

# Supplementary Material

for

## **Chloride binding by layered double hydroxides (LDH/AFm phases) and alkali-activated slag pastes: an experimental study by RILEM TC 283-CAM**

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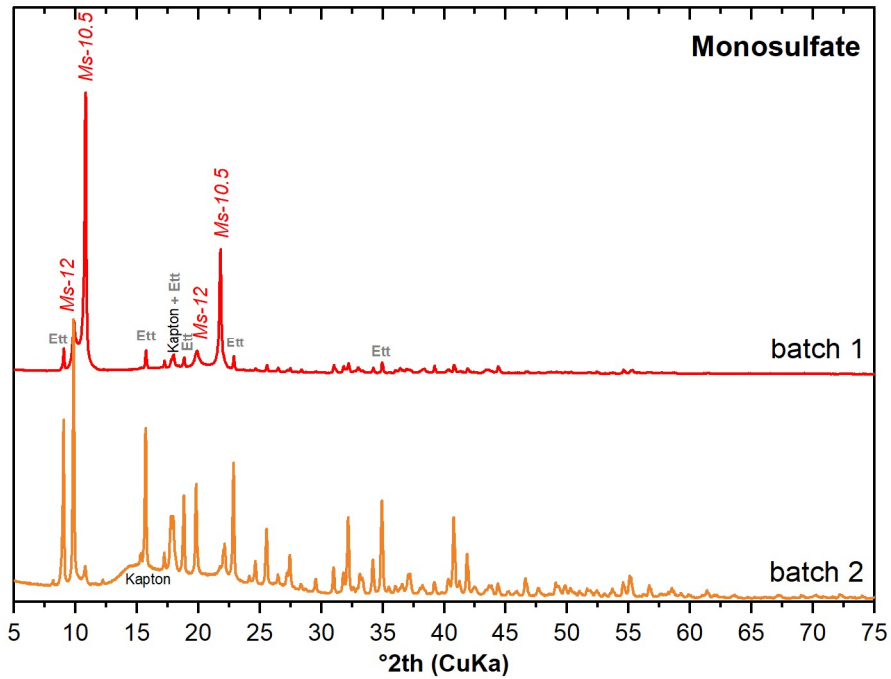
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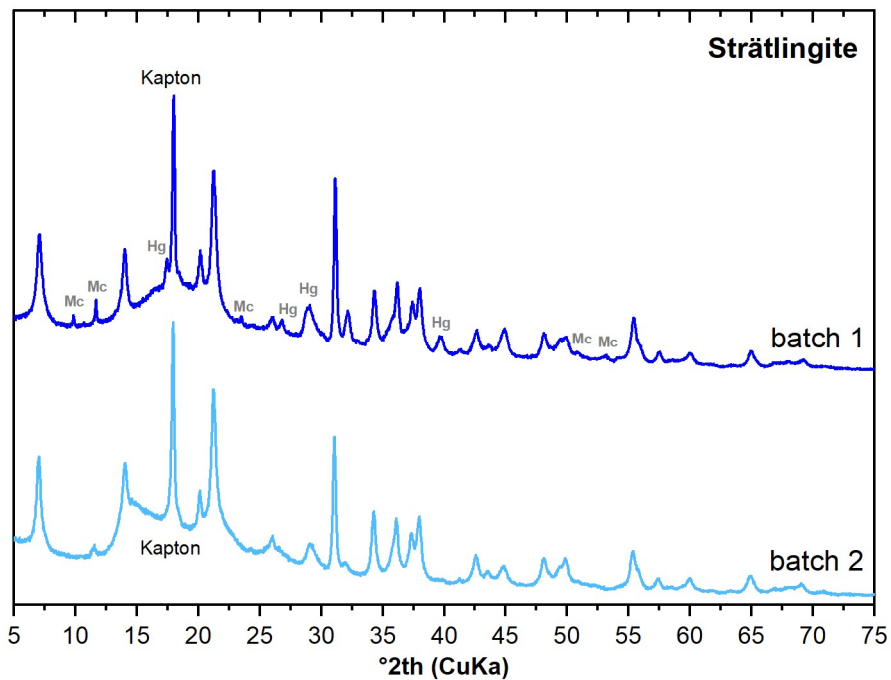
Supplementary Figs. S1–S13: pp. 2–9

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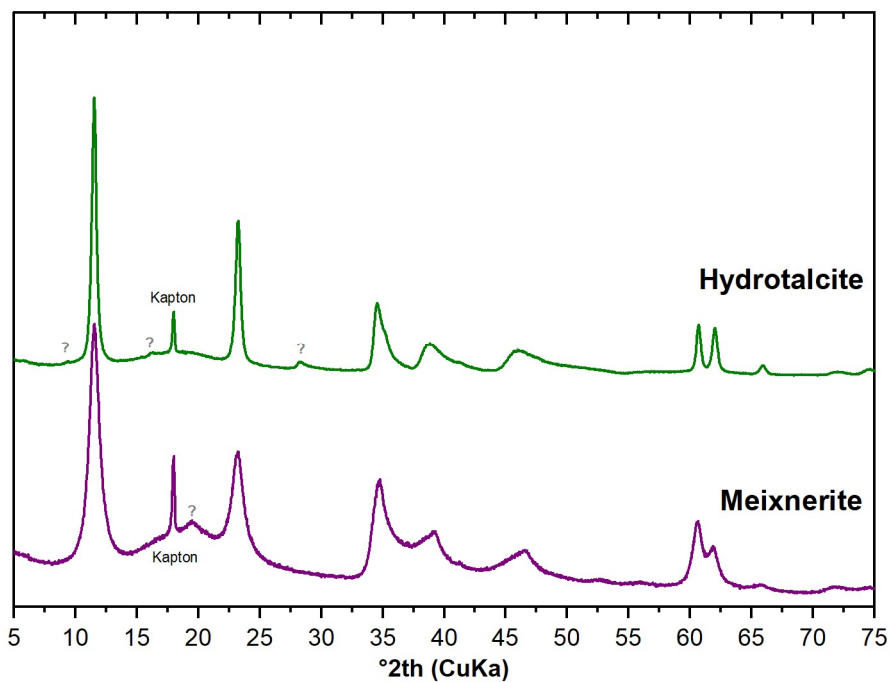
Supplementary Text 1: pp. 12–16



