Electronic Supplementary Material (ESI) for CrystEngComm.

Tracking the Transformations of Mesoporous Microspheres of Calcium Silicate Hydrate in Nanoscale upon Ibuprofen Release: An XANES and STXM Study

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1. TEM images of mesoporous CSH microspheres

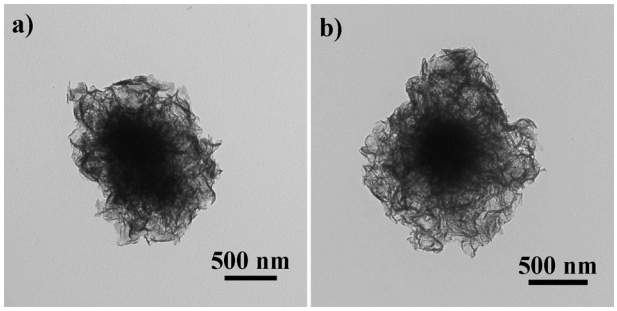


Figure S1. TEM images of mesoporous CSH microspheres before (a) and after (b) IBU loading.

2. XANES linear combination fitting (LCF) at the Ca K-edge and the P K-edge

ATHENA is a software package that has a capability for fitting an experimental XANES as a linear combination of standard spectra. This analysis could help interpret the kinetics of a series of spectra measured during a chemical reaction. By fitting each intermediate spectrum as a linear combination of the different standard spectra, one can determine the species and the relative amount of standards in a heterogeneous sample.

The analysis is based on a non-linear, least-squares minimization procedure. The parameter chi-square tends to be very small numbers -- much smaller than 1.

The R-factor reported in the text box is:

$$R = \frac{Sum\ ((data - fit)^2)}{Sum\ (data^2)}$$

where the sums are over the data points in the fitting region.

Interpretation of the statistical parameters requires the knowledge about the system which is measuring. The results of sample fractions must be meaningful in the context of any external knowledge about the system. More information can be found at http://cars9.uchicago.edu/~ravel/software/doc/Athena/mobile/analysis/lcf.html.

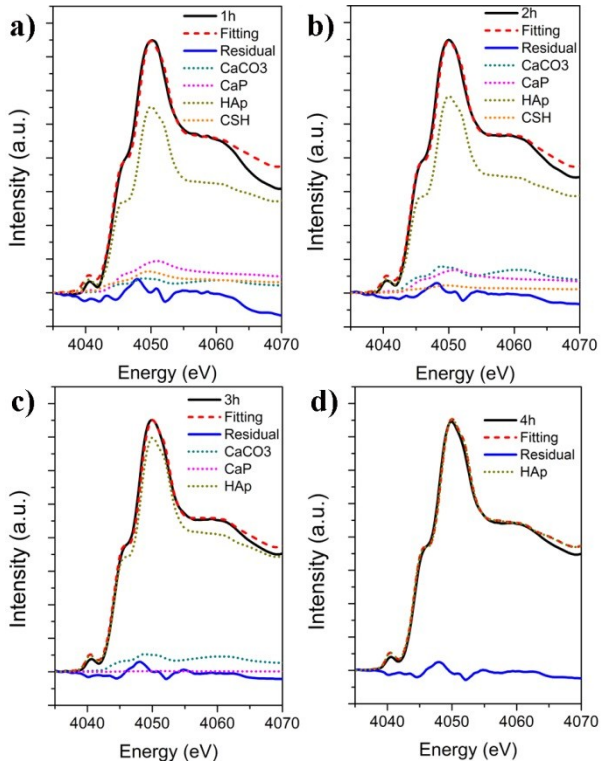


Figure S2. Comparison of Ca K-edge XANES spectra and linear combination fitting in the first 4 hours.

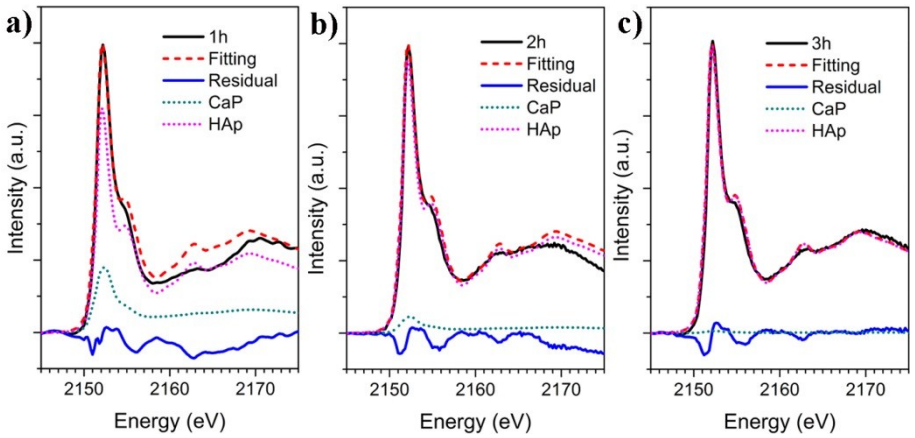


Figure S3. Comparison of P K-edge XANES spectra and linear combination fitting in the first 3 hours.

