

Article

Impregnation of Synthetic Saponites with Aldehydes: A Green Approach in the Intercalation of Bioactive Principles

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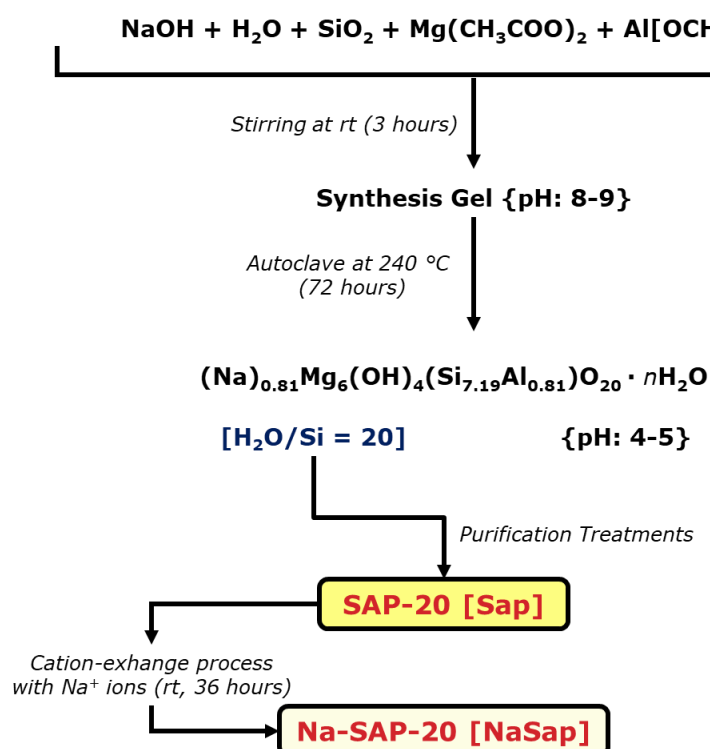
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Electronic Supplementary Information (ESI)

1. Figures



Scheme 1. Schematic representation of the hydrothermal synthesis of synthetic saponite clays SAP-20 (Sap) and Na-SAP-20 (NaSap), prepared with a H₂O/Si ratio of 20 [1,2].

