SULFATE LEACHING FROM RECYCLED AUTOCLAVED AERATED CONCRETE IN FLOOR SCREEDS IS CONTROLLED BY ETTRINGITE SOLUBILITY

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Abstract

Autoclaved aerated concrete (AAC) is a lightweight cellular concrete. Recycling AAC in concrete or unbound applications may cause problems because of high amounts of leachable sulfate. This study evaluates the recycling of AAC demolition waste as a replacement of sand in floor screed. The cement binder reacted with sulfate released from the AAC waste to form ettringite. Sulfate release was in line with ettringite solubility control and below leaching limits defined by Dutch environmental legislation. High pH conditions are necessary to avoid excessive sulfate leaching. Pollution of AAC waste with gypsum impurities was found to be detrimental to sulfate immobilisation.

Highlights

- Ettringite solubility controls sulfate leaching from products with recycled AAC.
- High pH conditions secure sulfate immobilisation.
- Gypsum pollution of recycled AAC is detrimental to sulfate immobilisation.
- Recycled AAC aggregate can replace 40% of the sand fraction in floor screed.

Keywords: Autoclaved aerated concrete; sulfate leaching; recycling; immobilisation

1 Introduction

Autoclaved aerated concrete (AAC) is a lightweight porous building material with a density of 400-800 kg/m^3 and thermally insulating capacities (0.1-0.2 W/mK) [1,2]. AAC is commonly manufactured using combinations of lime and Portland cement mixed with either finely ground quartz sand or Class F fly ash [1]. These raw materials are mixed with water and aluminium powder, which causes the production of H_2 due to the oxido-reduction of the aluminium powder. This reaction is enhanced by the elevated pH of the cement pore solution. The generation of H_2 gas leads to an overall volume expansion and results in the creation of spherical pores where gas remains trapped inside the cement matrix [2]. This way a highly porous, cellular, material is produced. Calcium sulfate (2-5 wt%), in the form of gypsum or anhydrite, is added to the raw materials to facilitate the formation of more crystalline calcium silicate hydrate phases (particularly tobermorite). This results in higher strength and an end product less susceptible to shrinkage and carbonation [3,4,5].

Because of its high porosity and low energy and material consumption [6], AAC can be seen as a sustainable building product. However, the recycling of AAC demolition waste still remains a challenge. AAC aggregate has a lower compressive strength (1-9 MPa) [1] than other stony materials in construction and demolition waste (C&DW). Moreover, the chemical composition of AAC aggregate can cause technical and environmental problems in traditional recycling applications for the stony fraction of C&DW (e.g. the use in foundations). AAC contains an average 12,600 mg/kg dm of leachable sulfate [7], which can lead to technical problems (e.g. efflorescence and internal sulfate attack) in building materials [8,9,10] and ecotoxicological effects (salinity, sulfide formation, eutrophication) caused by leaching in groundwater [11].

Since AAC demolition waste cannot be recycled in the applications that are used for the stony fraction of C&DW (mostly unbound foundations), it is labelled as a “problem fraction” by the Waste Agency of Flanders OVAM. Currently, it is possible to use AAC aggregate as a replacement for the sand fraction in the production of new AAC. However, this replacement is generally limited to 20% of the sand fraction and to the use of AAC production and construction waste. AAC demolition

52 waste can contain impurities, resulting in visual contaminations and problems in the AAC production
53 process [12].

54 Zaetang et al. (2013) investigated the use of AAC as a lightweight aggregate in pervious concrete.
55 The use of AAC aggregate strongly reduced (3-4 times) the density and thermal conductivity
56 compared with pervious concrete produced with natural aggregates [13]. The effects of sulfate
57 leaching were not investigated.

58 Schoon et al. (2013) investigated the use of AAC waste as an alternative raw material for Portland
59 clinker production. Use in this application was possible, but showed several difficulties (e.g. bound
60 H_2O, SiO_2 content and milling costs, impurities) [14]. In the study of Karczmarczyk et al. (2014) [15],
61 AAC aggregate (1-6 mm) was tested as a green roof substrate. The high water absorption and P-
62 removal capacity of AAC aggregate and its removal efficiency in low phosphorus concentrations
63 make it an interesting material for this application. However, AAC aggregate has a negative impact on
64 the water environment because of its sulfate release. Other recycling options for AAC waste include
65 the use of AAC aggregate as oil absorbent or (low-grade) filler for cat litter boxes [12].

66 To create sufficient viable recycling routes for AAC waste, the problem of sulfate leaching needs to
67 be solved, for instance by immobilisation of the leachable sulfate by chemical binding in cement
68 hydration products. Brouwer et al. (2000) described a method for chemically immobilizing sulfate
69 from screening sands, which contain up to 6 wt% sulfate, by the use of Portland cement [16]. In this
70 approach the sulfate is captured by reaction with $3CaO.Al_2O_3$ (C_3A) from the cement to form
71 ettringite. The C_3A content in Portland cements is limited to a few percent (3-8 wt%), and sulfates in
72 the form of gypsum or anhydrite are already added to control the setting of the cement. Therefore,
73 the uptake potential of additional sulfate by regular Portland cement is rather limited. In addition, too
74 high dosages of sulfate (oversulfation) effectively delay the hydration of the clinker phases and thus
75 the strength development [17,18,19].
76

As an alternative, Ambroise and Péra (2004, 2008) treated demolition waste containing calcium sulfate by means of calcium sulfo-aluminate (CSA) clinker. Here, depending on the initial sulfate dosage of the cement, a much higher uptake potential can be achieved by reaction with the main ye'elimite [$Ca_4(AlO_2)_6SO_4$] component. They showed that calcium sulfate was entirely consumed when the ratio of CSA to calcium sulfate was 4:1 or higher [20,21].