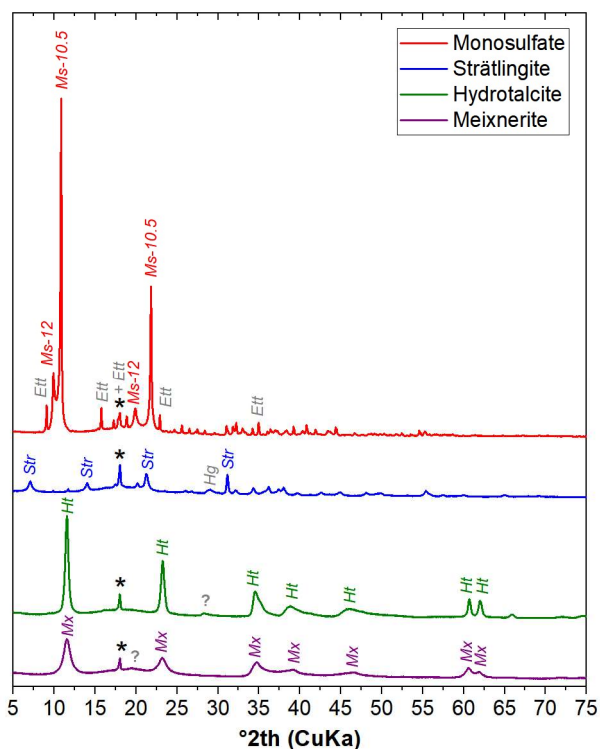
**Supplementary Fig. S1.** X-ray diffraction patterns of monosulfate, batches 1 and 2. Patterns are plotted with their relative intensities. Selected peaks are labelled: Ett, ettringite; *Ms-12*, monosulfate 12-hydrate; *Ms-10.5*, monosulfate 10.5-hydrate; Kapton, Kapton foil used to cover the samples.



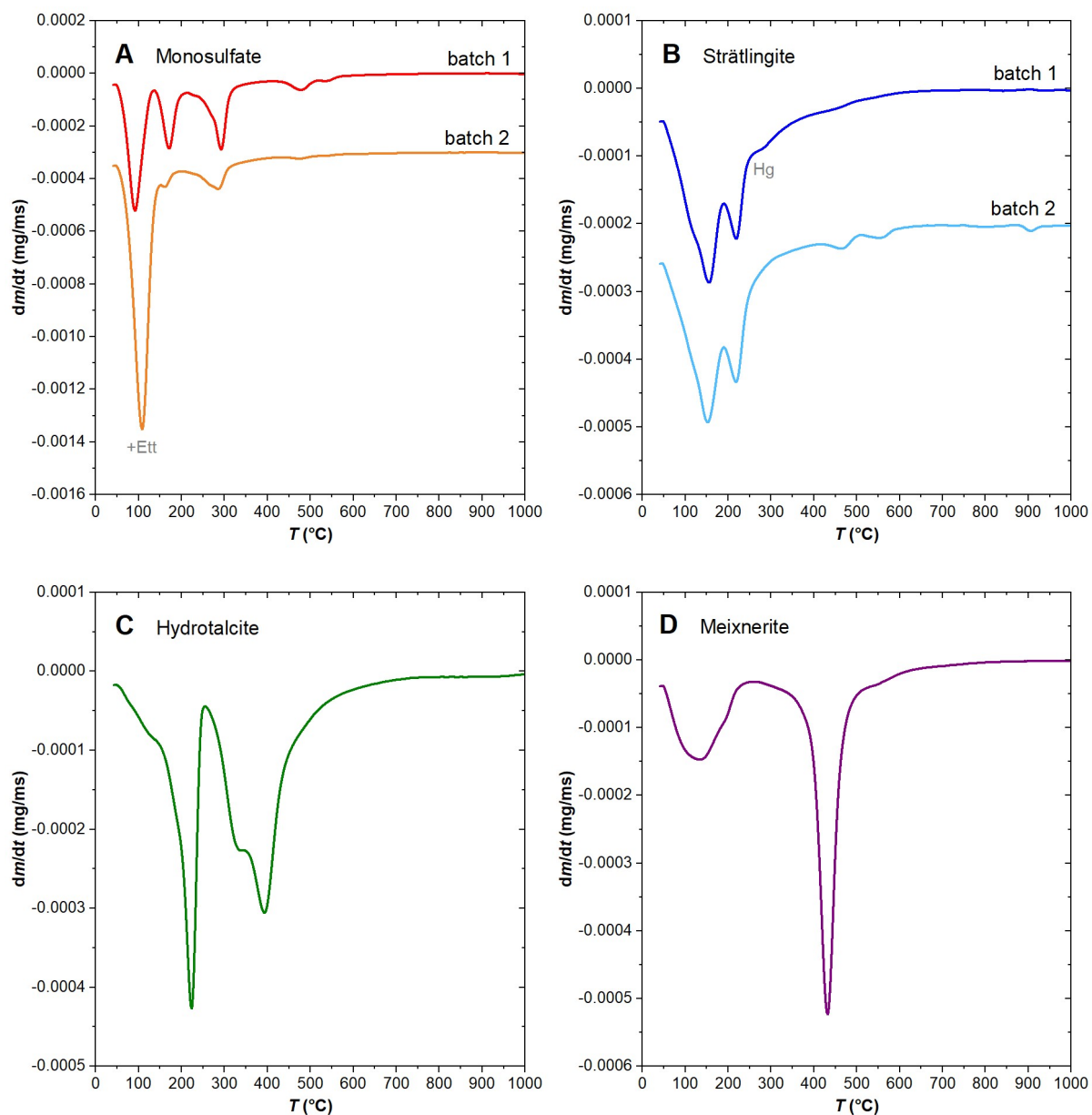
**Supplementary Fig. S2.** X-ray diffraction patterns of strätlingite, batches 1 and 2. Patterns are plotted with their relative intensities. Selected peaks are labelled: Mc, monocarbonate; Hg, hydrogarnet; Kapton, Kapton foil used to cover the samples.



