Supplementary Materials: Cyclodextrin-Driven Formation of Double Six-Ring (D6R) Silicate Cage: NMR Spectroscopic Characterization from Solution to Crystal Phase

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1. FT-IR and TGA of $K_{12}Si_{12}O_{30}$•2α-CD•$36H_2O$

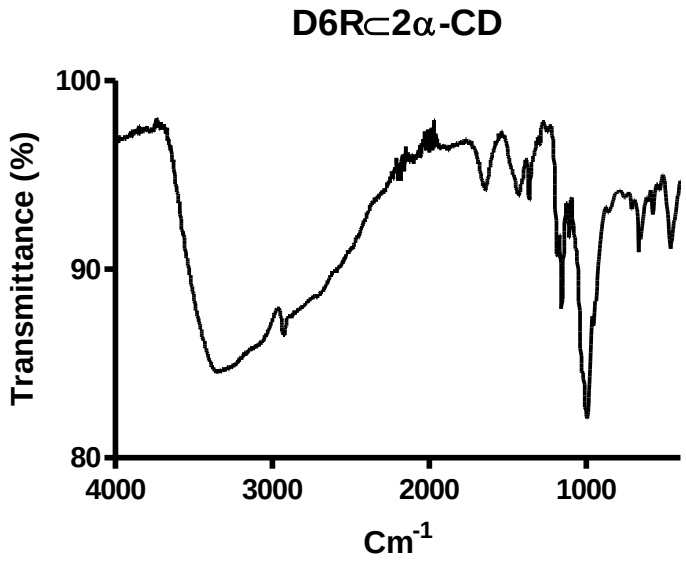

Figure S1. FT-IR spectrum of $K_{12}Si_{12}O_{30}$•2-CD•$36H_2O$.

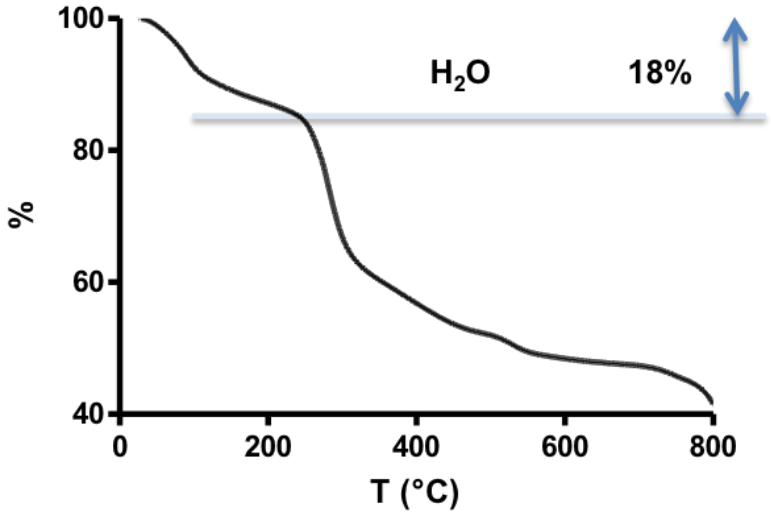

Figure S2. TGA curve of $K_{12}Si_{12}O_{30}$•2-CD•$36H_2O$.

