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Characterizing Sewage Sludge Ashes in Dry and Wet States for use as SCM

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Abstract. The increasing production of sewage sludge, a residue from wastewater treatment, has led to the need for disposal methods that reduce environmental impact. The calcination of this mud residue produces the sewage sludge ashes (SSA), a powdery by-product with valuable characteristics that can serve as a secondary material source. This study explores the feasibility of incorporating SSA, as a partial substitution for Ordinary Portland Clinker, in cement-based materials. The chemical, mineralogical and physical properties of SSA were characterized to understand its impact on the cement matrix. Following that, cement paste samples and mortars were prepared with substitution rates of 0, 10, 20, 30%, and 25% respectively. The results of the work show that SSA has a possible porosity, resulted in an increase in the water demand after SSA incorporation. In addition, SSA was found to contain heavy metals, phosphorus, sulfate and lime which affects the cement hydration reaction. The modified SSA-cement pastes showed a delay in the early hydration period. A long-term positive effect on mechanical properties of mortars was noticed, with a Strength Index Activity of 86% at 28 days. The results obtained in this study encourages the use of SSA in cement-based materials in cement-composites.

Keywords: Sewage sludge ash, Portland cement, Sustainability, Hydration, Strength Activity Index

1 Introduction

Waste water coming from domestic, commercial and industrial disposals, is treated in sewage treatment plants before being reintroduced into the environment. The steps required to treat water prior to discharge into waterways produce a residue called sewage sludge (SS). Over the past few years, there has been a steady increase in the amount of SS generated internationally due to the improved rules and regulation on waste water collection and treatment. This increase in SS generation has resulted in a need for effective management and sustainable utilization of SS, in order to minimize the environ-

mental impact of its disposal. Many developed countries are adopting alternative approaches, besides landfilling, to treat wastewater sludge such as the sludge incineration [1]. This process can be done in a fluidized bed combustor, generating a nonhazardous mineral residue: sewage sludge ash (SSA) [2]. FMI process, a French company, is designing and manufacturing innovative and efficient thermal recovery units for sewage sludge treatment. SSA are produced from the incineration of the sludge in a fluidized bed combustor at a temperature of 850°C. The ashes collected after incineration are powdery by-products with properties that make them valuable as a new source of secondary material [3], and potentially useful in manufactured concrete products [2].

The construction industry is one of the major consumers of natural resources and nonrenewable energy. Construction activity has seen remarkable growth over the last few years. The cement production has taken place to be the third largest consumer of industrial energy, producing 6-7% of the global carbon dioxide (CO₂) emissions [4]. As stated in [4] the annual cement production is expected to reach 4.68 Gt/year by 2050. This growth increases CO₂ emissions from cement production. The majority of cement binders used are Portland cement, and a significant portion of the CO₂ emissions from its production is a result of the decarbonation of limestone during the clinkering process. Reducing these emissions is the most pressing concern for the cement industry. The use of supplementary cementitious materials (SCM) to partially replace clinker is a promising solution, as it can significantly decrease CO₂ emissions without negatively affecting the strength, the durability, or the final product cost. This method can reduce emission by 30 to 40% [5]. In addition to the need for reducing CO₂ emissions in cement production, there is also a growing demand for the use of sustainable materials in construction, which highlights the importance of exploring the potential use of waste materials such as SSA as SCMs.

Given these facts, the use of SSA as a SCM in cement-based products has gained an increasing attention in recent years in order to valorize these waste materials and reduce their environmental impact [6]. The valorization of ash is thus fully in line with a circular economy approach on two levels. Firstly, it will complement the sludge incineration approach with a new real-life application of the final generated residue. Secondly, the re-use of the produced ashes joins cement factories efforts in their long-term ecological transformation strategy, in order to reduce CO₂ footprints per ton of cement produced. According to previous studies, the chemical characterization of SSA implies the presence of major oxides: SiO₂, Fe₂O₃, CaO and CaCO₃. The physical characterization of SSA shows an irregular morphology, as well as a high porosity between its particles, leading to an excess of water demand after incorporation in the cementitious composites. [7] mentions that a substitution of cement by more than 10% of SSA affects the workability of the composite, in addition to the mechanical properties of the cementitious composite. [8] studies the hydration mechanism of a cement paste after SSA incorporation. The presence of heavy metals in this material tends to increase the setting time of the cement paste, and causes a delay of the main hydration peak by isothermal calorimetry analysis. This study introduces several approaches of characterization techniques to examine SSA, both in its dry state and following contact with water. Furthermore, the study explores SSA behavior upon integration in the cement matrix.