**Supplementary Fig. S3.** X-ray diffraction patterns of hydrotalcite and meixnerite. Patterns are plotted with their relative intensities. Selected peaks are labelled: ?, unidentified impurities in hydrotalcite and meixnerite; Kapton, Kapton foil used to cover the samples.

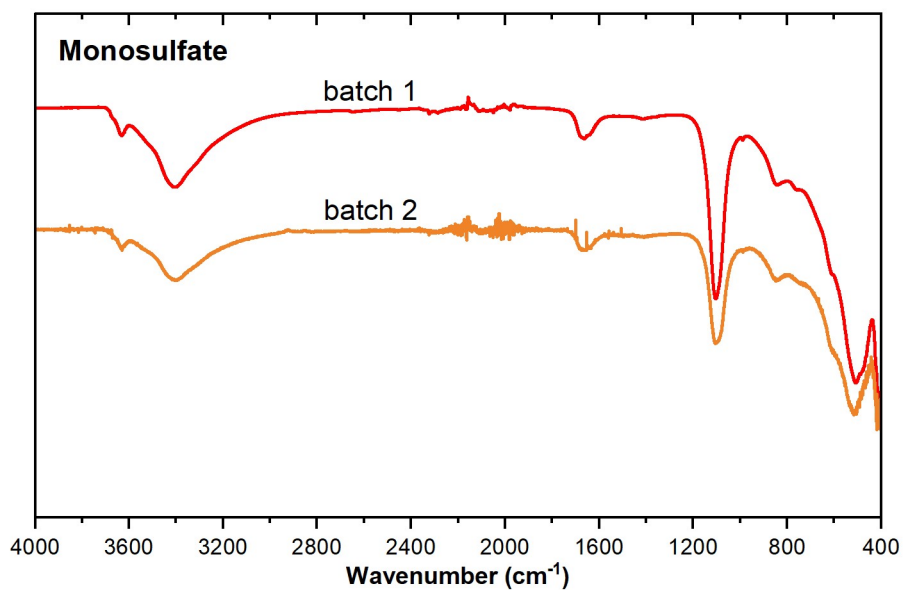


**Supplementary Fig. S4.** X-ray diffraction patterns of the LDH/AFm phases (batch 1). Patterns are plotted with their absolute intensities (a.u.) to demonstrate differences in ‘crystallinity’. Selected peaks are labelled: *Ett*, ettringite; *Ms-12*, monosulfate 12-hydrate; *Ms-10.5*, monosulfate 10.5-hydrate; *Str*, strätlingite; *Hg*, hydrogarnet; *Ht*, hydrotalcite; *Mx*, meixnerite; ?, unidentified impurities in hydrotalcite and meixnerite; \*, Kapton foil used to cover the samples.



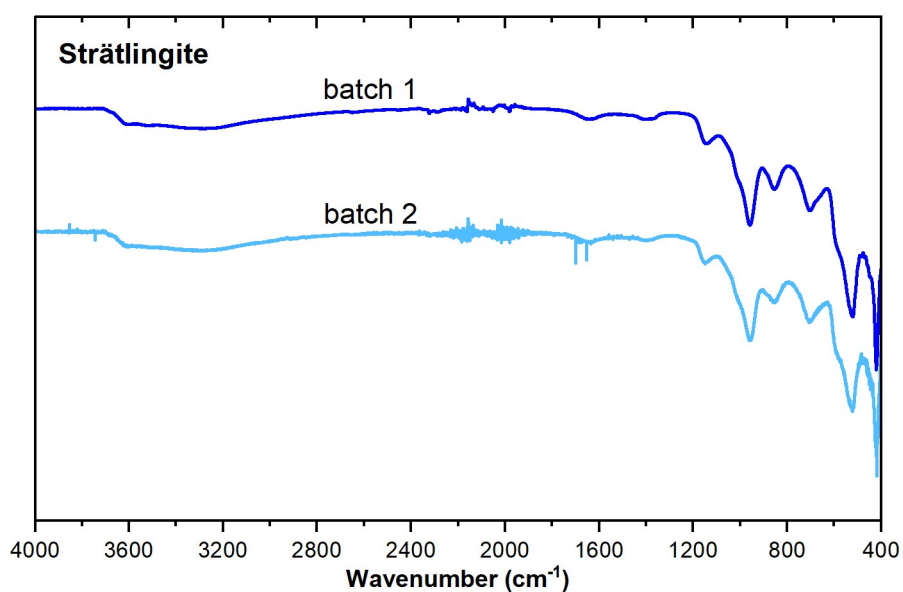
**Supplementary Fig. S5.** Differential thermogravimetry curves of **A**, monosulfate, batches 1 and 2; **B**, strätlingite, batches 1 and 2; **C**, hydrotalcite; **D**, meixnerite. The curves of monosulfate batch 2 and strätlingite batch 2 are shifted by  $-0.0003$  mg/ms and  $-0.0002$  mg/ms, respectively, for better visibility. Selected signals of impurities are labelled: +Ett, contribution of ettringite; Hg, hydrogarnet.

The thermogravimetric analyses were performed with a Netzsch STA 449 C Jupiter device. Sample masses of 10 mg were used, and the analyses were conducted under flowing nitrogen gas at a heating rate of  $10$  °C/min, starting at  $40$  °C.