3. Fluorescence yield of biomineralization of CSH-IBU microspheres at the Si K-edge

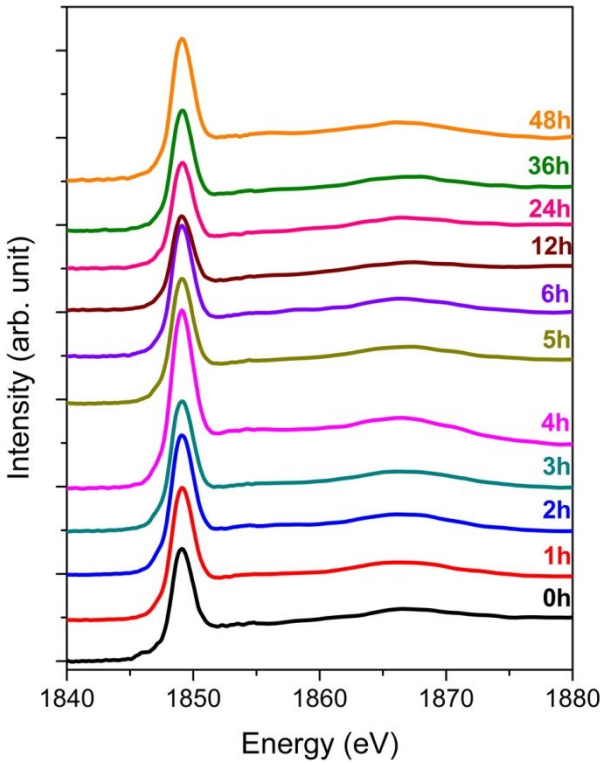


Figure S4. Si K-edge FLY XANES spectra of CSH-IBU microspheres soaked in SBF solution for various time periods.

4. Changes of Ca, Si and P during the biomineralization.

Figure S6 shows the change in concentration of Ca, Si and P in the SBF solution measured by ICP after soaking CSH-IBU microspheres in SBF for different time periods. At early stages, the Si concentrations in SBF increased when the IBU was released from CSH microspheres, indicating the dissolutions of CSH microspheres in the SBF solution. Meanwhile, there was a steep decrease of P concentration in the first 6 h of soaking, corresponding to the phosphate consumption in the SBF solution in order to form HAp on the surface of CSH microspheres. It should be noted that there is also a rapid drop of Ca concentrations in the first 6h of soaking and the concentration remained almost steady after 1 day. This observation is different from the Ca change in the previous studies.[1-4] The reason why the rate is different is that the Ca dissolution rate of CSH microspheres is slower than the Ca precipitation rate from the SBF solution (the co-precipitation of Ca^{2+} and PO_4^{3-} from the SBF solution to form HAp on the surface of drug carriers).

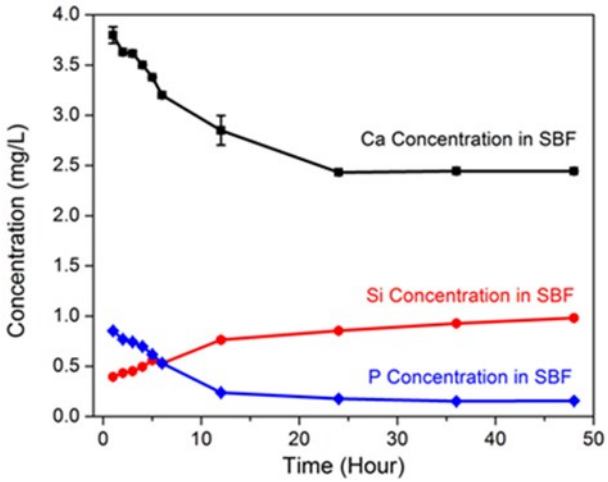
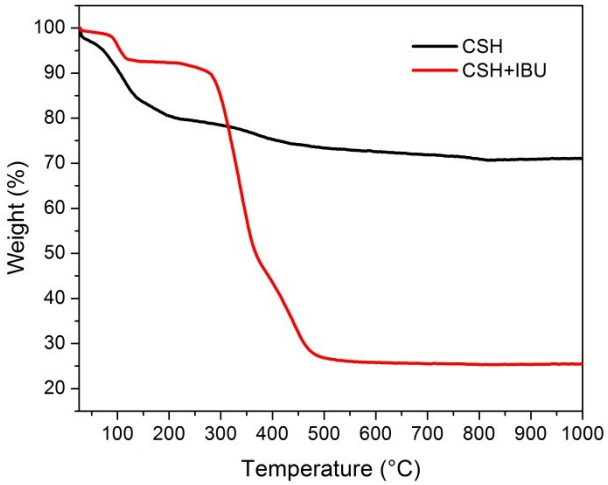


Figure S5. Concentration changes of Ca, P and Si after soaking CSH-IBU in SBF for different period of time.

5. IBU Loading Capacities Test in CSH microspheres:

To measure the drug loading capacity of CSH microspheres, the TG analysis of CSH and CSH loaded with IBU were carried out. As shown in Figure S7, there is a two-level-step profile for the weight loss of the CSH-IBU system; the weight losses at 80-100 °C and 280-480 °C originate from the evaporation of water and the clean burn of IBU in air, respectively. The drug loading capacity (DLC) (m_{IBU}/m_{CSH} wt.%) of CSH microsphere is calculated based on the TG curves, showing a high value of 180 wt.% (i.e. 1.80 g IBU is loaded per gram of carriers), which agrees with our precious studies.



Figures S6. TG curves of mesoporous CSH microspheres and CSH loaded with IBU.

1. P. Siriphannon, Y. Kameshima, A. Yasumori, K. Okada and S. Hayashi, *J. Eur .Ceram. Soc.*, 2002, 22, 511-520.
2. Z. Gou and J. Chang, *J. Eur. Ceram .Soc.*, 2004, 24, 93-99.
3. X. H. Wan, C. K. Chang, D. L. Mao, L. Jiang and M. Li, *Mat. Sci. Eng. C*, 2005, 25, 455-461.

4. X. Li, J. Shi, Y. Zhu, W. Shen, H. Li, J. Liang and J. Gao, *J. Biomed. Materi. Res. B Appl. Biomater.*, 2007, 83B, 431-439.