2 Materials and methods

The first step of this study involved the determination of chemical and physical properties of the studied SSA powder, which originated from Rhône-Alpes region in France. The cement used in this study is a standard CEM I 52.5 R CE CP2 NF, with a minimum clinker content of 95%, provided by the company VICAT. Table 1 presents the oxide composition and physical properties of the used CEM I.

2.1 Characterization of SSA

The density and specific surface area were determined using helium pycnometer (AccuPyc 1330, Micromeritics) and ASAP 2020 (Micromeritics), respectively. Particle size distribution was analyzed by laser diffraction using Mastersizer 3000 (Malvern Panalytical), under a pressure of 4 bars and a vibration rate of 20%. The oxide composition was analyzed using X-Ray Fluorescence (XRF) spectrometry. The mineralogical content was identified by X-Ray diffractometry (XRD) using Bruker D8-A25 diffractometer. The quantification of the mineralogical phases detected was carried out with the internal standard method to quantify the amorphous phase content in the powder. Prior to analysis, SSA powder samples were mixed with 10 wt% of the internal standard (ZnO) until well homogenized. The inorganic element content in SSA was quantified using inductively coupled plasma-mass spectrometry (ICP-MS, Agilent). Electrical conductivity was monitored in a dilute suspension with water-solid (w/s) weight ratio of 10 over a duration of 5 days, until a steady electrical conductivity was reached. This was done by mixing SSA powder with demineralized water in a 25°C thermostated reactor with a rubber stopper to prevent solution carbonation. A magnetic stirrer was used to control the stirring rate during the experiment. The duration of 5 days was chosen based on previous tests that have shown that SSA dissolution shows equilibrium within this period. The sample was tested three times in order to evaluate the repeatability.

To track and monitor the behavior of SSA after contact with water, samples were collected at different time intervals t (0, 0.1, 0.3, 1, and 72h), chosen based on the variation of the conductivity curve in function of time. These collected samples were then filtered by vacuum. The filtrate solutions at all times t were analyzed chemically by ICP (test inspired from NF EN 12457-2). The solid ashes from the filtration (SSAF) at only $t=1h$ (after the main peak of dissolution according to Fig. 3) were collected and dried in the oven at 105°C for 24h. The SSAF was mixed with water at w/s ratio of 0.9 (elevated ratio due to the high capacity of water absorption) and analyzed by calorimetry isotherm at 20°C, to track the heat release. The results obtained were compared to a reference SSA paste composed of water and SSA at the same w/s ratio.

2.2 Characterization of the cementitious paste

In this study, different cement pastes were prepared and tested. The substitution rates of CEMI by the SSA in the pastes were 10, 20 and 30% by weight of CEMI. The hydration

of cement pastes, with and without partial substitution with SSA, was studied by isothermal calorimetry, at a constant temperature of 20°C (TAM air, TA instrument). All samples were prepared with a w/s ratio of 0.5 and a mixing duration of 60 s. After mixing, a mass of 9 g of prepared paste was introduced into a cell and placed inside the calorimetry channel. The evolution of heat flow was recorded for 7 days. The data were integrated starting 30 mins to avoid any disturbance after the cell insertion into the channel. The cumulative heat was then calculated at 7 days.

Mechanical characterization of the normalized mortars was done by calculating the strength activity index (SAI) of mortar specimens, prepared with 25 wt% replacement of CEMI by SSA. The mortars were prepared in a 4x4x16 molds, according to French Standard NF EN 450-1. The composition of the reference mortar prepared is as follows: $m_{\text{cement}}= 450\text{g}$, $m_{\text{water}}= 225\text{g}$ and $m_{\text{sand}}= 1350\text{g}$ (NF EN 196-1). After demolding, compressive strength of the prepared prisms were measured at 1, 2, 7, and 28 days. SAI calculation was determined using Eq. 1.

$$\text{SAI (\%)} = \frac{f'_{ci}}{f'_{c(\text{reference})}} \times 100 \quad (1)$$

where f'_{ci} : Compressive strength of the tested mortar with SSA at time t
 $f'_{c(\text{reference})}$: Compressive strength of the reference mortar without SSA at time t

3 Results and discussions

3.1 SSA Characterization

SSA characterization is essential in order to confirm the potential of SSA incorporation in cementitious paste. The chemical composition and physical characteristics of SSA powder are presented in Table 1.