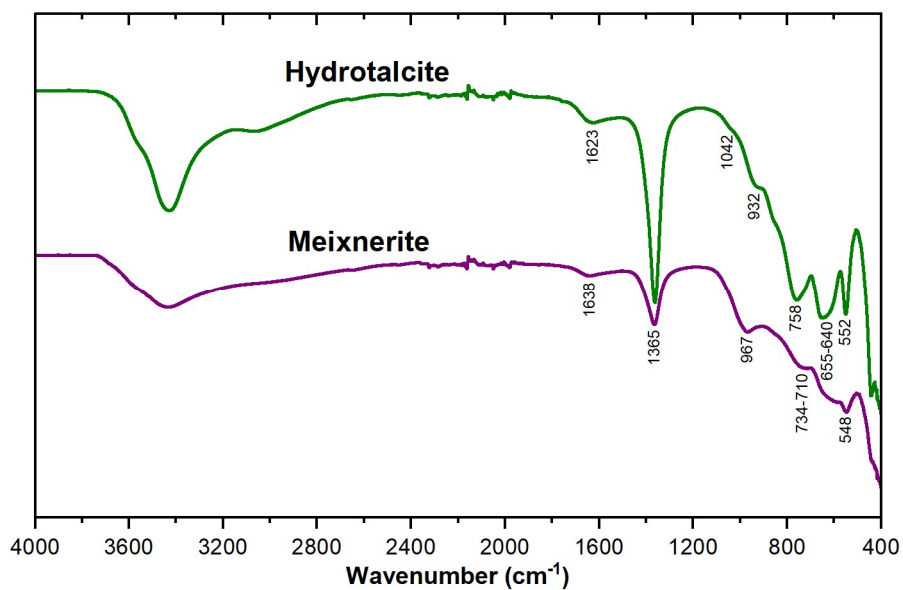
**Supplementary Fig. S6.** Infrared (ATR-FTIR) spectra of monosulfate, batches 1 and 2.

Attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectra of the samples were recorded at room temperature in the mid-IR region (wavenumber 4000–400  $\text{cm}^{-1}$ ) on a Thermo Scientific Nicolet iS 50 FTIR spectrometer with an optical resolution of better than 0.09  $\text{cm}^{-1}$ . For each sample, 32 scans were recorded and averaged. The spectra are plotted with no additional scaling, *i.e.*, with their absolute transmittances as outputted by the spectrometer software.



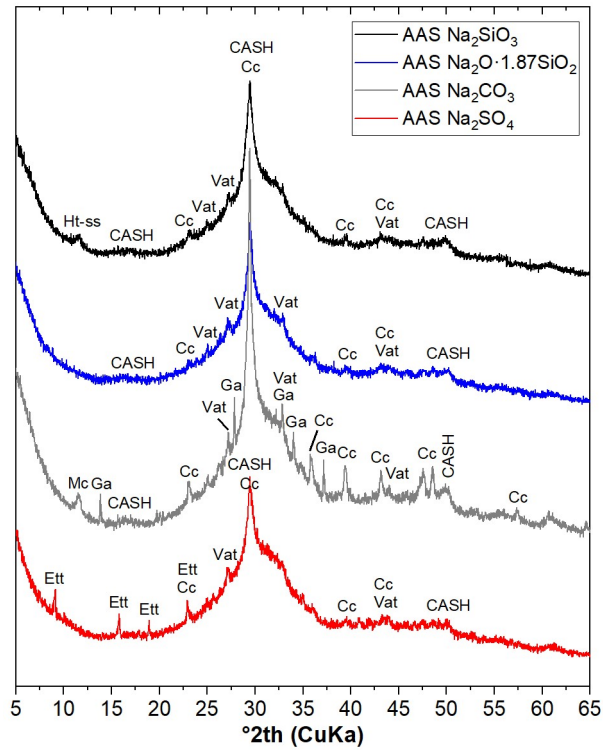
**Supplementary Fig. S7.** Infrared (ATR-FTIR) spectra of strätlingite, batches 1 and 2.

Experimental conditions and data presentation as described in the caption of Supplementary Fig. S6.

