



Stabilization/solidification of fly ash from fluidized bed combustion of recovered fuel and biofuel using alkali activation and cement addition



Janne Pesonen^{a,*}, Juho Yliniemi^b, Mirja Illikainen^b, Toivo Kuokkanen^a, Ulla Lassi^a

^a Research Unit of Sustainable Chemistry, P.O. Box 3000, 90014, University of Oulu, Finland

^b Research Unit of Fibre and Particle Engineering, P.O. Box 4300, 90014, University of Oulu, Finland

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ABSTRACT

The ability of stabilization and solidification (S/S) of recovered fuel–biofuel fly ash (FA) from fluidized bed combustion (FBC) to immobilize the heavy metals contained therein was investigated. The binders used were Portland cement, an alkali activator (sodium silicate + sodium hydroxide), and a mixture of the two. The leaching behavior, mineralogical composition, microstructure, and compressive strength of the resulting mortars were studied. Good S/S efficiency was achieved with Pb and Zn, according to the two-stage batch test. Increased leaching of Cr, Mo, and Sb was observed, especially after alkali activation. Based on sequential leaching, the addition of Portland cement and simultaneous alkali activation reduced the amount of easily bioavailable Zn and Cu, whereas alkali activation alone increased their amounts. The results indicate that of the studied S/S processes, the performance of alkali activation was the worst. The chemical retention of Cu and Zn as a carbonate or sulfate during cement hardening is a probable immobilization mechanism.

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1. Introduction

Key aspects of the current growth strategy and waste legislation of the European Union (EU) are to increase the use of biofuels for energy production and to reduce the amount of waste [1]. Recovered fuel (REF), or solid recovered fuel, is fuel that is produced from municipal or industrial waste [2]. Therefore, its use is encouraged by EU policies. As REF is made from waste materials, in some cases, it can contain high levels of heavy metals, such as Cu, Zn, or Pb. As heavy metals are enriched in ash during combustion, the resulting fly ash (FA) can be hazardous waste.

One potential option to handle such waste is stabilization/solidification treatment (S/S) using cementitious materials. Portland cement has long been used for S/S of various waste materials (sludge, soil, and industrial residues), with good results [3]. In recent years, promising S/S results for coal FA have been obtained using alkali activation (i.e., geopolymerization) [4–6]. In alkali activation, solid aluminosilicate precursors, such as coal FA or blast

furnace slag, are dissolved in an alkali solution, usually sodium hydroxide or sodium silicate, to produce a three-dimensional, aluminosilicate network structure [7]. Another option in S/S treatment that has not been studied previously is to use both alkali activation and Portland cement addition.

The most commonly used technique for the combustion of coal is pulverized coal combustion (PCC) [8]. PCC takes place at a high combustion temperature (1200–1300 °C), which, together with a fast cooling process, causes the FA to have quite high chemical reactivity (i.e., pozzolanic activity) [8,9]. REF and biofuel are typically combusted at much lower temperatures (800–1000 °C) using the fluidized bed combustion (FBC) technique. As a result, ash from FBC combustion has less pozzolanic activity than PCC ash [10,11]. Therefore, REF and biofuel ashes are less reactive than PCC ashes to alkali activation and cement addition, which could lead to reduced S/S efficiency. In addition, biofuel FA contains much more alkali and earth-alkali (Ca, K, and Na) compounds than PCC ashes, which can complicate the reaction chemistry of the S/S process.

In stabilization, the contaminants of a waste material are converted into less soluble, mobile, or toxic forms through chemical processes, but stabilization does not necessarily change the physical characteristics of the waste [12]. In solidification, the waste material is encapsulated in a solid monolithic block. The contaminants do not necessarily interact chemically with the solidifying reagent. Instead, they are embedded in the solidified matrix by physical processes. The distinction between chemical

Abbreviations: C-S-H, calcium silicate hydrate; FA, fly ash; FBC, fluidized bed combustion; FESEM, field emission scanning electron microscopy; ICP-OES, inductively coupled plasma optical emission spectrometer; L/S, liquid-to-solid ratio; N-A-S-H, sodium aluminosilicate hydrate; PCC, pulverized coal combustion; REF, recovered fuel; S/S, stabilization/solidification; XRD, powder X-ray diffraction.

* Corresponding author.

E-mail address: janne.pesonen@oulu.fi (J. Pesonen).

and physical immobilization is frequently not clear, and both mechanisms operate simultaneously. Briefly, chemical immobilization occurs at an atomic scale, whereas physical immobilization takes place at a micron scale [13].

In this paper, the S/S of FA from co-combustion of REF and biofuel (mainly bark) using alkali activation and Portland cement addition was investigated. The aim of this study was to compare the efficiency of different S/S processes using different leaching procedures. This type of comparison has not been made before and simultaneous use of Portland cement and alkali activator has not been studied at all. Also, there is only one study in the literature on the S/S of alkali-activated REF-biofuel FA [14]. The leaching tests used were: the two-stage batch test [15], static diffusion test for monolithic materials [16], and 4-step sequential leaching test [17,18]. The mineralogical compositions, microstructures, and compressive strengths of the resulting mortars were also studied.

2. Materials and methods

The REF-biofuel FA used in the present study was from a Finnish heat and electricity power plant, which uses a bubbling fluidized bed boiler to combust REF. The REF was packing material waste, such as plastic (not PVC), carton, paper, and wood collected from industrial and retail outlets. The REF was of quality class 1 [2]. The power plant fuel mixture consisted of 50% of REF and 50% biofuel (mainly wood bark). The samples were collected from electrostatic precipitators into 10 L containers.