2. Crystallographic structure of $K_{12}Si_{12}O_{30}$•2α-CD

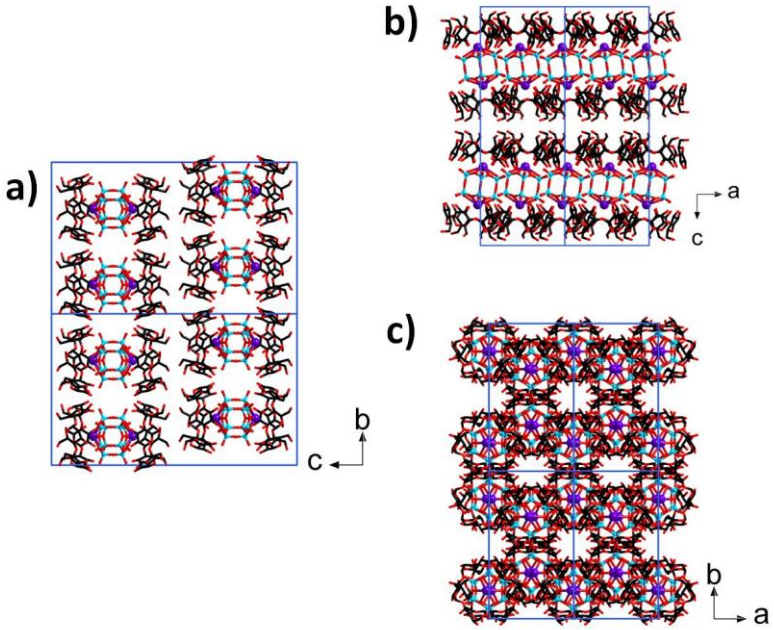

Figure S3. Crystallographic views of $K_{12}Si_{12}O_{30}$•2α-CD along (**a**) [100], (**b**) [010], and (**c**) [001].

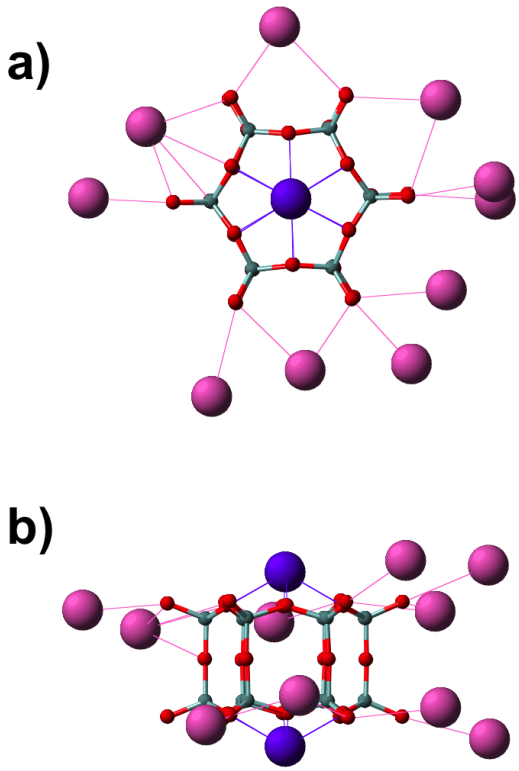

Figure S4. Disposition of K around the silicate cage in the triclinic form of $K_{12}Si_{12}O_{30}$•2α-CD. Top (**a**) and side (**b**) views with respect to the D_6 symmetry axis of the prismatic unit.