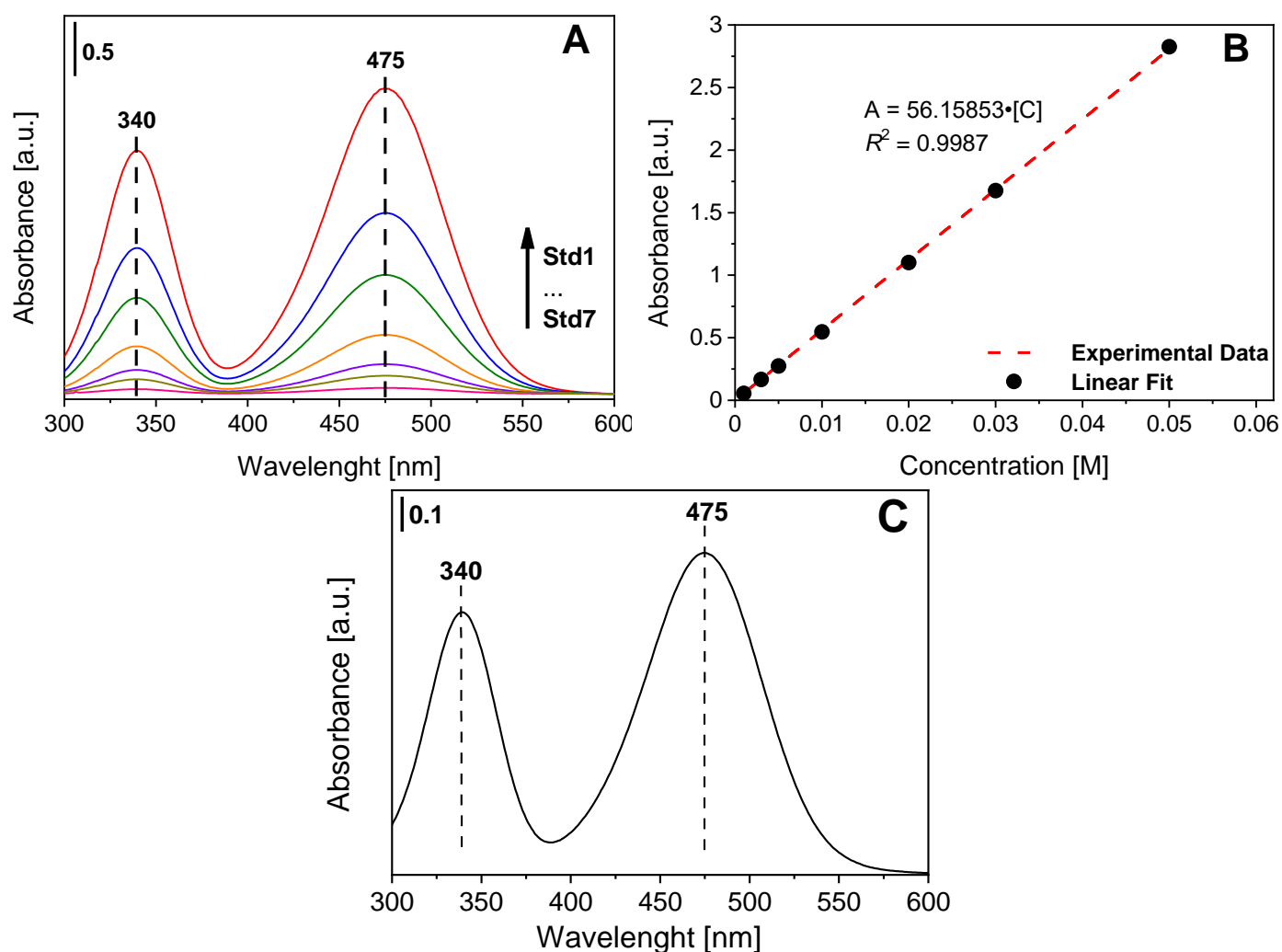


Figure S1. (A) UV-Vis spectra of $[\text{Co}(\text{NH}_3)_6]^{3+}$ standard aqueous solutions at room temperature (concentration range of 0.05–0.005 M) used for the calibration curve. (B) Calibration curve derived from UV-Vis spectra of the standard solutions ($\lambda_{\text{abs}} = 475 \text{ nm}$). (C) UV-Vis spectrum of the supernatant of NaSap sample, obtained after treatment of the solid in 0.02 M $[\text{Co}(\text{NH}_3)_6]^{3+}$ aqueous solution at room temperature for 60 h; the curve represents the average spectrum obtained after 3 replicates.