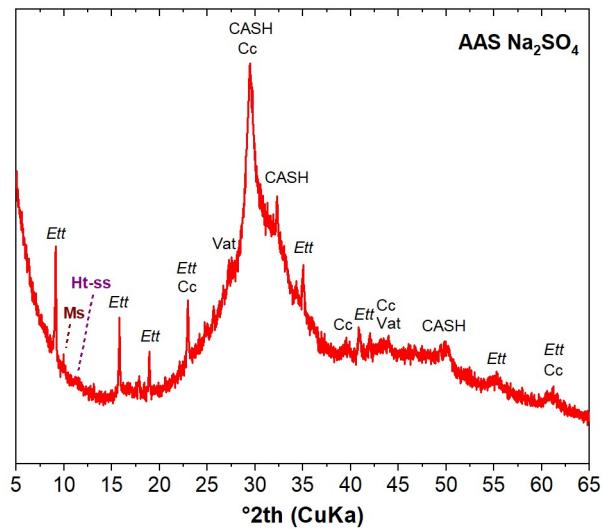


**Supplementary Fig. S8.** Infrared (ATR-FTIR) spectra of hydrotalcite and meixnerite.

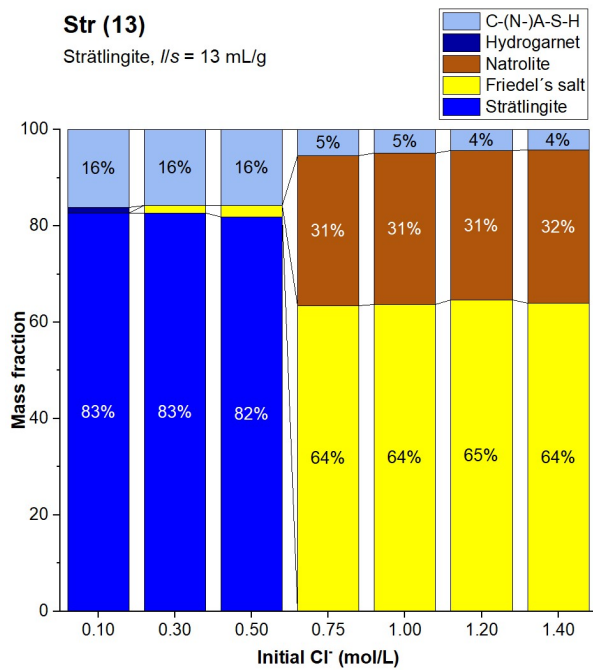
Experimental conditions and data presentation as described in the caption of Supplementary Fig. S6. The infrared spectra of hydrotalcite and meixnerite (Mg-Al-OH LDH) are usually distinguished by the different intensities of their carbonate bands at  $\sim 1365\text{ cm}^{-1}$ , as for the present materials, while differences between their (Mg,Al)-O bands in the range of approx.  $1000\text{--}500\text{ cm}^{-1}$  are generally not sufficient to differentiate between these phases. Cf. Bernard E, Zucha WJ, Lothenbach B, Mäder U (2022) Stability of hydrotalcite (Mg-Al layered double hydroxide) in presence of different anions. *Cem Concr Res* 152:106674, <https://doi.org/10.1016/j.cemconres.2021.106674>; and Koritnig S, Süsse P (1975) Meixnerit,  $\text{Mg}_6\text{Al}_2(\text{OH})_{18}\cdot 4\text{H}_2\text{O}$ , ein neues Magnesium-Aluminium-Hydroxid-Mineral. *Tschermaks Mineral Petrogr Mitt* 22:79-87, <https://doi.org/10.1007/BF01081303>.



**Supplementary Fig. S9.** X-ray diffraction patterns of the AAS pastes (batch 1) after 48-day curing (AAS  $\text{Na}_2\text{CO}_3$ ) or 28-day curing (all other pastes). Selected peaks are labelled: Ht-ss, hydrotalcite-like compound (Mg-Al-OH LDH); CASH, C-(N-)A-S-H gel; Cc, calcite; Vat, vaterite; Mc, monocarbonate; Ga, gaylussite; Ett, ettringite.

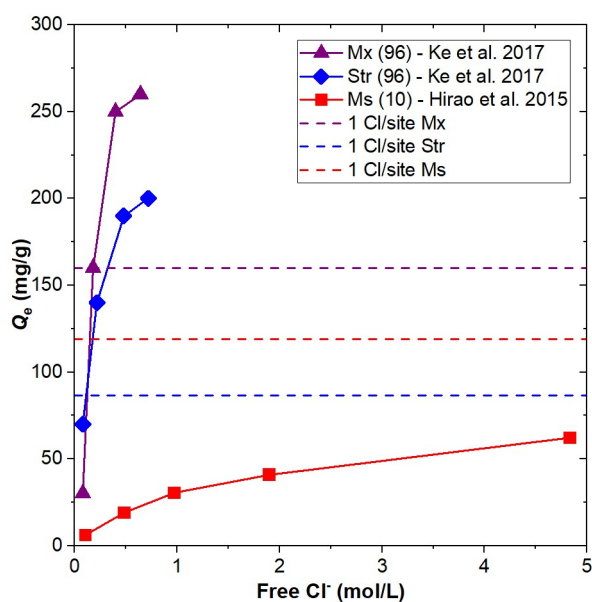


**Supplementary Fig. S10.** X-ray diffraction pattern of the AAS  $\text{Na}_2\text{SO}_4$  paste (batch 1) after 90-day curing. Selected peaks are labelled: *Ett*, ettringite; *Ms*, monosulfate 12-hydrate; Ht-ss, hydrotalcite-like compound (Mg-Al-OH LDH); Vat, vaterite; CASH, C-(N-)A-S-H gel; Cc, calcite.



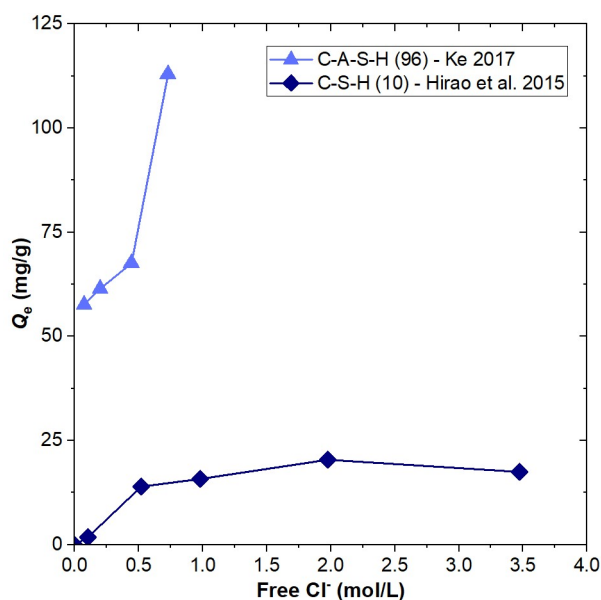
**Supplementary Fig. S11.** Modelled equilibrium solid-phase assemblages in the chloride binding experiments with strätlingite at a liquid volume/solid mass ratio of 13 mL/g.





**Supplementary Fig. S12.** Chloride sorption isotherms of a calcined Mg-Al LDH (Mx) and strätlingite (Str) in NaOH/NaCl solutions, and monosulfate (Ms) in NaCl solutions. The liquid volume/solid mass ratios used for the experiments were approx. 96 mL/g for Mx and Str, and 10 mL/g for Ms.

Data from Ke X, Bernal SA, Provis JL (2017) Uptake of chloride and carbonate by Mg-Al and Ca-Al layered double hydroxides in simulated pore solutions of alkali-activated slag cement. *Cem Concr Res* 100:1-13, <https://doi.org/10.1016/j.cemconres.2017.05.015>; and Hirao H, Yamada K, Takahashi H, Zibara H (2005) Chloride binding of cement estimated by binding isotherms of hydrates. *J Adv Concr Technol* 3:77-84, <https://doi.org/10.3151/jact.3.77>.