3. Effect of KCl on ^{1}H NMR of synthesis medium of D6R@α-CD

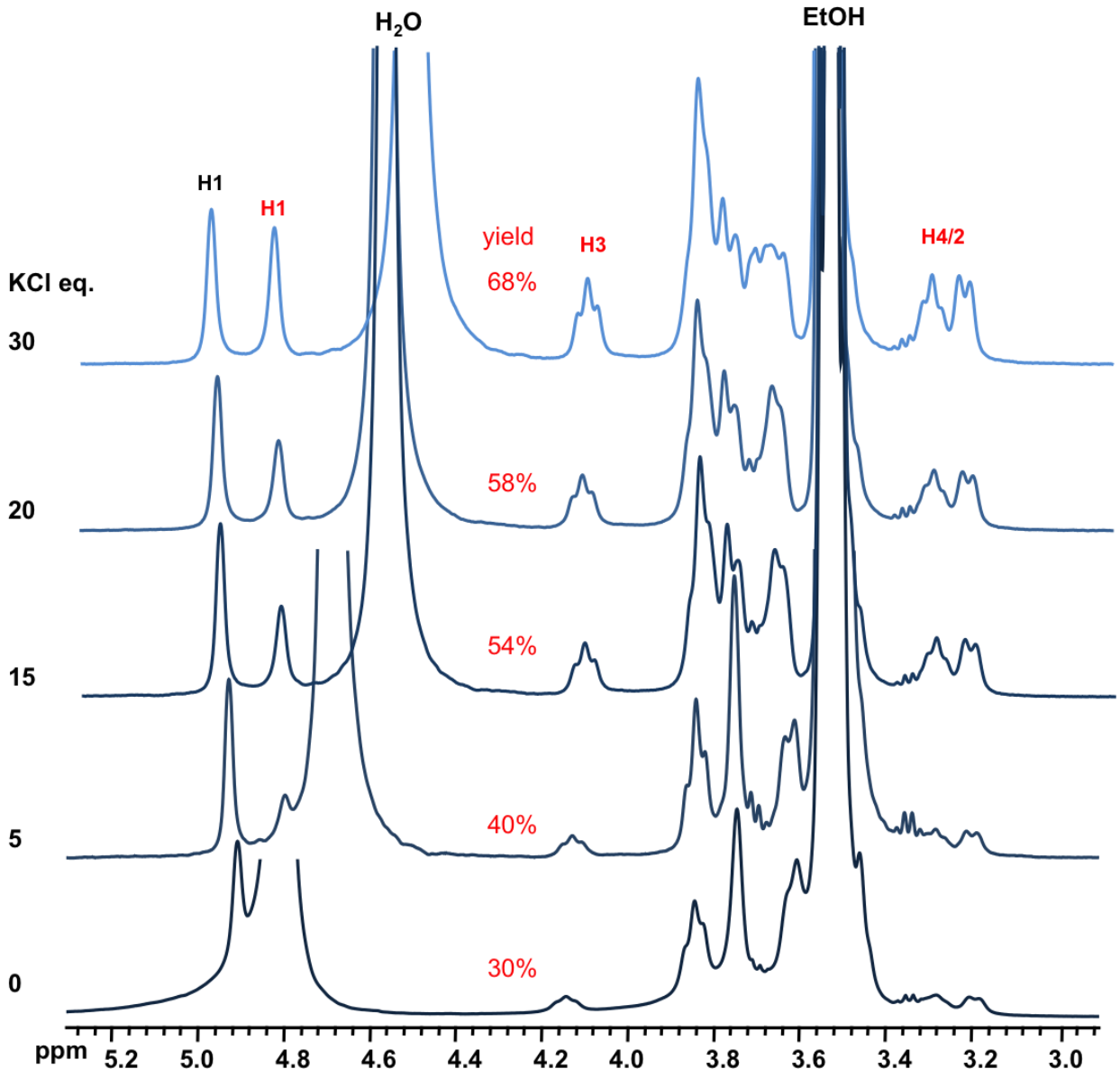

Figure S5. ^{1}H NMR spectra of the synthesis medium in the system 1 α-CD : 6 TEOS : 6 KOH : 700 D_2O : *x* KCl, *x* = 0, 5, 15, 20, and 30. The solutions were equilibrated for at least one week.