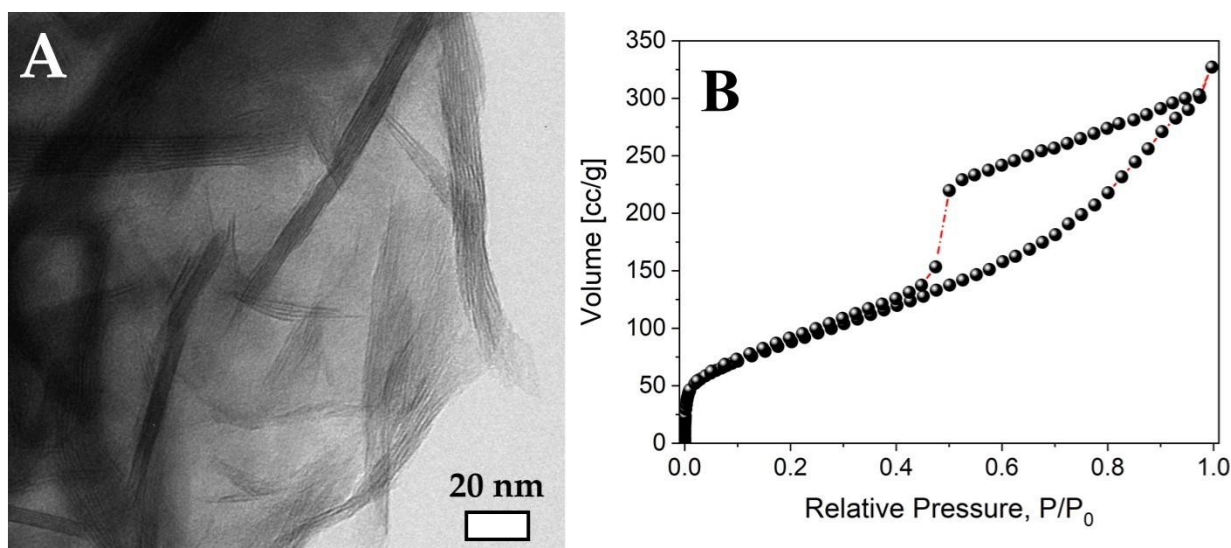
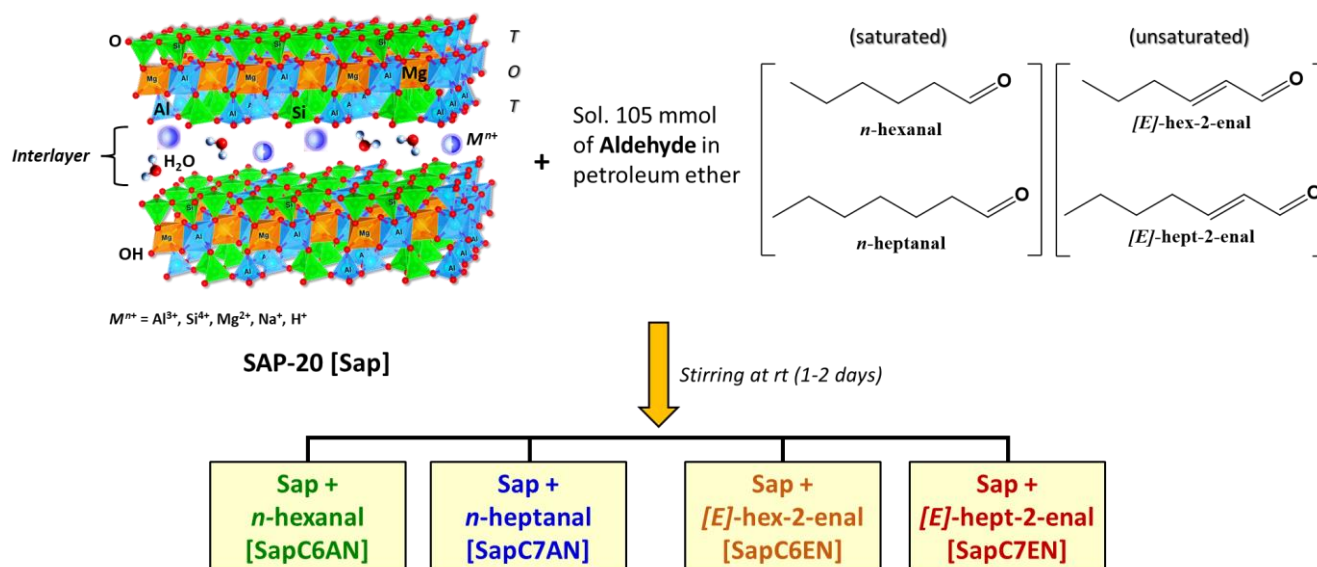


Figure S2. (A) TEM micrograph of NaSap sample. (B) N₂ adsorption/desorption isotherm at 77 K of NaSap.



Scheme 2. Schematic view of the immobilization procedure of different saturated (*n*-hexanal or C6AN, *n*-heptanal or C7AN) and unsaturated ([*E*]-hex-2-enal or C6EN, [*E*]-hept-2-enal or C7EN) aldehydes in the pristine Sap clay.

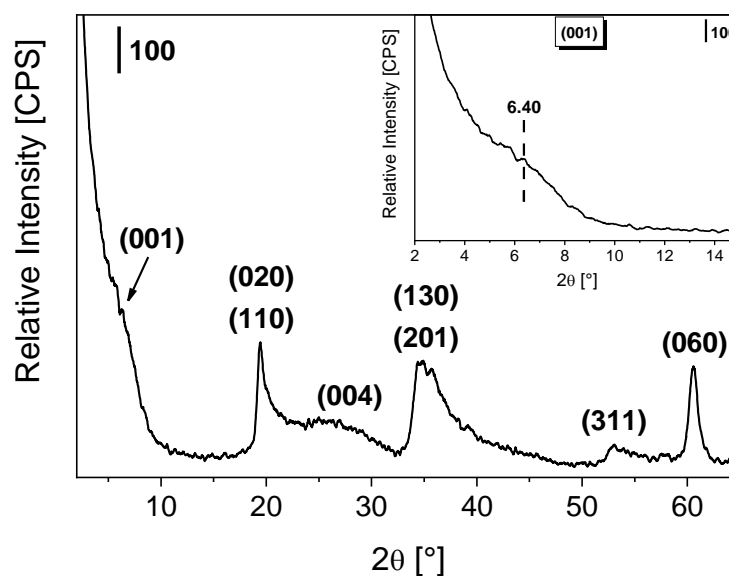


Figure S3. X-ray powder diffraction profile of Sap sample, recorded in the 2–65° 2θ range. Magnification of the 2–15° 2θ region, in which the (001) basal plane is present, is reported in the inset.