Table 1. Oxide composition, physical properties of SSA and CEMI

Oxides (wt%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	P ₂ O ₅	TiO ₂	MnO	Cl
CEMI	19.2	4.9	3.7	62.4	2.1	3.8	1.0	0.2	0.1	0.3	0.1	0.05
SSA	20.0	6.4	15.4	30.8	1.5	5.3	1.3	0.4	10.3	0.6	0.1	0.1

Physical Properties	LOI	Density (g/cm ³)	Specific surface area (m ² /g)	D50 (μm)
CEMI	1.6	3.2	1.9	10.5
SSA	7.6	3.0	7.8	23.5

The specific surface area of SSA is higher than that of cement, with 7.8 m²/g and 1.9 m²/g respectively. This result suggests that the SSA studied could have either a porosity higher than that of CEMI or a large number of fine particles. [9] links the higher specific surface area to an irregularity in the morphology of SSA particles, which suggests the

existence of a strong internal porosity, leading to a possible need for an additional water volume for the prepared cementitious paste with ashes. However, an additional amount of water could lead to a decrease in the mechanical properties of the paste. In this case, the use of water-reducing admixture is a possible solution [10]. The particles size distribution of the ashes, presented in Fig. 1, ranges between 0.1 and 500 μm , whereas that of CEMI ranges between 0.1 and 80 μm . The mean diameter for SSA and CEMI is 23.5 μm and 10.5 μm respectively. This suggests that SSA carries larger particles than cement, indicating that the high specific surface of SSA is potentially due to an irregularity in its morphology, which leads to higher porosity between its particles.

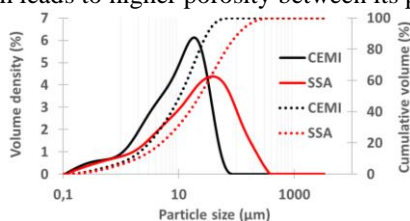


Fig. 1. Granulometric distribution curve for CEMI and SSA

The chemical oxide composition of SSA are presented in Table 1. Similar to few previous studies [10], the used SSA is majorly composed of calcium, iron, silica, aluminum, and phosphorus oxide.

Table 2. Mineralogical phases content (in %), determined by DRX

Phases (%)	Amorphours	Quartz	Hematite	Whitlockite	Anhydrite	Calcite	Portlandite	Lime
SSA	21.0	11.7	10.6	22.6	8.9	16	2.4	6.8
SSAF	26.9	8.9	10.0	23.6	4.7	17.8	7.8	0.3

Table 2 gives a summary of SSA mineralogical composition. The quantification results obtained by XRD proves the presence of 21% of an amorphous phase of unknown composition, in addition to different minerals including Quartz (SiO_2), Hematite (Fe_2O_3), whitlockite ($\text{H}_{0.8}\text{Ca}_{9.1}\text{Mg}_{0.6}\text{Fe}_{0.4}(\text{PO}_4)_7$), Anhydrite (CaSO_4), Calcite (CaCO_3), Portlandite ($\text{Ca}(\text{OH})_2$) and Lime (CaO). We notice the presence of soluble phases (anhydrite, portlandite, and lime) having a possible impact on the hydration reaction after SSA incorporation in the cement paste. The 11.7% quartz content, inert mineral, could be originating from the fluidized bed during the SS incineration process. Whitlockite presents the highest phase percentage of 22.6, containing Ca, Mg, Fe and P elements. According to [11], whitlockite possesses a weak solubility in a basic medium. The presence of the phosphorus in a soluble form could cause a delay in the setting time of cement composites [10]. In fact, the presence of phosphate ion limits the rate of CSH nucleation and growth, disturbing the progress of the hydration reaction [12]. The presence of 8.9% anhydrite could cause a delay in sulfates depletion [2]. An excess of sulfate

available in the cement matrix may delay the hydration reaction, more precisely, the transformation of ettringite into monosulfate. The sulfate should be highly soluble in order to avoid a delayed formation of ettringite, which causes undesirable swelling of the concrete composite at early and late age [13]. The high Fe_2O_3 content in SSA gives a red coloring to the powder. [13] observed the same SSA coloring aspect, mentioning that the use of Fe based salts to precipitate the phosphorus during the waste water treatment could be associated with the elevated Fe_2O_3 content.