4. Effect of temperature on D6R@α-CD stability

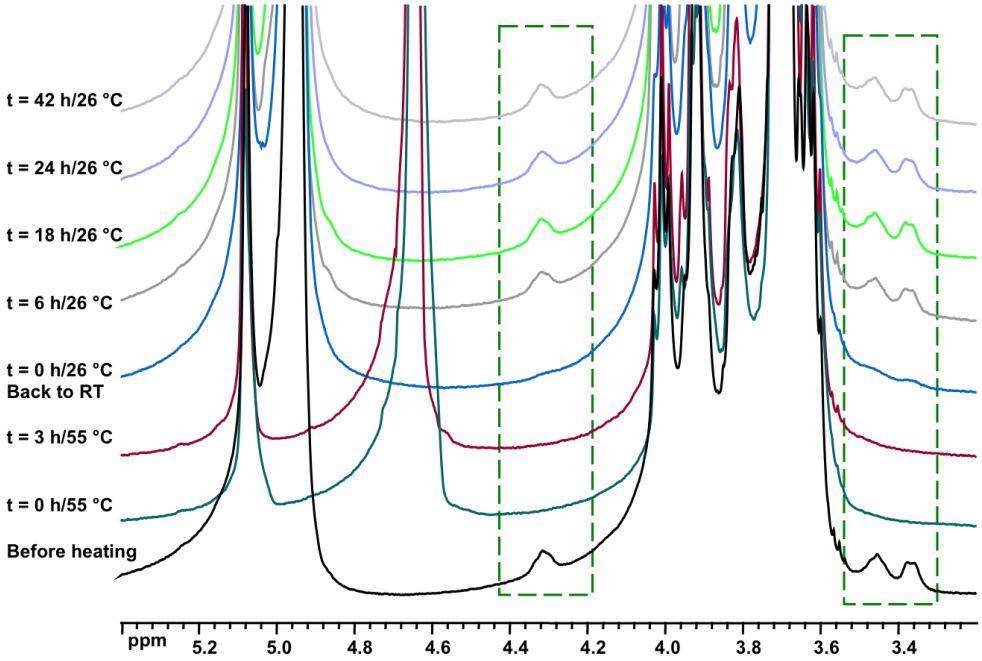

Figure S6. Variable temperature ^{1}H NMR spectra of equilibrated solution in the system 1 α-CD : 6 TEOS : 6 KOH : 400 D_2O.

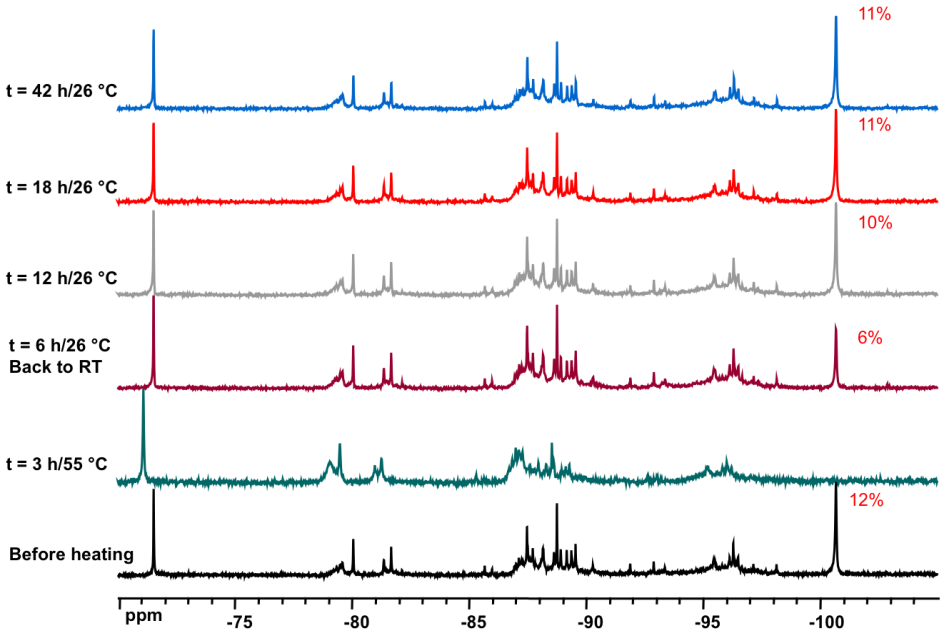

Figure S7. Variable temperature ^{29}Si NMR spectra of equilibrated solution in the system 1 α-CD: 6 TEOS: 6 KOH: 400 D_2O.