Sulfate dosing into CSA or calcium aluminate cements (CAC) is commonly used to control setting and effect volume expansion [22,23]. However, too high gypsum contents lead to uncontrolled volume expansion, cracking and failing of the hardened cement. This is likely controlled by the build-up of crystallisation pressure by ettringite in micropores [24]. Overall, at present sulfate levels in cementitious materials are being closely monitored and maximum levels are in place to avoid negative effects such as slow hardening, dimensional instability or cracking. In the future, product design and manufacturing will also need to take into account after-life reuse and recycling opportunities. Meeting environmental regulations will be one of the prime requirements to enable high-value recycling opportunities.

In this respect, this paper evaluates the recycling of AAC as fine aggregate (0-8 mm) in floor screed formulations made of Portland and blended cements in view of the Dutch sulfate leaching regulations [25]. This application was chosen because of low compressive strength requirements. Sulfate immobilisation mechanisms are described and used to establish product boundary conditions.

2 Materials and methods

2.1 Phase composition of AAC

The mineralogy of the AAC samples was examined by X-ray powder diffraction (XRD) analysis using a Philips X'Pert Pro diffractometer, equipped with a Cu anode X-ray tube operated at 40 kV and 35 mA and using an automatic divergence slit. Specimens were scanned from 2 to 120° (2θ), with a step size of 0.04° and a dwell time of 4 seconds per step.

2.2 Floor screed formulations with AAC waste

First, AAC was crushed with a disk mill to below 8 mm. Second, floor screed mixtures were produced with AAC aggregate (0-8 mm, 530 kg/m³) and C&DW mixed recycled aggregates (0-8 mm, 800 kg/m³, produced by an industrial C&DW crushing installation), using different types of cement (CEM I, CEM II, CEM III; 140 kg/m³). Tap water (280 l/m³) was added and the materials were mixed in a concrete mixer. The mixtures were poured in cylindrical moulds (h = 12 cm, d= 10 cm), unmoulded after 24 h and further cured for 28 days at 20 °C, 100% relative humidity before further testing.

The mechanisms of sulfate immobilisation (e.g. pH dependency) in the floor screed formulations were studied on samples with pure AAC aggregate. In a second stage, to study the effect of gypsum contamination, formulations were prepared with aggregates from crushed AAC demolition waste.

2.3 Total sulfur

The total sulfur content of the floor screeds was analysed with inductively coupled plasma atomic emission spectroscopy (ICP-AES) (ISO 11885) after crushing (<100 µm) and closed microwave digestion with $HCl/HNO_3/HF/H_3BO_3$.

2.4 Sulfate leaching

The leaching of sulfate from the AAC aggregate, mixed recycled aggregates and cement stabilized sand products was analysed using batch leaching tests. The European EN 12457-2:2002 compliance test for leaching of granular waste materials and sludges was carried out.

The materials (<4 mm) were brought in contact with the leaching liquid (deionized water, L/S = 10) with the aid of an overhead mixer (5 turns/min.). This method is based on the assumption that a state of equilibrium (or near-equilibrium) is reached between the liquid and the solid phases during the test period (24 h). After 24 h, the solid residue was separated from the liquid by filtration (size filter pores: 0.45 µm). The pH of the leaching liquid was measured immediately after filtration. The sulfate concentration of the leaching liquid was measured by liquid chromatography and conductivity detection (ISO 10304-1:2007).

2.5 Speciation-solubility modelling

In a first stage, speciation-solubility modelling was carried out using Visual MINTEQ 3.1, using updated and expanded data from the NIST Critical Stability Constants database, to determine the solubility of ettringite as a function of pH. In the model, ettringite was assumed to be the infinite solid phase. Diaspore and gypsum were added to the calculations as possible precipitation products. The pH was varied between 10.5 and 12.5. The ionic strength of the solution was calculated from the elements in solution.

In a second stage, the fate of sulfate released from the AAC by (partial) reaction with or leaching to the cement matrix was modelled. Thermodynamic modelling of the cement hydrate assemblage was carried out using the Gibbs free energy minimization programme GEMS v3.2. GEMS calculates the equilibrium phase assemblage and solution speciation at the defined input composition. The thermodynamic database used was the PSI/Nagra 12/07 general database for aqueous species and common solids [26]. This was supplemented with the cemdata14 database for cement hydrates [27,28]. The effect of sulfate on the cement hydrate assemblage and pore solution composition and the sulfate uptake threshold was evaluated for CEM I floor screed formulations. The Portland cement was taken to be fully hydrated. Averaged literature compositions for Portland cement and AAC waste were adopted [14,29].