**Supplementary Fig. S13.** Chloride sorption isotherms of C-A-S-H (nominal Ca/Si = 1.0, Al/Si = 0.1; named CNASH-B in the original publication) in NaOH/NaCl solutions, and C-S-H in NaCl solutions. The liquid volume/solid mass ratios used for the experiments were approx. 96 mL/g for C-A-S-H, and 10 mL/g for C-S-H.

Data from Ke X (2017) Improved durability and sustainability of alkali-activated slag cements. PhD thesis, University of Sheffield; and Hirao H, Yamada K, Takahashi H, Zibara H (2005) Chloride binding of cement estimated by binding isotherms of hydrates. *J Adv Concr Technol* 3:77-84, <https://doi.org/10.3151/jact.3.77>.

**Supplementary Table S1.** Chemical compositions of the ground granulated blast-furnace slags (GGBFS) used for batch 1 (BAM) and batch 2 (ETH).

<b>Component</b>	<b>GGBFS Batch 1 (wt.%)</b>	<b>GGBFS Batch 2 (wt.%)</b>
CaO	43.8	42.1
MgO	4.9	5.1
SiO <sub>2</sub>	33.4	35.0
Al <sub>2</sub> O <sub>3</sub>	12.0	11.4
Fe <sub>2</sub> O <sub>3</sub>	0.3	0.3
Na <sub>2</sub> O	0.3	0.2
K <sub>2</sub> O	0.8	0.5
SO <sub>3</sub> <sup>a</sup>	1.8	2.0
Mn <sub>2</sub> O <sub>3</sub> <sup>b</sup>	0.4	0.1
LOI <sup>c</sup>	1.2	1.7

<sup>a</sup> Sulfur species, including reduced sulfur (presumably mainly S<sup>2-</sup>), expressed as SO<sub>3</sub>

<sup>b</sup> Manganese species in different oxidation states, expressed as Mn<sub>2</sub>O<sub>3</sub>

<sup>c</sup> Loss on ignition

**Supplementary Table S2.** Examples of experimental parameters applied in previous studies of chloride binding in hydrated cement pastes and alkali-activated materials, compared to the present study.

Reference <sup>a</sup>	Material	l/s (mL/g)	Time
Delagrave <i>et al.</i> (1997)	Cement paste	2.5 or 5	3 weeks
Ipavec <i>et al.</i> (2013)	Cement paste	10	2 months
De Weerd <i>et al.</i> (2015)	Cement paste	0.5	2 months
Machner <i>et al.</i> (2018)	Cement paste	0.5	≥1 month
Ukpata <i>et al.</i> (2019)	Cement paste	4	6 weeks
Avet and Scrivener (2020)	Cement paste	4	2 months
Ke <i>et al.</i> (2017a)	AAS paste	~6.7	2 months
Zhang <i>et al.</i> (2019)	AAS/FA paste	3.2	2 months
<i>Present study</i>	AAS paste	4	3 weeks
Hirao <i>et al.</i> (2005)	AFm, AFt, CH, and C-S-H/CH	10	2 days
Ke (2017)	C-A-S-H	~96	4 months
Ke <i>et al.</i> (2017b)	Mg-Al LDH and AFm	~96	1 or 3 weeks
<i>Present study</i>	Mg-Al LDH and AFm	13, 26, 53, and 108	3 weeks

<sup>a</sup> References:

Avet F, Scrivener K (2020) Influence of pH on the chloride binding capacity of Limestone Calcined Clay Cements (LC<sup>3</sup>). *Cem Concr Res* 131:106031. <https://doi.org/10.1016/j.cemconres.2020.106031>

De Weerd K, Colombo A, Coppola L, Justnes H, Geiker MR (2015) Impact of the associated cation on chloride binding of Portland cement paste. *Cem Concr Res* 68:196-202. <https://doi.org/10.1016/j.cemconres.2014.01.027>

Delagrave A, Marchand J, Ollivier J-P, Julien S, Hazrati K (1997) Chloride binding capacity of various hydrated cement paste systems. *Adv Cem Based Mater* 6:28-35. [https://doi.org/10.1016/S1065-7355\(97\)90003-1](https://doi.org/10.1016/S1065-7355(97)90003-1)

Hirao H, Yamada K, Takahashi H, Zibara H (2005) Chloride binding of cement estimated by binding isotherms of hydrates. *J Adv Concr Technol* 3:77-84. <https://doi.org/10.3151/jact.3.77>

Ipavec A, Vuk T, Gabrovšek R, Kaučič V (2013) Chloride binding into hydrated blended cements: the influence of limestone and alkalinity. *Cem Concr Res* 48:74-85. <https://doi.org/10.1016/j.cemconres.2013.02.010>

Ke X (2017) Improved durability and sustainability of alkali-activated slag cements. PhD thesis, University of Sheffield

Ke X, Bernal SA, Hussein OH, Provis JL (2017a) Chloride binding and mobility in sodium carbonate-activated slag pastes and mortars. *Mater Struct* 50:252. <https://doi.org/10.1617/s11527-017-1121-8>

Ke X, Bernal SA, Provis JL (2017b) Uptake of chloride and carbonate by Mg-Al and Ca-Al layered double hydroxides in simulated pore solutions of alkali-activated slag cement. *Cem Concr Res* 100:1-13. <https://doi.org/10.1016/j.cemconres.2017.05.015>

Machner A, Zajac M, Ben Haha M, Kjellsen KO, Geiker MR, De Weerd K (2018) Chloride-binding capacity of hydrotalcite in cement pastes containing dolomite and metakaolin. *Cem Concr Res* 107:163-181. <https://doi.org/10.1016/j.cemconres.2018.02.002>

Ukpata JO, Basheer PAM, Black L (2019) Slag hydration and chloride binding in slag cements exposed to a combined chloride-sulphate solution. *Constr Build Mater* 195:238-248. <https://doi.org/10.1016/j.conbuildmat.2018.11.055>

Zhang J, Shi C, Zhang Z (2019) Chloride binding of alkali-activated slag/fly ash cements. *Constr Build Mater* 226:21-31. <https://doi.org/10.1016/j.conbuildmat.2019.07.281>

## Supplementary Text 1

### Synthesis protocols for LDH/AFm phases

**Monosulfate**  $\text{Ca}_4\text{Al}_2\text{SO}_4(\text{OH})_{12}\cdot 6\text{H}_2\text{O}$   
=  $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SO}_3\cdot 12\text{H}_2\text{O}$  [*@ 11 % RH:  $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SO}_3\cdot 10.5\text{H}_2\text{O}$* ]

Ref.: T. Matschei *et al.*, *Cem. Concr. Res.* 37 (2007) 1379–1410.