5. ^{13}C CPMAS NMR of D6R@α-CD

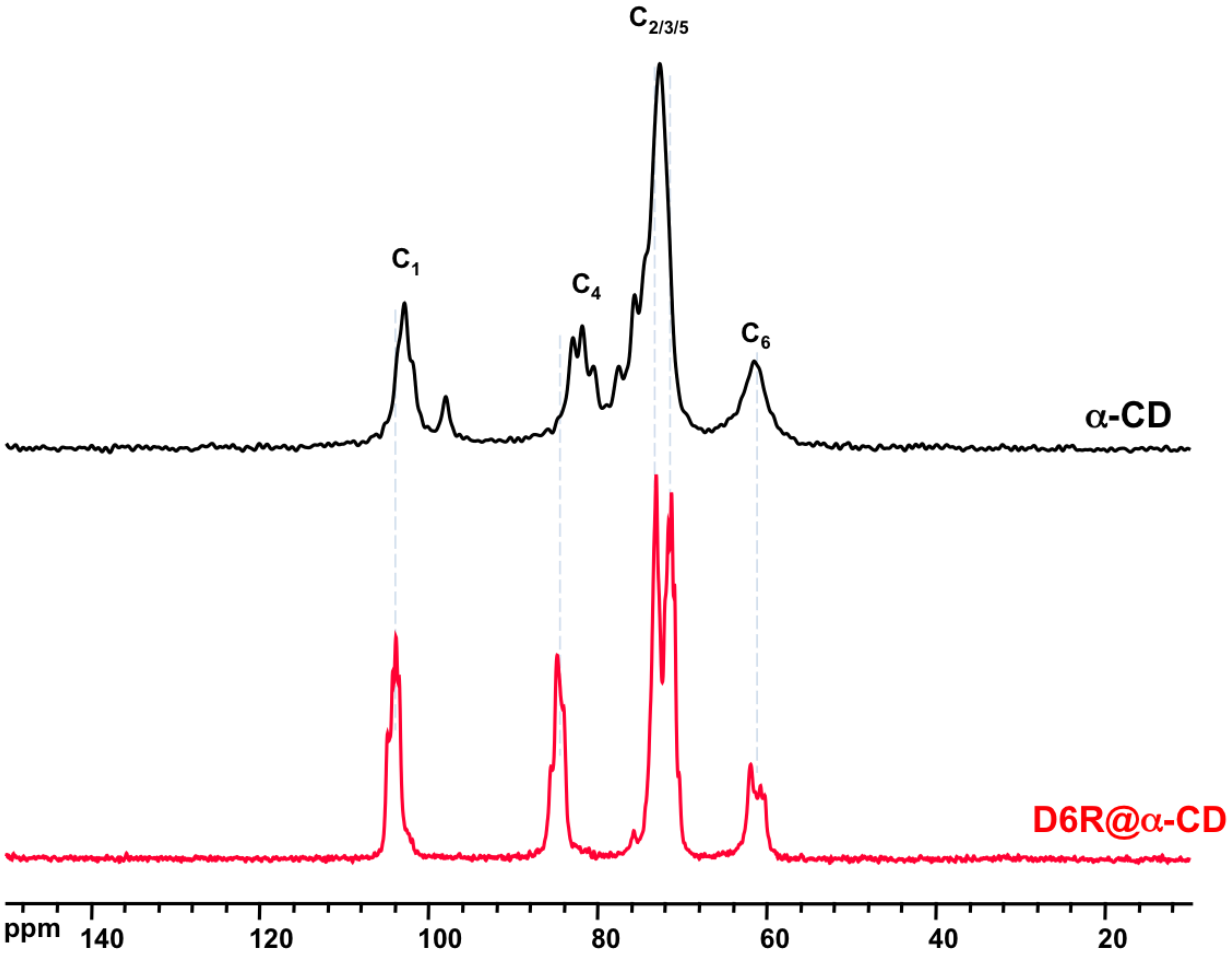

Figure S8. ^{13}C CPMAS NMR spectra of D6R@α-CD and α-CD for comparison.