2.6 Electron microscopy

The microstructure of the floor screed products was investigated by electron microscopy. Representative samples were cut from the centre of the hardened floor screed specimens and vacuum-impregnated using a low-viscosity epoxy resin. Polished sections of the impregnated samples were prepared by gradually polishing the exposed sample surface down to 1 µm diamond grit size. A water-free polishing lubricant was used to avoid artificial reactions with the freshly exposed surface. The polished sections were Pt-coated preceding microscopic analysis. A FEI Nova NanoSEM 450 electron microscope equipped with field emission gun was operated at 15 kV acceleration voltages. Images were acquired in backscattered electron mode at a working distance of 6.2 mm.

Energy dispersive spectroscopy (EDS) point analyses and multispectral element mappings were acquired in order to study the impact of gypsum addition on the binder chemistry. Specific attention was paid to the distribution of sulfur in the cement matrix.

3 Results and discussion

3.1 Raw material characterisation

The phase composition of AAC demolition waste was measured by XRD (Figure 1). The AAC aggregate mainly contains quartz [SiO_2], 11Å-tobermorite [$Ca_5Si_6O_{16}(OH)_2.4H_2O$], calcite [$CaCO_3$], vaterite [$CaCO_3$] and gypsum [$CaSO_4.2H_2O$]. No crystalline calcium-aluminate hydrate phases (e.g. ettringite, AFm phases) were found. The average chemical composition for AAC production waste from the Flemish AAC factory (Xella) is given in Table 1 [14]. Xella represents more than 90% of the Flemish market. Table 1 also gives the typical Portland cement compositions used in the thermodynamic modelling of the cement hydrate assemblages. It is important to note that the AAC shows higher SO_3 and lower Al_2O_3 content when compared to Portland cement. This results in the presence of unbound gypsum in the AAC matrix and renders the material liable to sulfate leaching.

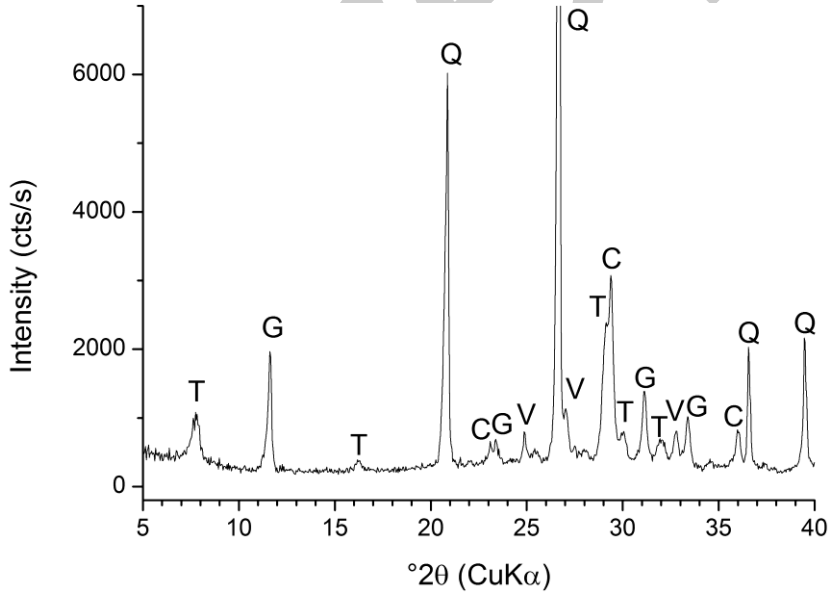

Figure 1. XRD pattern of the AAC aggregate from demolition waste. The main reflections are assigned to tobermorite (T), gypsum (G), quartz (Q), vaterite (V) and calcite (C).

171 Table 1. Chemical composition of the AAC aggregate (average of production waste, Schoon et al. [14]) and
172 Portland cement (averaged compositions used in thermodynamic modelling [29])

Oxide	AAC	Portland cement
	wt. %	
SiO_2	53.90	19.51
Al_2O_3	1.93	5.37
Fe_2O_3	0.68	2.96
CaO	28.47	63.15
MgO	0.36	1.57
K_2O	0.48	1.00
Na_2O	0.20	0.25
SO_3	2.10	4.13
TiO_2	0.08	0.26
P_2O_5	0.10	0.14
CO_2	3.29	0.54

173

174 The leaching of sulfate by dissolution of soluble gypsum is investigated by sulfate leaching testing.
175 Sulfate concentrations in the leachates of the pure AAC aggregate and the mixed recycled aggregates
176 were 11,000 and 2,400 mg/kg dm, respectively. For both materials this is above the Dutch limit value
177 of 1,730 mg/kg dm, which prevents direct reuse. Table 2 compares these leaching values to literature
178 reports [7,30,31]. The results here are in line with previous tests obtained by similar experimental
179 procedures.

180

Table 2. Sulfate leaching of the raw materials and comparison with literature data.