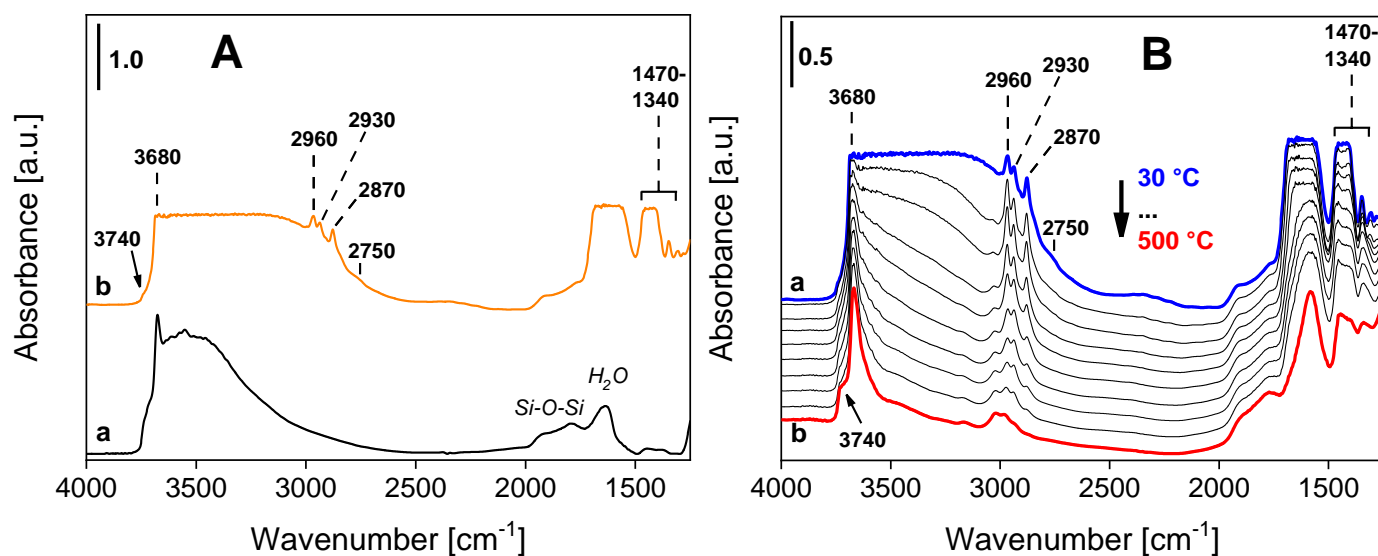


Figure S4. (A) FT-IR spectra, in the 4000–1250 cm^{-1} region, of Sap (a) and SapC6EN (b) samples, measured in vacuum at room temperature on self-supporting pellets. (B) Variable temperature FT-IR spectra of SapC6EN sample, measured in vacuum on a self-supporting pellet, from 30 °C (a) to 500 °C (b).

