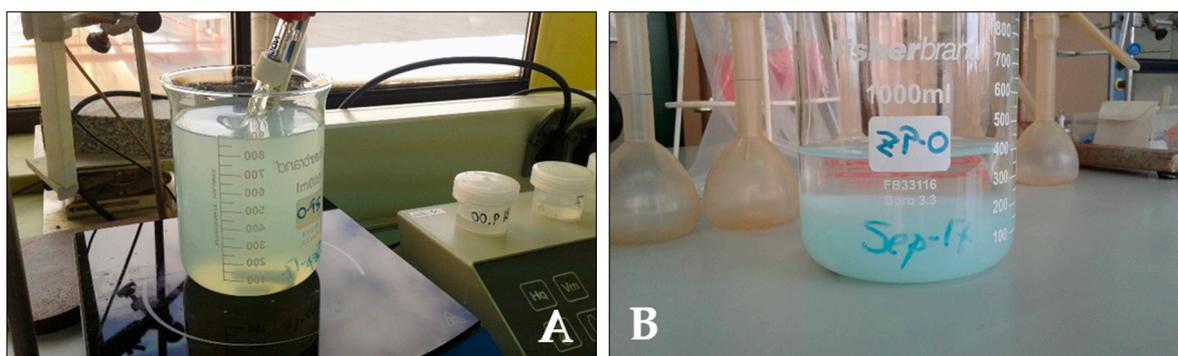


Figure S4. XRD pattern obtained for synthetic hydrobasaluminite obtained by neutralization of acidic, Al-rich water from La Zarza mine, and later used for the dissolution experiments in ammonium oxalate at pH 3.0. The vertical lines indicate the positions of the two main hump peaks typical of this poorly crystalline mineral.



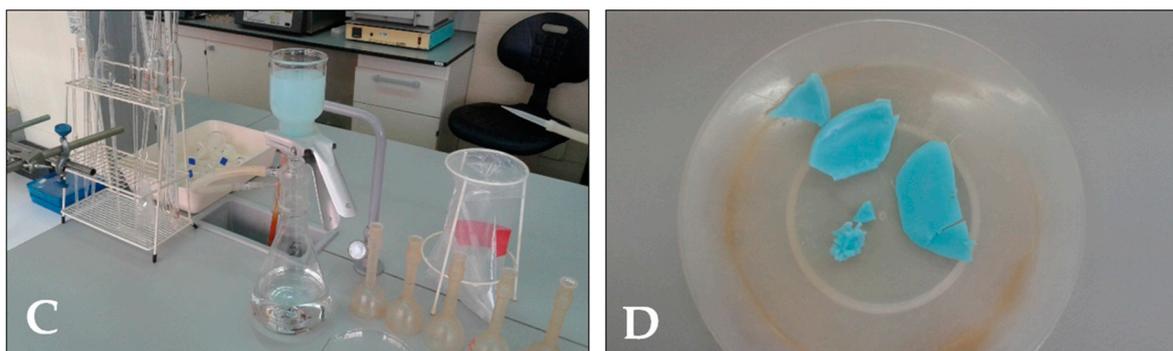


Figure S5. Sequence of pictures showing the synthesis of one of the hydrobasaluminite precipitates used for the oxalate dissolution experiments (Hyb 2 obtained from La Zarza acidic mine water). (A) colloidal hydrobasaluminite suspension formed in the acidic solution during partial neutralization to pH 5.0 with 1 M NaOH. (B) Aspect of the suspension settled in the bottom of the glass beaker. (C) vacuum-filtration of the suspension, showing a characteristic blueish color diagnostic of a high Cu content of the precipitate. (D) fragments of the Cu-rich aluminum oxyhydroxysulfate precipitate after filtration.



Figure S6. Final aspect of the oxalate solutions after the end of the experiments; note the color contrast between the transparent solution after dissolution of hydrobasaluminite from Herrerías mine (left) and the blueish solution after dissolution of hydrobasaluminite from La Zarza mine (right).

References

1. Sánchez-España, J. Crystallization in acidic media: from nanoparticles to macrocrystals. In *Crystallization under extreme conditions, Proceedings of the Seminarios de la Sociedad Española de Mineralogía, Oviedo, 4 July 2017*; Sociedad Española de Mineralogía: Madrid, 2017; pp. 15–34.