Material	pH	Sulfate leaching sample (mg/kg dm)	Sulfate leaching literature (mg/kg dm)	Reference
AAC aggregate	11.6	11,000	12,600	Lang-Beddoe & Schober (1999) [7]
Mixed recycled aggregates	9.3	2,400	1,040	OVAM (2006) [30]
			390-4,700	Vrancken & Laethem (2000) [31]

3.2 Sulfate leaching of the floor screed products

3.2.1 Sulfate leaching and pH

The results of the batch leaching tests on the floor screed samples with pure AAC aggregate are shown in Figure 2. Clearly, sulfate leaching increased with decreasing leachate pH over the pH range of 12.5 to 10.5. This pH dependency of sulfate leaching is in line with the modelled ettringite solubility (Fig. 2). A second observation is that floor screeds made of blended cements (CEM II, CEM III) are less resistant to sulfate leaching. Apparently, the blended cements show a lower pH buffering capacity than CEM I Portland cement and a lower leaching solution pH is established over the duration of the leaching test.

The observation that formulations containing blended cements are less resistant to sulfate leaching can be explained by the occurrence of a pozzolanic reaction in the blended cements. The pozzolanic reaction of fly ash and slag may consume readily accessible $Ca(OH)_2$ and thus partially deplete the alkalinity of the cement. However, comparison to other experimental studies on blended cement hydration [32,33] learns that it is improbable that all portlandite in the system is consumed over the relatively short times of hydration experienced by the floor screed products (7 days). Similarly, if portlandite would still be present in the system, thermodynamic calculations indicate that a pH of around 12.4 should be established in the leaching solutions.

200 The observed low pH indicates that the establishment of a thermodynamic equilibrium may be
201 kinetically hindered or affected by ongoing reactions such as the pozzolanic reaction. Matschei and
202 Glasser (2011) [34] investigated cement hydrate equilibration times in water and concluded that for
203 a substance to act as a buffer it should be available in sufficient quantity and at high reactivity
204 (e.g. high exposed surface area to the solution) and should dissolve sufficiently fast to quickly reach
205 equilibrium. In case of portlandite, dissolution was found to be sufficiently fast to reach equilibrium
206 over a time span of a few minutes [34].
207 Therefore, the low pH encountered in the leaching solutions of the blended cement products cannot be
208 explained by slow portlandite dissolution. Rather, high pH and portlandite saturation may not be
209 reached because of occlusion of portlandite by surrounding pozzolanic reaction products (reduction of
210 reactive surface area), combined with an ongoing pozzolanic reaction during leaching (competitive
211 reaction). The latter effect is common in blended cements, portlandite undersaturation and a reduction
212 in alkalinity are common features [29,32]. Moreover, the evaluation of portlandite saturation in
213 a solution contacted with a hydrating cement lies at the base of the EN 196-5 pozzolanicity test
214 (Frattini test), where undersaturation is used to diagnose the occurrence of a pozzolanic reaction.
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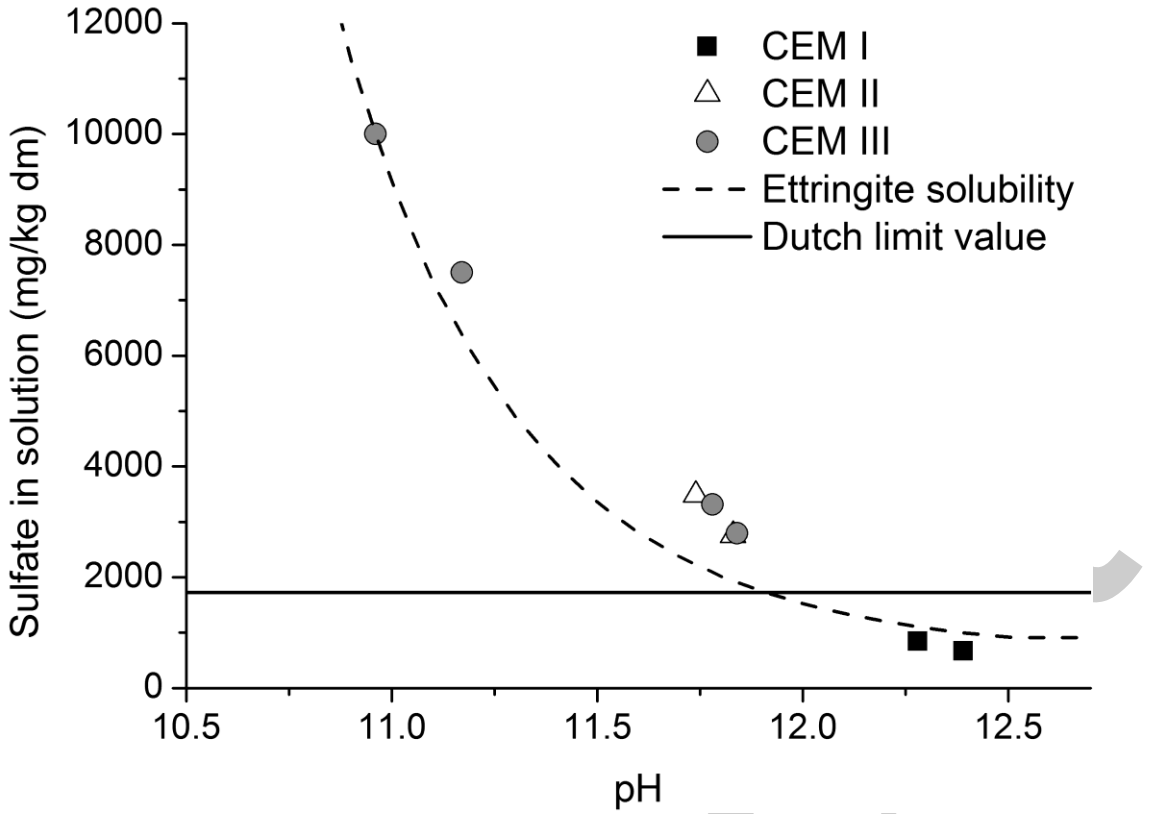
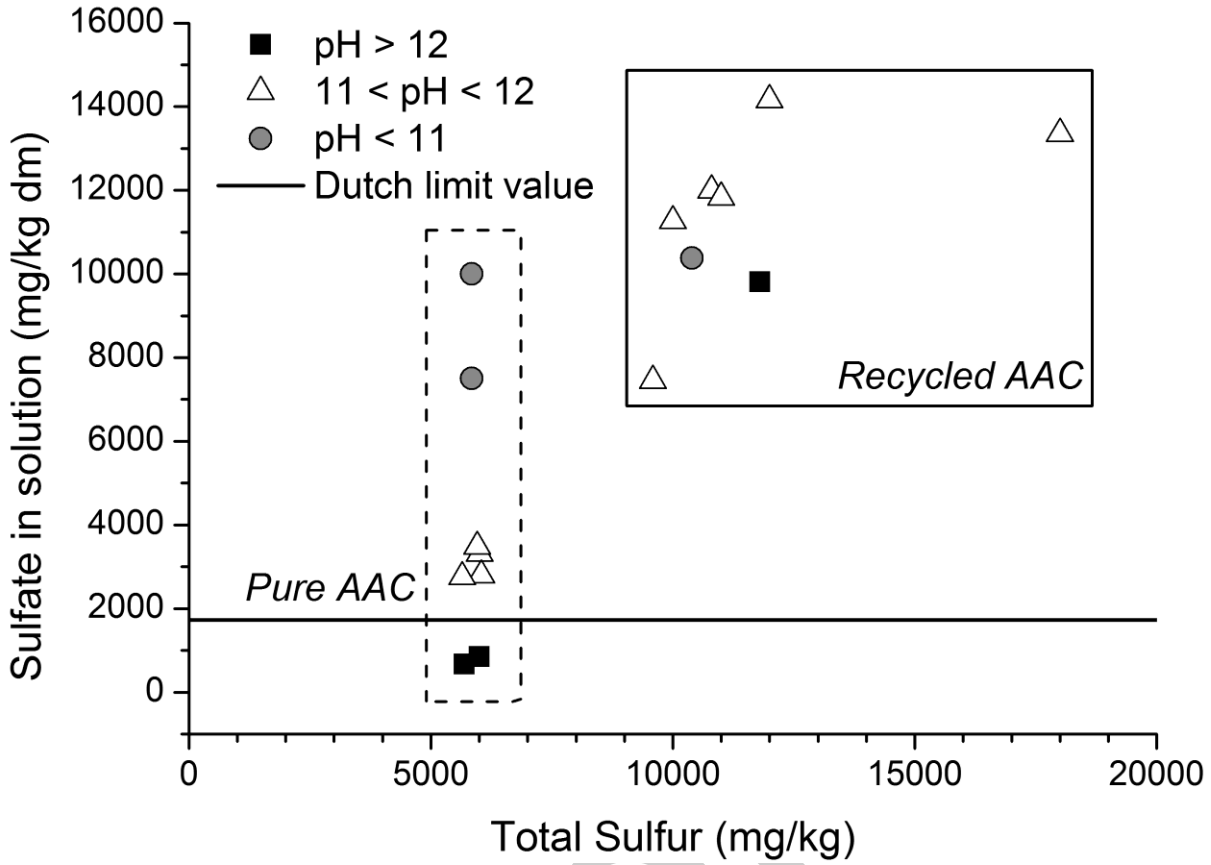