Chemicals required:  $3\text{CaO}\cdot\text{Al}_2\text{O}_3$  (“aluminat”),  $\text{CaSO}_4$  or  $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$ , and ultrapure water (not necessarily degassed).

Conditions required: All steps (except 2. and 4.) need to be done in the **glove box** under Ar or  $\text{N}_2$  atmosphere. Constant temperature in the range 20–25 °C (if not specified otherwise).

*If required:* Calcine the required amount of  $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$  at 200 °C in a muffle furnace for 2 hours to remove the  $\text{H}_2\text{O}$ . Allow the calcined material (*i.e.*,  $\text{CaSO}_4$ ) to cool naturally in the furnace until a furnace temperature of ~150 °C is reached (make sure that *T* is always >105 °C). Remove the crucible with the  $\text{CaSO}_4$  from the furnace and immediately transfer it to a desiccator with dry silica gel; allow the  $\text{CaSO}_4$  to cool to room temperature in the desiccator. [Measure the mass of the material before and after calcination to check for complete dehydration.]

1. **[glove box]** Suspend a 1:1 molar mixture of  $3\text{CaO}\cdot\text{Al}_2\text{O}_3$  and  $\text{CaSO}_4$  in ultrapure water. See table below for the composition of the suspension.

Water (g)	$3\text{CaO}\cdot\text{Al}_2\text{O}_3$ (g)	$\text{CaSO}_4$ (g)
200.00	9.9743	5.0257

2. Age the suspension in a sealed bottle at 85 °C for 7 days.
3. **[glove box]** Vacuum filtration (using “Blauband” filter paper, *i.e.*, slow filter paper) and washing with ultrapure water at least five times.
4. Dry filter residue in a desiccator in a  $\text{CO}_2$ -free atmosphere [**Ar or  $\text{N}_2$** ] above saturated LiCl solution (11 % RH) for 3 weeks.

If necessary for subsequent analyses/experiments, grind the material [**in a glove box under Ar or  $\text{N}_2$  atmosphere**]. Store the material in sealed containers until required for analyses/experiments.

**Strätlingite**  $\text{Ca}_2\text{Al}_2\text{SiO}_2(\text{OH})_{10}\cdot 3\text{H}_2\text{O}$   
 $= 2\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2\cdot 8\text{H}_2\text{O}$  [*@ 11 % RH:  $2\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2\cdot\{7\dots 7.5\}\text{H}_2\text{O}$* ]

Ref.: Matschei, T. *et al.*, *Cem. Concr. Res.* 37 (2007) 1379–1410.

Chemicals required:  $\text{Ca}(\text{OH})_2$ , waterglass solution with 27.3 wt.%  $\text{SiO}_2$  and 15.0 wt.%  $\text{Na}_2\text{O}$ ,  $\text{NaAlO}_2$  and ultrapure water (not necessarily degassed).

Conditions required: All steps (except 5. and 7.) need to be done in the **glove box** under Ar or  $\text{N}_2$  atmosphere. Constant temperature in the range 20–25 °C.

*Optional:* Calcine the  $\text{NaAlO}_2$  at 500 °C in a muffle furnace for 1 hour to remove impurity  $\text{CO}_2$  and  $\text{H}_2\text{O}$ . Allow the calcined material to cool naturally in the furnace until a furnace temperature of ~150 °C is reached (make sure that  $T$  is always >105 °C). Remove the crucible with the material from the furnace and immediately transfer it to a desiccator with dry silica gel; allow the material to cool to room temperature in the desiccator.

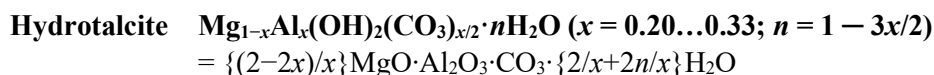
1. **[glove box]** Suspend each of the solid chemicals in separate bottles/beakers containing ultrapure water. See table below for the compositions of the suspensions.

	Water (g)	Solute (g)
$\text{Ca}(\text{OH})_2$	151.22	7.5610
Waterglass sln.*	11.25 g of solution	
$\text{NaAlO}_2$	167.30	8.3650

\* Composition: 27.3 wt.%  $\text{SiO}_2$ ; 15.0 wt.%  $\text{Na}_2\text{O}$ ; remainder  $\text{H}_2\text{O}$

2. **[glove box]** Add  $\text{NaAlO}_2$  suspension to  $\text{Ca}(\text{OH})_2$  suspension.
3. **[glove box]** Add mixed suspension prepared in step 2 to the waterglass solution.
4. **[glove box]** Make up the suspension with ultrapure water to 450 ml.
5. Stir suspension in a sealed bottle for 4 weeks.
6. **[glove box]** Vacuum filtration (using “Blauband” filter paper, *i.e.*, slow filter paper) and washing with ultrapure water at least five times to remove alkalis.
7. Dry filter residue in a desiccator in a  $\text{CO}_2$ -free atmosphere [**Ar or  $\text{N}_2$** ] above saturated  $\text{LiCl}$  solution (11 % RH) for 3 weeks.

