



Reaction products and strength development of wastepaper sludge ash and the influence of alkalis



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ABSTRACT

Wastepaper sludge ash (WSA) from a newsprint paper mill was investigated for its mineralogical composition and its reaction products and strength development after activation with water and sodium and potassium hydroxide solutions. The results showed the WSA to consist of calcite, free lime, gehlenite, tricalcium aluminate, belite, talc, quartz and probably a glassy phase. The principle reaction product was monocarboaluminate ($\text{CO}_3\text{-AFm}$) for the water- as well as for the alkali-activated WSA. Formation of monocarboaluminate and strength gain was more rapid for the alkali-activated WSA until 1 day of curing. However, afterwards reactions proceeded much slower when alkali solutions were used, leading to an about twice as high compressive strength for water-activated WSA mortars after 28 days of curing. The observed behavior is tentatively ascribed to a less uniform microstructure of the alkali-activated WSA. Significant differences between NaOH- and the KOH-activated WSA were not observed.

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1. Introduction

In paper recycling, several types of waste sludge are produced; these sludges contain organics as well as inorganic fillers of the recycled paper (kaolin clay, calcium carbonate, talc, titanium dioxide). Part of them is reused in cement and ceramics industry, for soil improvement etc. [1]; a recent attempt is to produce metakaolin from the sludge by controlled calcination [2–4]. However, a large fraction of the sludge is still incinerated, which serves to recover energy and particularly to reduce the volume of waste material to be handled. The ashes generated in this process, often referred to as wastepaper sludge ash (WSA), differ widely in their chemical and mineralogical composition, depending on the composition of the feedstock, the production and waste treatment processes and the conditions of combustion [1,5]. Despite the volume reduction by incineration, the amount of waste, i.e. WSA, is still very large, which poses a significant problem to the paper industry, if no added-value application can be found for the WSA, i.e., if it has to be landfilled or otherwise handled entailing costs.

In this context, several studies investigated the use of WSA as cementitious material, either alone, in blends with Portland cement or mixed with other binder materials [6–13]. These studies confirmed that WSA generally possesses cementitious properties. Thus, in principle it may be used as binder or as supplementary

cementitious material, which would not only reduce the amount of waste from paper industry to be landfilled, but would also contribute to lowered anthropogenic carbon dioxide emissions by partial replacement of Portland cement. However, only few studies [6,12,13] provided information about which compounds formed on hydration of WSA or blends containing WSA. Since the phase assemblage of the hardened pastes determines their mechanical properties and durability, it is essential to provide more data on the evolution of the reaction products, including the influence of specific compounds, which may be introduced intentionally or contained in the cementitious materials, used for mixing with the WSA. The present work aims to contribute to these issues by analyzing a WSA for strength development and reaction products after addition of water as well as after alkali-activation with sodium hydroxide and potassium hydroxide solutions.

2. Materials and methods

2.1. Starting materials

The wastepaper sludge ash (WSA) used as starting material in the present study was collected from a paper mill that produces newsprint, using exclusively recovered paper as raw material. It is a mixture of bottom ash and fly ash generated in co-combustion of sludge and smaller amounts of rejects from the recycling process and wood biomass in the fluidized bed of an on-site co-generation power station. Its main oxide composition and loss on ignition (LOI) are shown in Table 1. The fractions of CO_2 and free lime

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Table 1
Chemical composition of the WSA.

Oxide	Amount in %
SiO ₂	22.33
Al ₂ O ₃	11.97
Fe ₂ O ₃	0.59
TiO ₂	0.36
CaO	44.18
MgO	2.42
Na ₂ O	0.24
K ₂ O	0.40
SO ₃	3.64
Cl ⁻	0.45
LOI	13.34

(CaO_{free}) in the WSA were determined to be 11.7% and 10.3%, respectively. The mineralogical composition of the WSA is discussed in Section 3.1.

The WSA is generated and collected as powder, containing agglomerates of considerable size. It was ground shortly (6 s) in a vibratory disc mill to break up larger agglomerates. After this treatment, the particle size distribution of the WSA was: 100% < 351.5 μm, 90% < 125.8 μm, 50% < 16.6 μm, 10% < 1.8 μm (determined by laser granulometry, dispersion in propan-2-ol). The density of the WSA was determined to be 2.66 g cm⁻³.

NaOH and KOH solutions of molar concentration 2 M were prepared from NaOH and KOH pellets (purissimum grade), respectively, and deionized water.

2.2. Paste samples

Pastes were prepared by addition of either deionized water (referred to as water-activated WSA), 2 M NaOH solution, or 2 M KOH solution (jointly referred to as alkali-activated WSA) to the WSA at a volume of liquid/mass of solid ratio of 1.00 cm³ g⁻¹ (equivalent to a water/solid ratio by mass of 1.00 for the water-activated paste). For the alkali-activated pastes this means that the amounts of Na₂O and K₂O in the samples were 6.2% and 9.4% by WSA weight, respectively. At volume of liquid/mass of solid ratios significantly lower than 1.00 cm³ g⁻¹ the pastes were not flowable enough to be mixed and cast without excessive air voids inside.