Figure 2. Sulfate leaching of floor screed products (curing: 7 days) with pure AAC aggregate as a function of the pH of the leachate (L/S = 10).

3.2.2 Effect of gypsum contamination on sulfate leaching

In a second phase, AAC aggregate from demolition waste, containing gypsum contamination, was used for the production of floor screed products. Figure 3 shows sulfate leaching as a function of total sulfur content of the floor screed products (both with pure AAC aggregate and AAC aggregate from demolition waste). It is inferred that in the present formulation, floor screed products having high total sulfur contents experience strong sulfate leaching. This means that in case gypsum contaminated AAC aggregate is used, the total sulfur content of the system increases significantly and the immobilisation capacity by ettringite formation can become exhausted. Superimposed on the pH effect, there appears to be a total sulfur threshold beyond which sulfate can no longer be bound by formation of ettringite.



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230 Figure 3. Sulfate leaching of the floor screed products (curing: 28 days) as a function of the total sulfur content.
231 The leachate results are subdivided by solution pH.

232

233 Thermodynamic modelling of sulfate uptake by a CEM I based floor screed supports the concept of
234 a sulfate uptake threshold that should not be exceeded. Figure 4 gives the modelled Ca and sulfate
235 concentrations in the leachate as a function of the reactive gypsum present in the AAC waste for a
236 CEM I based floor screed.