If necessary for subsequent analyses/experiments, grind the material [**in a glove box under Ar or  $\text{N}_2$  atmosphere**]. Store the material in sealed containers until required for analyses/experiments.



Refs.: S. Miyata, *Clays Clay Miner.* 28 (1980) 50–56; see also T. Hibino *et al.*, *Clays Clay Miner.* 43 (1995) 427–432.

Chemicals required:  $\text{MgCl}_2$ ,  $\text{AlCl}_3$ ,  $\text{NaOH}$ ,  $\text{Na}_2\text{CO}_3$  and ultrapure water (not necessarily degassed).

Conditions required: Constant temperature in the range 20–25 °C (if not specified otherwise).

1. Prepare aqueous solutions of  $\text{MgCl}_2$  (0.7 M),  $\text{AlCl}_3$  (0.3 M),  $\text{NaOH}$  (2 M) and  $\text{Na}_2\text{CO}_3$  (0.2 M) separately in ultrapure water. See table below for the compositions of the suspensions/solutions [calculated for  $x = 0.30$  and  $\text{CO}_3^{2-}/\text{Al}^{3+} = 0.67$  ( $\text{CO}_3^{2-}/\text{Al}^{3+} = 0.50$  is required by stoichiometry)].

	Concentration (M)	Volume (ml)	Weight of solute (g)
$\text{MgCl}_2$	0.7	100	6.6648
$\text{AlCl}_3$	0.3	100	4.0002
$\text{NaOH}$	2.0	100	7.9994
$\text{Na}_2\text{CO}_3$	0.2	100	2.1198

2. Mix all solutions.
3. Make up the resulting suspension with ultrapure water to 450 ml and adjust the pH to  $10 \pm 0.2$  using  $\text{NaOH}$  solution.
4. Stir the suspension at 40–50 °C for 20 min.
5. Vacuum filtration (using “Blauband” filter paper, *i.e.*, slow filter paper) and washing with ultrapure water at least five times.
6. Immerse filter residue in 0.1 M  $\text{Na}_2\text{CO}_3$  solution (see table below for the composition of the solution), at a concentration of ~1 g of precipitate per 20 ml of solution, in a closed bottle for 7 days to remove residual  $\text{Cl}^-$  in the precipitate through ion exchange; stir or shake bottle once per day.

	Concentration (M)	Volume (ml)	Weight of solute (g)
$\text{Na}_2\text{CO}_3$	0.1	750	7.9492

7. Vacuum filtration (using “Blauband” filter paper, *i.e.*, slow filter paper) and washing with ultrapure water at least five times.
8. Dry filter residue at 80 °C for 20–24 hours in an Erlenmeyer flask with  $\text{CO}_2$  adsorbent ( $\text{NaOH}$ ) on top.
9. Immerse dried filter residue in ~10 ml of ultrapure water per 1 g of precipitate.
10. Aging of the suspension in an Erlenmeyer flask with  $\text{CO}_2$  adsorbent ( $\text{NaOH}$ ) on top for 4 days at 90–95 °C.
11. Vacuum filtration (using “Blauband” filter paper, *i.e.*, slow filter paper) and washing with ultrapure water at least five times.

12. Dry filter residue in a desiccator above saturated LiCl solution (11 % RH) for 3 weeks.

If necessary for subsequent analyses/experiments, grind the material. Store the material in sealed containers until required for analyses/experiments.

**Mg-Al-OH LDH**      **approx. meixnerite,  $\text{Mg}_{0.75}\text{Al}_{0.25}(\text{OH})_{2.25}\cdot 0.5\text{H}_2\text{O}$**   
 =  $6\text{MgO}\cdot\text{Al}_2\text{O}_3\cdot 13\text{H}_2\text{O}$   
**actual stoichiometry depending on starting hydrotalcite**

Ref.: X. Ke *et al.*, *Cem. Concr. Res.* 81 (2016) 24–37. [Regarding calcination of hydrotalcite and remaining carbonate, see T. Hibino *et al.*, *Clays Clay Miner.* 43 (1995) 427–432 and T. Stanimirova *et al.*, *Clay Miner.* 39 (2004) 177–191. Regarding stoichiometry of synthetic Mg-Al-OH LDH, see G. Mascolo & O. Marino, *Mineral. Mag.* 43 (1980) 619–621]

Chemicals required: Hydrotalcite, NaOH and ultrapure water (not necessarily degassed).

Conditions required: Steps 4. and 6. need to be done in the **glove box** under Ar or N<sub>2</sub> atmosphere. Constant temperature in the range 20–25 °C (if not specified otherwise).

1. Heat the required amount of hydrotalcite in a crucible at a heating rate of 5 °C/min to 500 °C and hold it at that temperature for 3 h. (Some carbonate may remain in the material, depending on Mg/Al ratio etc.)
2. Allow the calcined material to cool naturally in the furnace until a furnace temperature of ~150 °C is reached (make sure that *T* is always >105 °C).
3. Remove the crucible with the material from the furnace and immediately transfer it to a desiccator with dry silica gel; allow the material to cool to room temperature in the desiccator.
4. **[glove box]** Immerse the material in 1 M NaOH solution (see table below for the composition of the solution), at a concentration of ~1 g of solid per 10 ml of solution.

	Concentration (M)	Volume (ml)	Weight of solute (g)
NaOH	1.0	200	7.9994

5. Aging of the suspension in a sealed bottle for 20–24 hours.
6. **[glove box]** Vacuum filtration (using “Blauband” filter paper, *i.e.*, slow filter paper) and washing with ultrapure water at least five times.
  - a. Dry filter residue in a desiccator in a CO<sub>2</sub>-free atmosphere **[Ar or N<sub>2</sub>]** above saturated LiCl solution (11 % RH) for 3 weeks.

If necessary for subsequent analyses/experiments, grind the material **[in a glove box under Ar or N<sub>2</sub> atmosphere]**. Store the material in sealed containers until required for analyses/experiments.