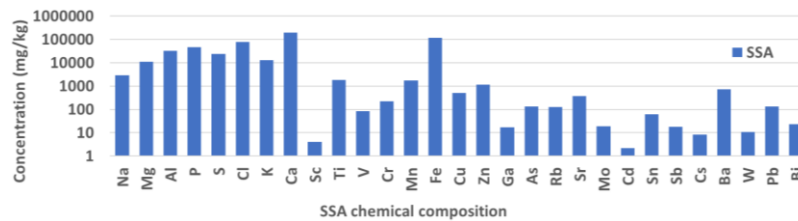


Fig. 2. Chemical components of sewage sludge ashes (SSA) in mg/kg

SSA chemical composition determined by ICP represented in fig. 2 is in accordance with the oxide composition by XRF (except SiO_2 , which is hard to dissolve by acid attack method used for the ICP sample preparation). In addition, the presence of heavy metals such as Cr, Cu, Zn, As, Se, Cd, Mo, Sb, Ba, Pb is detected, originally present in the treated waste water. SSA also contain P and S elements. Several authors have reported the effect of orthophosphate on the cement hydration [14]. In the same way that sulfates limits C_3A reaction, the adsorption of phosphate ions slows down C_3S dissolution rate [15]. Fig. 3 shows the average electrical conductivity curve. After contact with water, a sudden increase in the electrical conductivity was observed. This increase is linked to an increase of ions concentration in the solution, in other words, a dissolution aspect of the ashes in water. However, a decrease is reflected in a precipitation phenomenon. This conductivity reaches a maximal value of 12.1 mS/cm after few minutes of wetting, and slightly decreases with time until reaching a value of 8 mS/cm at $t=5$ days, reflecting an equilibrium state in the solution.

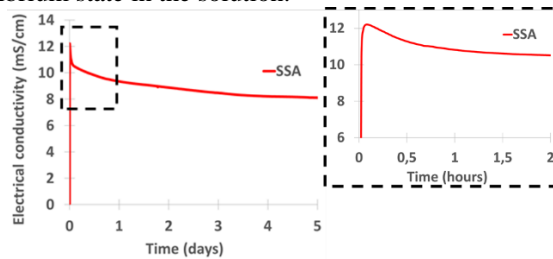


Fig. 3. Average electrical conductivity curve of SSA in water

The ICP analysis of the collected solution samples are represented in Fig. 4, along with those of SSA. A few minutes after the SSA immersion in water, at $t=0.1\text{h}$ (Fig. 4a), an increase in the concentration of all elements was observed, especially for Na, S, K and Ca (Fig. 4a), which are highly soluble in water, and contributed to the peak observed

in Fig.3 after SSA immersion. Additionally, minor inorganic elements were also detected in the filtered solution such as Cr, Cu, Zn, Sr, Ba and Pb (Fig. 4b). The presence of hydroxide ions (OH^-) in high concentration confirms the basicity of the solution, after SSA immersion in the originally neutral solution. This concentration remains constant after 72h confirming the basic media. The results of this test (at 72h) confirms the possible presence of this element dissolved in the cement matrix, after SSA incorporation, and a probability to affect the cement hydration reaction.