237 The modelling predicts sulfate leaching to comply with the limit value as long as reactive
238 gypsum/sulfate can be converted into ettringite by reaction with hemicarbonate, monocarbonate or
239 hydrotalcite phases. In case excess reactive gypsum/sulfate is present, sulfate concentrations in the
240 leachate are predicted to increase steeply and exceed the limit value. Since gypsum equilibrates
241 relatively fast (few minutes) with a leaching solution [34], it will control sulfate concentrations
242 through its solubility product. This is consistent with our experimental observations that suggest
243 noncompliant sulfate leaching in the presence of excess gypsum.

Thus, to enable the reuse of AAC waste in cementitious products it is crucial to avoid the intake of gypsum during recycling. In addition, reducing the free gypsum level in the AAC product itself will lower the risk of exceeding the sulfate uptake threshold. A maximum total sulfur content of the product, $S_{tot,max}$ [mg/kg dm], for compliance with sulfate leaching regulations can be calculated by mass balance, assuming that 1) all sulfur reacts with the binder, and 2) Al-ettringite is the ultimate cement hydrate sulfate sink, as:

$$S_{tot,max}\ [mg/kg\ \ dm] = 9435\ X_{bind}\ ((Al_2O_3)_{bind}\ [wt\%\ ])$$

where X_{bind} corresponds to the weight fraction of the cement binder in the final product, and $(Al_2O_3)_{bind}$ identifies with the Al_2O_3 content in wt.% of the cement binder. Since some of the sulfur will be taken up into the C-S-H cement hydrate [34], the actual sulfate uptake limit will be somewhat higher. However, equation 1 is useful and can serve as a first, safe indication of maximal sulfur levels for recycled products.