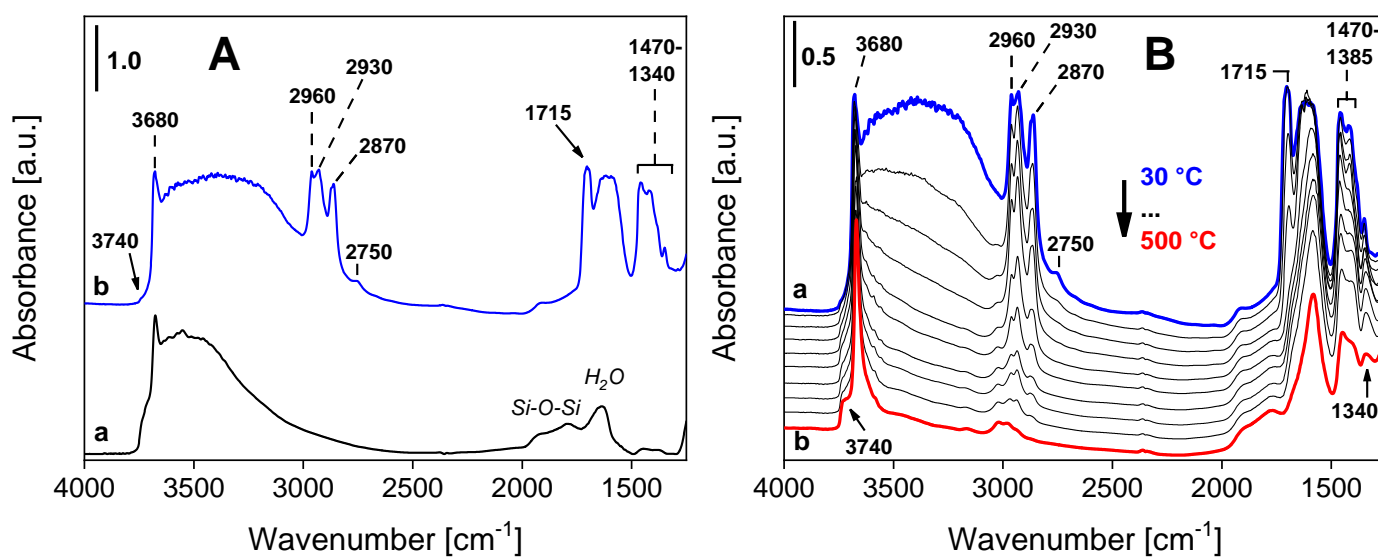


Figure S5. (A) FT-IR spectra, in the 4000–1250 cm^{-1} region, of Sap (a) and SapC7AN (b) samples, measured in vacuum at room temperature on self-supporting pellets. (B) Variable temperature FT-IR spectra of SapC7AN sample, measured in vacuum on a self-supporting pellet, from 30 °C (a) to 500 °C (b).

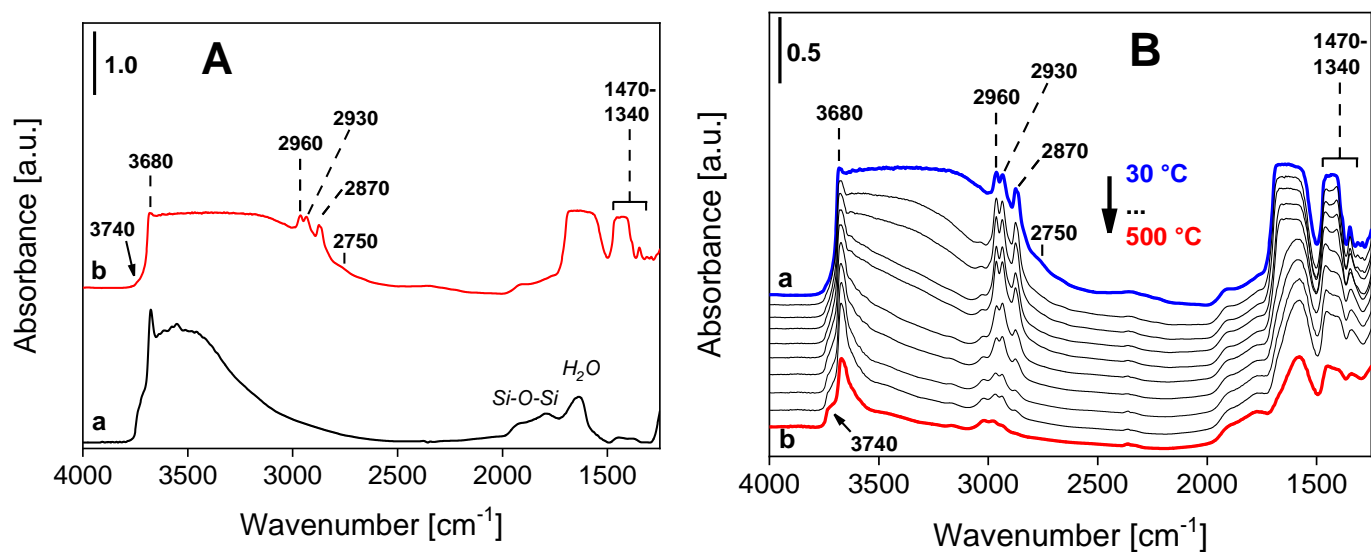


Figure S6. (A) FT-IR spectra, in the 4000–1250 cm^{-1} region, of Sap (a) and SapC7EN (b) samples, measured in vacuum at room temperature on self-supporting pellets. (B) Variable temperature FT-IR spectra of SapC7EN sample, measured *in vacuo* on a self-supporting pellet, from 30 °C (a) to 500 °C (b).

