



Recycling of autoclaved aerated concrete in floor screeds: Sulfate leaching reduction by ettringite formation



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HIGHLIGHTS

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

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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.

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1. Introduction

Autoclaved aerated concrete (AAC) is a lightweight porous building material with a density of 400–800 kg/m³ and thermal insulating capacities (0.1–0.2 W/m K) [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 aluminum powder, which causes the production of H₂. The generation of H₂ gas leads to an overall volume expansion and results in the creation of spherical pores [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 a product less susceptible to shrinkage and carbonation [3–5].

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 on 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 [8–10] and ecotoxicological effects (salinity, sulfide formation, eutrophication) caused by leaching to 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 labeled 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 uncontaminated AAC production and construction waste. AAC demolition waste can contain impurities, resulting in visual contaminations and problems in the AAC production process [12].

Zaetang et al. [13] investigated the use of AAC as a lightweight aggregate in pervious concrete. The use of AAC aggregate strongly reduced (3–4 times) the density and thermal conductivity com-

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pared with pervious concrete produced with natural aggregates [13]. The effects of sulfate leaching were not investigated. Other recycling options for AAC waste include the use as raw material for Portland clinker production, as oil absorbent or (low-grade) filler for cat litter boxes [12,14].

To create sufficient viable recycling routes for AAC waste, the problem of sulfate leaching needs to be solved, for instance by immobilisation of the leachable sulfate by chemical binding in cement hydration products. Brouwer et al. [15] described a method for chemically immobilizing sulfate from screening sands, which contain up to 6 wt% sulfate, by the use of Portland cement [15]. In this approach the sulfate is captured by reaction with $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ (C_3A) from the cement to form ettringite. The C_3A content in Portland cements is limited to a few percent (3–8 wt%), and sulfates in the form of gypsum or anhydrite are already added to control the setting of the cement. Therefore, the uptake potential of additional sulfate by regular Portland cement is rather limited. In addition, too high dosages of sulfate (oversulfation) effectively delay the hydration of the clinker phases and thus the strength development [16–18].

As an alternative, Ambroise and Péra [19,20] treated demolition waste containing calcium sulfate by means of calcium sulfoaluminate (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 [$\text{Ca}_4(\text{AlO}_2)_6\text{SO}_4$] component. They showed that calcium sulfate was entirely consumed when the ratio of CSA to calcium sulfate was 4:1 or higher [19,20].

Sulfate dosing into CSA or calcium aluminate cements (CAC) is commonly used to control setting and affect volume expansion [21,22]. 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 crystallization pressure by ettringite in micropores [23]. 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.

Boehme [24] showed that the mechanical properties of screed formulations consisting of sand, cement and up to 25% of AAC aggregate comply with the requirements for use as a traditional screed in houses [24]. 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 s 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/m^3) and C&DW mixed recycled aggregates (0–8 mm, 800 kg/m^3 , produced by an industrial C&DW crushing installation), using different types of cement (140 kg/m^3) (CEM I, CEM II/B-M (S-V) or CEM III/A). Tap water (280 l/m^3) was added and the materials were

mixed in a concrete mixer. This extra amount of water was added because of the water absorption of the crushed AAC. The mixtures were poured in cylindrical molds ($h = 12\text{ cm}$, $d = 10\text{ cm}$), unmoulded after 24 h and further cured 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, containing gypsum contamination.

2.3. Total sulfur

The total sulfur content of the floor screeds was analyzed with inductively coupled plasma atomic emission spectroscopy (ICP-AES) (ISO 11885) after crushing ($<100\text{ }\mu\text{m}$) and closed microwave digestion with $\text{HCl}/\text{HNO}_3/\text{HF}/\text{H}_3\text{BO}_3$.

2.4. Sulfate leaching

The leaching of sulfate from the AAC aggregate, mixed recycled aggregates and cement stabilized sand products was analyzed 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 ($<4\text{ mm}$) 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: $0.45\text{ }\mu\text{m}$). 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 modeling

In a first stage, speciation-solubility modeling 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 modeled. Thermodynamic modeling 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 center 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 $1\text{ }\mu\text{m}$ 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 characterization

The phase composition of AAC demolition waste was measured by XRD (Fig. 1). The AAC aggregate mainly contains quartz [SiO_2], 11 Å-tobermorite [$\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2\cdot 4\text{H}_2\text{O}$], calcite [CaCO_3], vaterite [CaCO_3] and gypsum [$\text{CaSO}_4\cdot 2\text{H}_2\text{O}$]. No crystalline calcium-

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