The sizes of the FA and Portland cement particles were measured with a Beckman Coulter LS 13320, and their volumetric-based median size (d_{50}) was recorded. The main chemical components of the FA and Portland cement were determined by X-ray fluorescence (Omnian Analytix Axios^{max}) from a melt-fused tablet. The chemical composition and particle sizes of the ash and white Portland cement samples are presented in Table 1.

The experimental design is presented in Table 2. A mixture of sodium silicate (Zeopol[®] 25, Huber) and NaOH solution was used as the alkali activator. The SiO₂/Na₂O molar ratio of the sodium silicate solution was 2.5, and the water content was approximately 66 wt%. NaOH solution (5 M) was prepared from NaOH pellets (p.a. ≥99% Merck KGaA) and deionized water. The cement used in the mortars was white Portland cement (CEM I; Finnsementti).

The mortars were prepared by first mixing the dry matter thoroughly. This dry mixture was then slowly added to the liquid alkali-activator solution while stirring. To ensure good workability of the mortars, some deionized water was added to the mixture. The stirring was continued for 3–5 min. Directly after the mixing, the mortars were packed into cubic molds (35 × 35 × 35 mm), and these were then placed in air-tight plastic bags for 24 h ($T = 22\text{ °C}$). The samples were then removed from the molds and stored in the plastic bags until they were tested (28 days later).

The pseudo-total concentrations of the elements in the REF-biofuel FA, Portland cement, and sample mortars were characterized by microwave-assisted wet digestion using a 3:1 ratio of HNO₃ and a HCl acid mixture per 0.5 g of sample. They were analyzed with a Thermo Electron IRIS Intrepid II XDL Duo inductively coupled plasma optical emission spectrometer (ICP-OES). Duplicate measurements were made for each sample.

Leaching of heavy metals was studied using three different methods: (1) a two-stage batch test [15]; (2) static diffusion test for monolithic materials [16]; and (3) a 4-step sequential leaching procedure [18,19].

The SFS-EN 12457–3 is a two-step leaching method designed for waste materials [15]. It is used as a standard quality control test in Finland for fly and bottom ash used in earthworks. In the first step, the sample was crushed and sieved to a particle size of <4 mm and mixed for 6 h with deionized water (liquid-to-solid ratio; L/S = 2) in a mechanical end-over-end tumbler. In the second step, the residue from step one was mixed for 18 h with deionized water (L/S = 8). The eluates from both steps were collected and analyzed using the ICP-OES method. The cumulative release (L/S = 10) was then calculated. Deviating from the standard, a sample size of 17.5 g was used instead of 175 g, and the filter-paper retention size was 1 μm instead of 0.45 μm. Duplicate samples of each batch were analyzed, and the averages were calculated.

The purpose of the static diffusion test is to simulate the leaching of inorganic components from monolithic materials under aerobic conditions as a function of time [16]. Using this test, it is possible to determine whether the leaching is controlled by diffusion, dissolution, depletion, or surface wash-off. In the test, solid cubic mortar samples were subjected to leaching in closed tanks using deionized water as the leachant. The water was replaced eight times during the test (0.25, 1, 2.25, 6, 9, 16, 35, and 65 days). The eluates were filtered, and the filtrates were collected and analyzed using the ICP-OES method. Cumulative leaching was then calculated. Deviating slightly from the standard due to measurement technical reasons, the filter-paper retention size was 1 μm instead of 0.45 μm, and the smallest dimension of the test piece was 35 mm instead of 40 mm.

The 4-step sequential leaching procedure (Table 3) used was based on the BCR method [18]. However, extraction with deionized water acidified to pH 4 with HNO₃ [19] was added as a first stage to simulate the effect of acid rain. This modified sequential leaching scheme has previously been used to fractionate elements in FA and bottom ash [17,19]. The sample mortars were first crushed and sieved to a particle size of <0.5 mm. The eluates from each step were filtered (1 μm filter-paper retention) and analyzed using the ICP-OES method. Duplicate samples from each batch were analyzed, and averages were calculated.

The compressive strengths of the mortars were measured according to the European cement standard EN 196–1 [20] using a Zwick Z100 Roell test machine, with TestXpert II software. The compressive strengths were measured after 28 days of curing, and the average of three individual measurements was taken in each case. Standard deviations were also calculated.

The main crystalline phases of the powdered samples were identified with a Siemens 5000 X-ray diffractometer using CuKα radiation (40 mA and 40 kV) and a graphite monochromator. The step interval, integration time, and angle interval used were 0.04°/step, 2.5 s/step, and 10–60°, respectively. Powder X-ray diffraction (XRD) analyses of the sample mortars were performed after 28 days of curing. The ICDD database was used to identify the crystalline phases [21].

A field emission scanning electron microscope (Zeiss Sigma Ultra Plus) was used to analyze the cross-sections of the mortars.

Table 1
Chemical composition and particle sizes of the ash and white Portland cement samples.

	d_{50} (μm)	CaO (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	Na ₂ O (%)	K ₂ O (%)	MgO (%)	P ₂ O ₅ (%)	TiO ₂ (%)	SO ₃ (%)	Cl ⁻ (%)
REF-biofuel FA	29	27.7	32.8	12.1	5.9	2.7	1.7	2.6	1.5	1.8	5.5	1.2
Portland cement	9.8	68.1	23.7	2	0.4	0.2	0.1	0.6	0.2	0	2.1	0

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