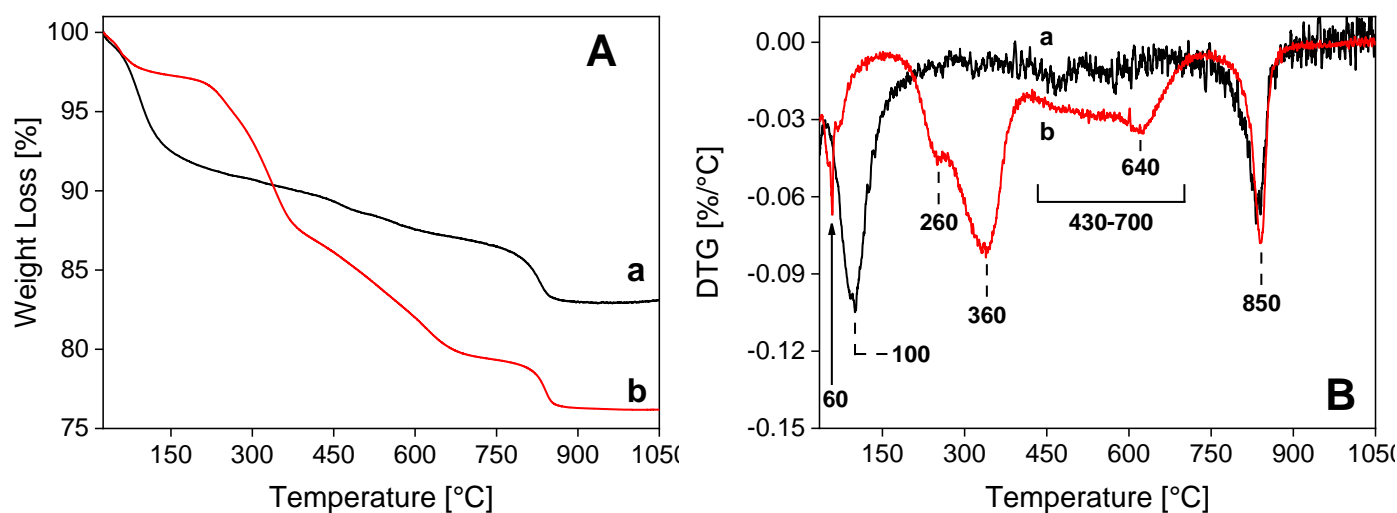


Figure S7. TG (A) and DTG (B) curves of NaSap (a) and CTASap (b) samples. The thermogravimetric analyses were carried out under argon flow.

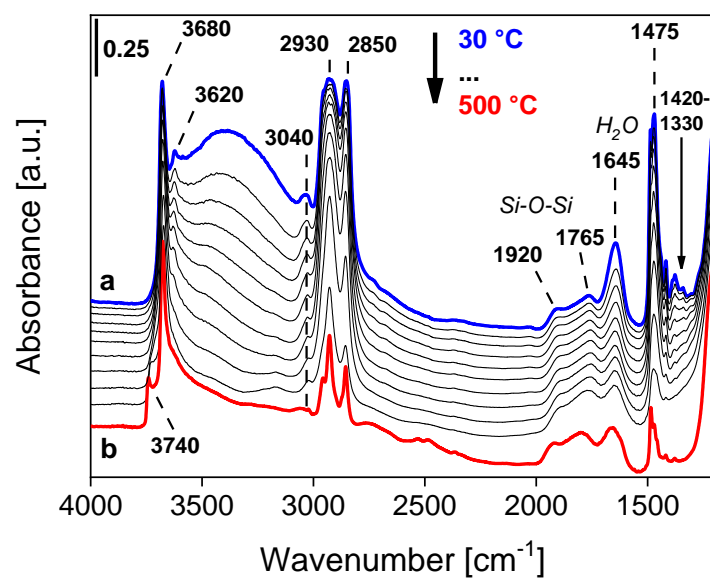


Figure S8. Variable temperature FT-IR spectra of CTASap sample, measured *in vacuo* on a self-supporting pellet, from 30 °C (a) to 500 °C (b).