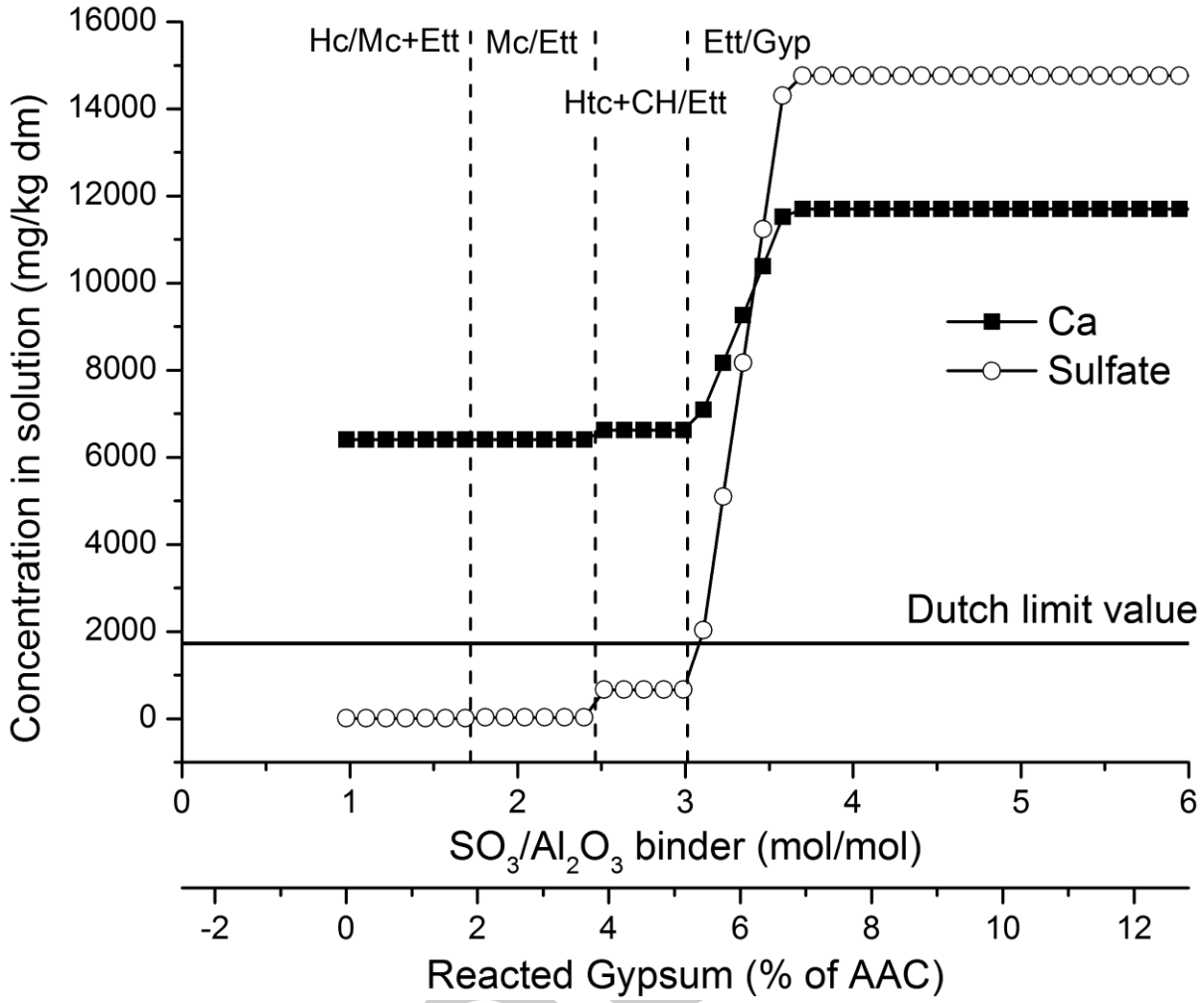

Figure 4. Modelled effect of reactive gypsum and sulfate additions on Ca and sulfate concentrations in the leachate of a CEMI floor screed product (L/S = 10). No distinction is made between gypsum from the AAC itself or from contaminations included during recycling. Thermodynamic equilibrium is assumed and sulfate concentrations are controlled by binder chemistry and the respective hydrate buffering pairs: hemicarbonate/monocarbonate + ettringite (Hc/Mc+Ett), monocarbonate/ettringite (Mc/Ett), hydrotalcite + portlandite/ettringite (Htc+CH/Ett), ettringite/gypsum (Ett/Gyp). The Dutch leaching limit value is exceeded once extra gypsum can no longer be bound into ettringite.

3.3 Microstructure

The microstructure of floor screeds with and without gypsum contamination was investigated by electron microscopy. Given the high amount of aggregates in the floor screeds, microanalysis of the chemistry selected areas by EDS was preferable over bulk characterization techniques such as XRD.

Figures 5a-c show a selection of representative backscattered electron images of floor screeds contaminated with gypsum particles. Gypsum aggregate particles were clearly observed as porous aggregates of elongated gypsum crystals (Fig 5c). The outer rim of the gypsum grains was often observed to be very porous and ill-defined. The latter features are indicative for an interfacial reaction between the gypsum and the cement. On the other hand the core of the gypsum particles appears undisturbed and high in S (Fig. 5d). The persistence of gypsum in floor screed samples hydrated for 28 days indicates that gypsum is stable and that the Al_2O_3 reservoir of the cement is depleted, as supported by excessive levels of sulfate in the leachate solutions of the corresponding samples (Figure 3).

Contrary to gypsum particles in direct contact with the cement matrix, the gypsum contained in the AAC particles do not show signs of reaction (Fig. 5b). Enclosure within the AAC aggregate particles drastically reduces the available reactive surface area and exposure to the cement pore solution. Moreover, if contamination of the AAC waste by external gypsum sources (e.g. plaster boards) is excluded, than the rather small amounts of gypsum in the AAC itself can be easily contained by reaction with the Portland cement.

The chemical composition of the cement matrix in both gypsum-contaminated and gypsum-free 28 days hydrated floor screeds was investigated by EDS microanalysis. Points were selected within the cement matrix to verify the S content, and for the gypsum contaminated samples, within the (former) gypsum grains to check for formation of cement hydrate phase. Figure 6 shows the EDS results in a S/Ca vs Al/Ca plot. This plot enables to distinguish between sulfur bearing phases such as gypsum, ettringite and monosulfate. In the reference sample the EDS results scatter around the trend lines between C-S-H and regular cement hydrates such as ettringite and monosulfate. In the gypsum contaminated samples, the EDS points taken in the cement matrix are shifted upwards to higher S/Ca levels, indicating a change in the hydrate assemblage towards the co-occurrence of ettringite and gypsum.

The thermodynamic modelling results in section 3.2.2 indicate that this shift coincides with a strong increase in sulfate concentrations in solution and excessive leaching. The EDS point analyses taken within the gypsum particles do not show the presence of cement hydrates containing either aluminates or silicates. This is to be expected since sulfate is (much) more soluble than either aluminate or silicate. Sulfate thus diffuses out from the gypsum particles into the cement matrix.