Mixing of the pastes was done manually. They were cast into cubes of edge length of 20 mm and cured in the moulds (not capped), stored in a closed box over an open water surface at 23 °C. After the desired curing time of 1, 3 or 7 days (and 28 days for the water-activated WSA) the hardened pastes were demoulded and dried by vacuum at a pressure <0.04 Pa and a temperature of 40 °C for 6–9 days (usually 7 days) and then stored in a desiccator over silica gel until required for testing. The mass loss after vacuum drying ranged from 55.0% to 68.6%, depending on activator and sample age.

2.3. Strength testing (paste samples)

For compressive strength determination the paste samples were tested using a 10 kN-testing machine at a loading rate of 240 N s⁻¹. Each strength value reported is the average of the results of five specimens.

2.4. X-ray diffraction analyses

For X-ray diffraction (XRD) analysis, the paste samples were ground in a vibratory disc mill. Powder diffraction patterns were recorded in $\theta/2\theta$ geometry in the range 3–63° 2 θ at 1° min⁻¹ (0.02° steps) [paste samples] or 4° min⁻¹ (0.02° steps) [unhydrated WSA], using CuK α radiation. The X-ray tube operated at 40 kV and

40 mA. The specimen holder was rotated at 30 rpm during measurements to improve particle orientation statistics.

2.5. Thermogravimetric analyses

Thermogravimetric analyses were conducted on WSA and powdered paste samples of approximately 0.30 g at a constant heating rate of 5 K min⁻¹ in flowing nitrogen. Recorded results are expressed as thermogravimetric (TG) and differential thermogravimetric (DTG) curves.

2.6. Mortar samples

For auxiliary strength tests, mortar samples were prepared from another batch of WSA from the same paper mill, with chemical composition (including CO₂ and CaO_{free}) very similar to that of the WSA, used for the tests described above. The WSA was not ground, since it was presumed that mixing together with aggregates would lead at least to a partial destruction of agglomerates.

For each kind of activator and curing time, a set of three prismatic test specimens 40 mm × 40 mm × 160 mm in size were prepared from 1350 g of CEN-standard sand (0–2 mm), 450 g of WSA and 405 cm³ of activator (deionized water, 2 M NaOH or 2 M KOH solution). The presence of aggregates allowed the volume of liquid/mass of WSA ratio to be 0.90 cm³ g⁻¹, i.e. lower than for the paste samples, at an acceptable workability. Mixing of the mortars was done in a mixer according to EN 196-1. The mortars were cast in two layers into steel moulds, vibrating the mould after casting each layer for 45 s. The moulds were covered with a plate of glass and stored in a closed box over an open water surface at 23 °C. After 24 h the specimens were demoulded and either immediately used for 1 day strength tests or put back into the closed box until the desired total curing time of 3, 7 or 28 days. The latter samples were subjected to strength testing directly after removal from the box, i.e. without drying, too. Flexural and compressive strength testing was conducted according to EN 196-1.

3. Results

3.1. Mineralogical composition of the WSA

From XRD analysis calcite (CaCO₃), lime (CaO), portlandite (Ca(OH)₂), talc (Mg₃Si₄O₁₀(OH)₂), gehlenite (Ca₂Al(AlSi)O₇) and quartz (SiO₂) are readily identified as crystalline compounds in the WSA (Fig. 1). In addition, the hump in the diffractogram at about 25–35° 2 θ suggests the presence of an amorphous phase.

The reflection at glancing angle 33.3° 2 θ is attributed to tricalcium aluminate (Ca₃Al₂O₆). This assignment is supported by the fact that the peak disappeared already after one day of hydration

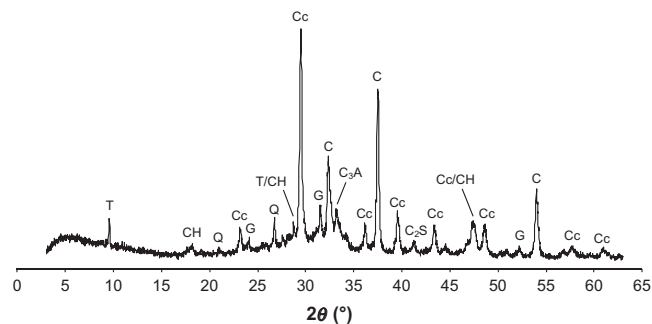


Fig. 1. Powder X-ray diffractogram (CuK α radiation) of the unhydrated WSA. Main peaks are labeled T: talc, CH: portlandite, Q: quartz, Cc: calcite, G: gehlenite, C: lime, C₃A: tricalcium aluminate, C₂S: belite.

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