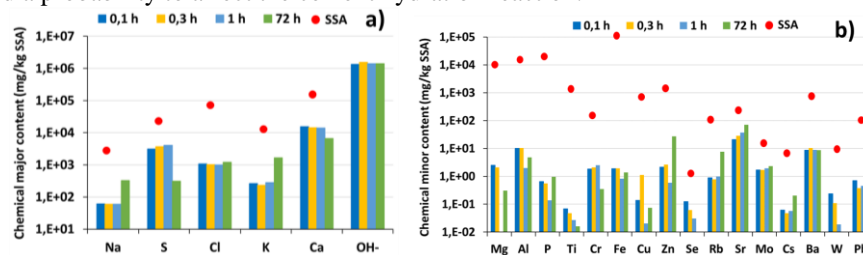


Fig. 4. Chemical analysis by ICP of the filtered solution compared to SSA: a) major elements content, b) minor elements content

3.2 Effect of SSA incorporation on cement hydration

To better understand the effects of SSA incorporation on cement hydration, we analyzed the heat evolution curve of the control cement paste and three different substitution rates (10%, 20% and 30% by weight of cement), illustrated in Fig. 5. Fig. 6 illustrates the investigation of heat release in water for only SSA and SSAF, aimed at improving our understanding of how SSA behaves when in solution. All results were normalized with respect to the binder. Until the end of the induction period (Fig.5), an exothermic reaction takes place between, defined by a large exothermic signal, linked to the dissolution of anhydrous phases and a partial precipitation of cement hydrates. The SSA-modified pastes present an exothermic reaction during the induction period. This reaction is more pronounced with higher substitution rates, ranging from 1h to 3h for 30% SSA. In Fig. 6, upon contact with water, an intense heat release is observed and was defined by an increased signal, up to 58 mW/g binder, due to the reactive lime (7%) and anhydrite (9%) content in SSA. This result confirms the exothermic phenomenon brought by the presence of SSA in the cementitious material (Fig. 5a). As for SSAF-water paste heat evolution, we observe a major decrease in signal, down to 28.8 mW/g binder, compared to SSA, after contact with water. The immersion of SSA in demineralized water for one hour activated the dissolution of the soluble phases, which was confirmed by XRD analysis (Table 2). The soluble phases, such as anhydrite and lime content, dissolved quickly. The lime is hydrated after contact with water, hence the increase in portlandite content (7.8%).

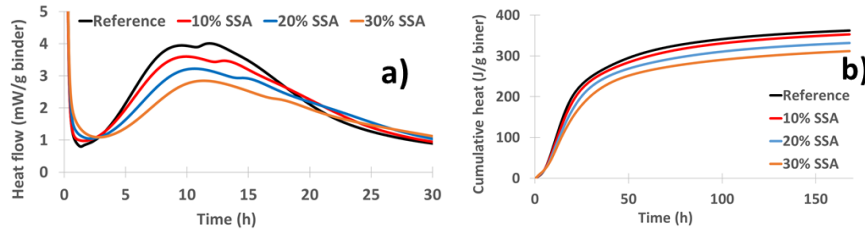


Fig. 5. a) Normalized heat flow and b) cumulative heat of 3 different rates of SSA incorporation 10%, 20% and 30% by calorimetry isotherm

As a result, the signal drop with SSAF ($t=0.2$ h) occurred at a faster rate compared to SSA ($t=0.4$ h) and stabilized along the testing period for both samples. The main peak in the heat flow representation (Fig. 5a) corresponds to the hydration of the main component of CEMI (C_3S) indicating the nucleation and growth of CSH and portlandite. The hydration peak of the control sample (4 mW/g binder) is consistently decreasing as the SSA rate increases (2.8 mW/g binder for 30% SSA). This decrease is due to the replacement of a quantity of CEMI by SSA, resulting in a reduced CEMI content in the paste. As a result, a dilution effect occurs, which leads to the precipitation of less hydrates for a same volume of water [16].

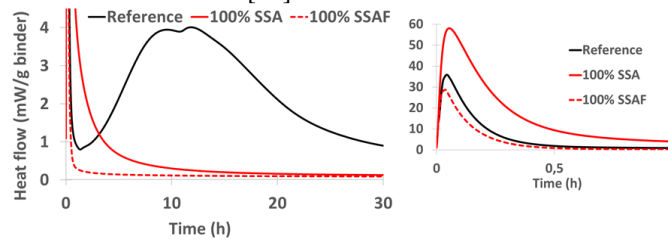


Fig. 6. Heat flow of the two SSA and SSAF paste, compared to the cement reference paste at 20°C