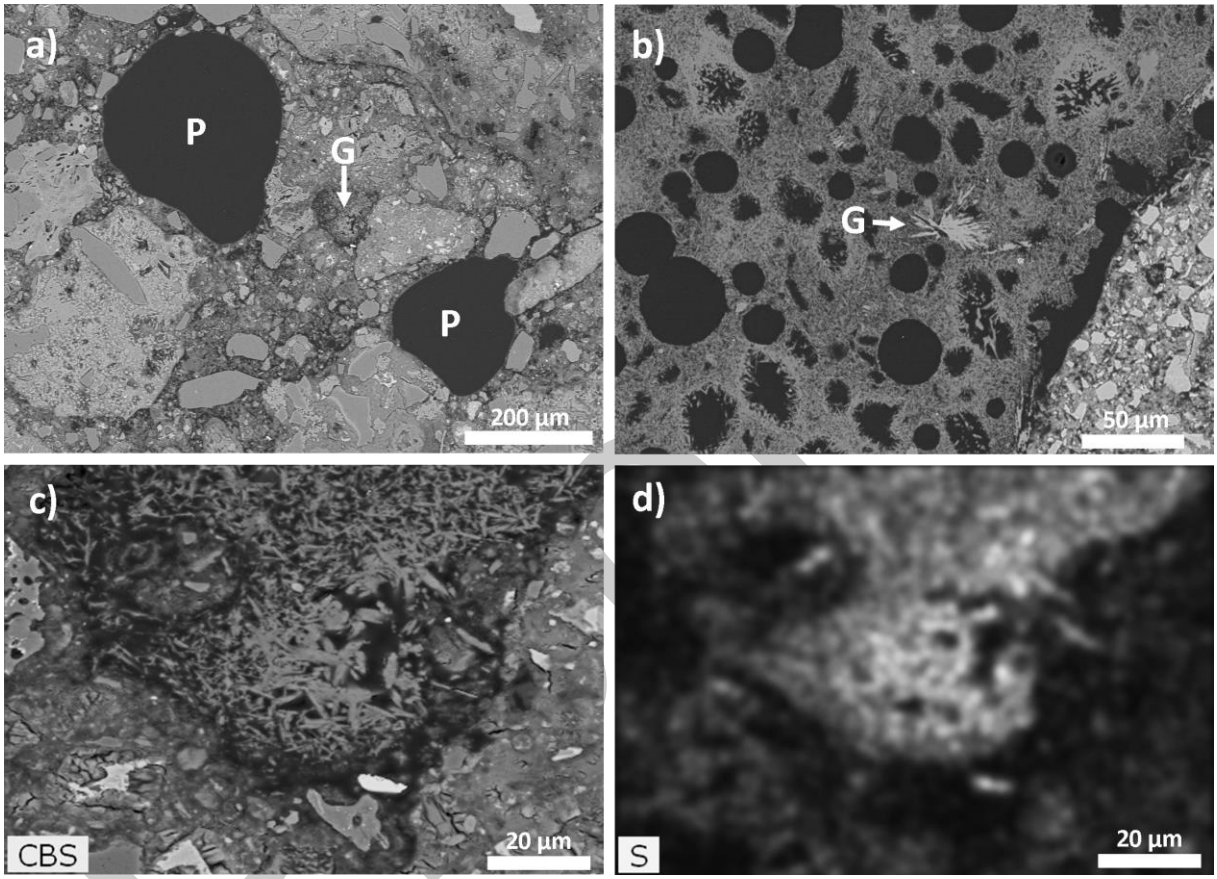

Figure 5. Electron microscopy images of floor screeds made with crushed AAC demolition waste and mixed C&DW mixed recycled aggregates. The floor screeds contained gypsum impurities and were found to exceed sulfate leaching regulations. a) Overview image showing large pores (P) and an unreacted gypsum grain (G) embedded in the floor screed matrix. b) Close-up on AAC aggregate grain, gypsum crystals enclosed within the AAC are marked. c) Gypsum grain indicated in a). d) S element mapping of the area in c).

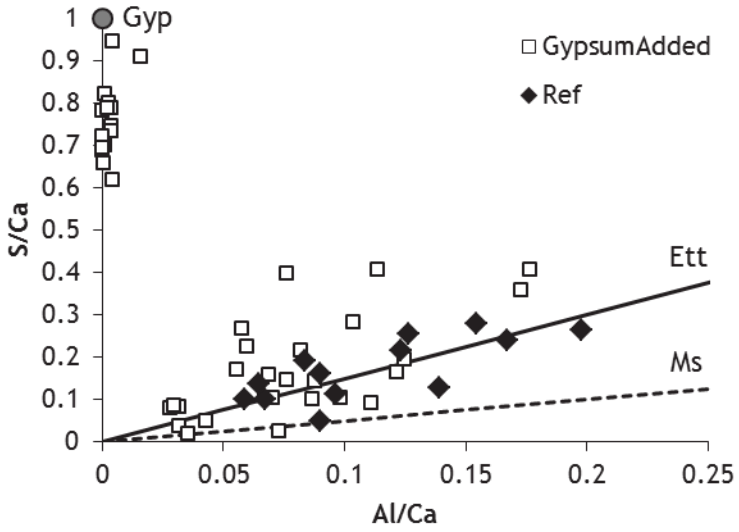

Figure 6. EDS point analyses plotted as S/Ca vs. Al/Ca for a reference floor screed made containing no gypsum particles and a floor screed mix in which gypsum particles were identified and sulfate leaching was off-limits (cf. Fig. 5). Both samples were cured for 28 days. Average S/Ca ratios of the cement matrix were higher in the gypsum (Gyp) containing samples indicating a shift in the hydrate assemblage from monosulfate (Ms) + ettringite (Ett) to Ett+Gyp.

4 Conclusions

This research shows that AAC demolition waste can be recycled as fine aggregate (0-8 mm) in floor screed formulations, creating a new valorisation route for a waste stream that currently still is being landfilled. The reaction of the cement binder with the soluble sulfate from the AAC waste caused the formation of insoluble ettringite, hereby strongly reducing sulfate leaching. Two conditions are crucial for a secure sulfate immobilisation, namely high pH conditions (>12) and very low gypsum levels contamination in the recycled AAC waste.

The pH dependency of sulfate leaching is in line with the modelled ettringite solubility. Sufficiently high pH conditions can be obtained by the use of CEM I. Blended cements show a lower pH buffering capacity due to the occurrence of pozzolanic reactions.

When the AAC waste is contaminated with gypsum (e.g. from plasterboards), the immobilization capacity by ettringite formation can become exhausted. These observations from leaching experiments

are confirmed by thermodynamic modelling and SEM analysis (EDS and multispectral element mappings) of the microstructure.

As a final conclusion, with the use of CEM I and a good acceptance policy of AAC waste, it is possible to successfully recycle AAC demolition waste in a high-grade construction product.

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