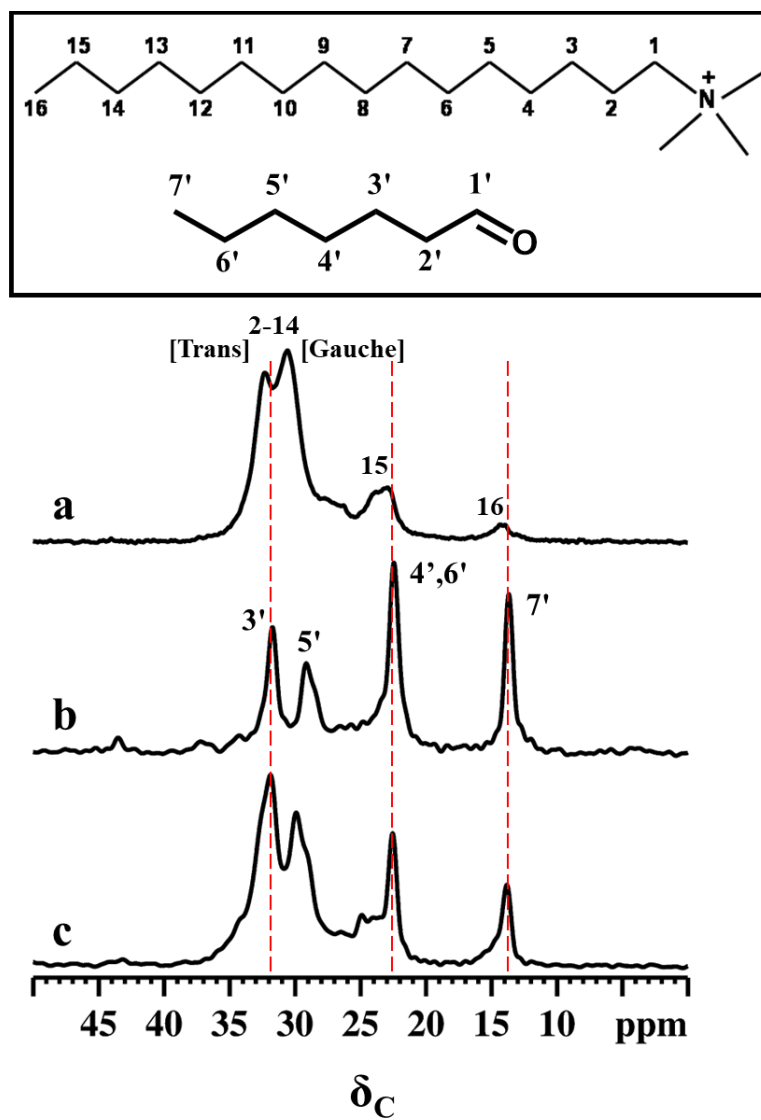


Figure S9. ^{13}C CPMAS NMR spectra of CTASap (a), SapC7AN (b) and CTASapC7AN (c) samples. Only aliphatic region is shown. Inset show the molecular structure of CTA⁺ and C7AN molecules.

2. Tables

Table S1. CHN analysis of CTASap sample.

Sample	C [wt. %]	N [wt. %]	C/N ratio	CTA ⁺ [wt. %]	CTA ⁺ [mmol/g]
CTASap	13.43 ± 0.06	0.84 ± 0.02	15.87 ± 0.41	17.42 ± 2.68	0.61 ± 0.09

Table S2. CTA⁺ content in the CTASap sample obtained from TG analysis in Figure S7.

Sample	g CTA ⁺ / 100 g clay	CTA ⁺ [mmol/g]
CTASap	19.31	0.68

References

1. Marchesi, S.; Carniato, F.; Guidotti, M.; Botta, M.; Marchese, L.; Bisio, C. Synthetic Saponite Clays as Promising Solids for Lanthanide Ion Recovery. *New J. Chem.* **2020**, *44*, 10033–10041, doi:10.1039/C9NJ05983K.
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