The second peak in the hydration period (Fig. 5a) corresponds to the depletion of sulfate, leading to the secondary formation of ettringite [17]. This peak is observed at 12h for control paste, against 18h for 30% SSA, resulting in a 6h delay. For a higher rate of substitution, there is a higher sulfate content in the cement matrix, causing a delay of sulfate depletion, and an additional ettringite precipitation. The increasing duration of the acceleration period observed with all pastes containing SSA could be due to the chemical composition of the SSA, which contains heavy metals, phosphorus, zinc, that disturb the hydration reaction of cement and delays its setting time [10]. [15] explain that the presence of Zn in the cement matrix can delay the formation of portlandite and therefore delay the paste setting time. The cumulative heat of SSA-modified pastes, presented in Fig. 5b, show a decrease after 7 days with the increase of SSA substitution rate (312 J/g binder for 30% SSA) compared to the control paste (362 J/g binder).

3.3 Strength activity index of mortar with SSA incorporation

Fig. 7 shows the results of the strength activity index results at 1, 2, 7 and 28 days for the reference mortar (without substitution) and mortars containing 25 wt% of SSA. SAI

is defined as the compressive strength ratio of the tested mortar containing SSA to the control mortar at the same age.

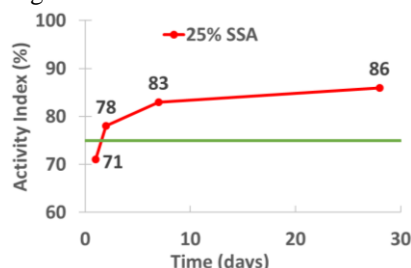


Fig. 7. Strength activity index of prisms prepared with 25 wt% CEMI substitution, and the 75% limitation according to the EN standards

The results show that SAI is less than 100%, indicating a decrease in the mechanical properties of the studied SSA-mortar. The calculated SAI is equal to 71% at 1 day, increasing with time to reach 86% at 28 days. According to results obtained by [13], a decrease in the mechanical properties after SSA incorporation could be possibly associated to a slow and continuous reaction of SSA in cementitious matrix. As mentioned earlier, SSA could hold a porosity within its particles, resulting in porosity in the prepared mortar after SSA addition, which reduces mechanical properties. Also, the cumulative heat (see fig. 5b) is coherent with the decrease in mechanical properties, due to the presence of less hydrates in the paste. Despite the reduction in mechanical properties with respect to the reference, SAI calculated is higher than 75%, that is the minimum value according to European standards. Therefore, SSA could possibly: (i) be a reactive material, given a SAI higher than the limit, or (ii) have a filler effect [18].

4 Conclusion and perspectives

In summary, sewage sludge ash is a by-product of the sludge incineration process and has potential as a sustainable SCM in the cement industry. By integrating SSA, it is possible to reduce waste generation, carbon emissions, and resource consumption, while also creating new opportunities for economic and social development. However, the use of SSA in cement production has some challenges and limitations, such as its effect on workability and mechanical properties. This study aims to explore the properties of SSA using various characterization techniques and investigates its behavior upon integration in the cement matrix.

The following results were found:

1. The physical characterization of SSA incinerated at 850°C showed porosity, resulting in increased water demand;
2. The oxide composition of SSA suggests a low pozzolanic activity;
3. The chemical characterization revealed the presence of heavy metals, highlighting the need for careful selection and processing of the material to minimize its environmental impact;

4. The presence of soluble phases in SSA are observed, which may affect the hydration at early and late age;
5. The mechanical properties for a CEM I substitution rate of 25%, decreased of only 14% at 28 days, with respect to the reference.

The outcome of this work is a comprehensive evaluation of the properties of SSA, providing valuable insights into the potential integration of SSA as an environmentally sustainable and circular material in the cement industry. Despite discrepancies in the properties of SSA and the slight decrease in the mechanical properties of the SSA modified cement matrix, there still exist an interesting potential of SSA incorporation in cement composites. Further investigations are required to gain a better understanding and evaluation of the behavior of the SSA in the cement matrix, including: (i) the pozzolanicity, (ii) the properties variation of produced SSA in different seasons of the year, (iii) the microstructure and hydrates formation during the hydration reaction, (iv) the impact of leached heavy metals on the environment. In addition, durability tests need to be performed on concrete samples, as it remains a gap in the literature.

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