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Comparative Characteristics of Hydrated Lime with Fine Sewage Sludge Ash (FSSA) and Coal Fly Ash (CFA)

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Abstract

The disposal of waste has become an environmental issue due to the limited available landfilling space. This paper aims to compare the characteristics of hydrated lime with fine sewage sludge ash (FSSA) and coal fly ash (CFA). Multiple techniques, X-ray fluorescence (XRF), X-ray diffraction (XRD), the Fourier transform infrared (FTIR), compressive strengths, thermophysical properties, and setting time were used to assess the physicochemical characteristics of the lime-based materials. X-ray fluorescence and X-ray diffraction were used to determine the chemical composition and phases of ashes, lime and binders. The results showed that the chemical composition of ashes is similar to that of cement. Besides glass, the main minerals identified in CFA and FSSA are quartz (SiO_2) and anhydrite ($CaSO_4$). Moreover, calcium aluminium oxide ($Ca_3Al_2O_6$) was detected for CFA and phosphorus calcium silicate (Ca_2SiO_4-$Ca_3(PO_4)_2$) for FSSA and minor phases were detected for both. FTIR measurements were carried out to characterize the inorganics components of different samples. Compressive strengths of mortars with different formulations have shown that both have a long-term positive effect which might be related to a pozzolanic activity. For the CFA the L_3 binder consisting of 60% of coal fly ash and 40% lime has a higher compressive strength than the others while for the FSSA the L_4 binder consisting of 80% fine ash and 20% lime has a higher compressive strength than the others. Both binders setting start times are greater than that of cement but shorter than that of lime. The study of the thermophysical properties of binders shows that they have a higher thermal resistance than cement mortar. Moreover, binders heat up less quickly because of their low effusivity compared to cement. Lime-based materials system could be a promising option to both relieve the waste disposal pressure and provide a potential sustainable construction material.

Keywords

Coal Fly Ash, Fine Sewage Sludge Ash, Lime, Binder, Microstructure, Thermomechanical Behavior

1. Introduction

Since ancient times lime-based materials have been widely studied and applied in construction. Specifically, the lime-volcanic ash mortars were first applied as a wall covering on Santorini Island in 1500 BC [1] [2].

Lime together with volcanic ash and crushed sintered clay products in concrete were used by the Romans for the construction of buildings and coastal works, which had resisted erosion from seawater for over 20 centuries [3]. However, the invention of Portland cement (PC) in the 19th century caused a recession in the use of lime-binders. Contribution to climate change by greenhouse gases (GHG) emissions in the atmosphere (CO_2 in particular) which are around 7% from cement manufacturing is partly responsible for global warming [4] [5] [6] [7] [8]. Environmentally friendly cement-based materials are a topic of interest and cement replacement materials play an important role in the construction industry considering economical, technological and ecological points of view [9] [10] [11].

Then lime-based materials have again become popular as alternatives to PC recently due to the high cost, carbon footprint and energy intensity of PC. In addition, concrete based on PC would normally suffer from long-term degradation problems, especially under harsh conditions [3]. More importantly, the application of PC-based materials in repairing ancient buildings is not allowed according to the requirements regulated by European supervisory authorities which specify that the repairing materials should be compatible with the original materials [1].

Wastes are commonly used as partial replacements for PC, as they are not only cost-effective, but also improve some properties of the produced mortars and concrete. Their role in the lime-based materials is their participation in pozzolanic reactions with calcium hydroxide to form hydration products, which are similar to those produced from PC hydration including calcium silicate hydrates (CSH) and calcium aluminate silicate hydrates (CASH) [12].

Coal fly ash is considered as an industrial by-product derived from coal combustion in thermal power plants while the FSSA is derived from the sludge dewatered, dried and burnt in a fluidized bed incinerator. Their improper disposal has become an environmental concern and resulted in a waste of recoverable resources.

Then the main application fields of these ashes are currently attributed to construction, ceramic, environmental, landfills and agricultural sectors. According to statistics, industrialized countries such as the US and EU mostly use the fly ash in concrete and cement production, waste stabilization, mining applications, as structural fills and embankments, for remediation and restoration that overall accounts for more than 60% - 70% of all Coal Fly Ash [1] [13]. Sludge ash is also very interesting for many applications because it can be mixed with cement or binders such as lime to give rise to new materials with several attractive physical characteristics such as the increase in resilience, and the long-term strength of mortars [14] [15] [16]. In fact, new environmental challenges and research opportunities arise on the use of these solid wastes as well as their basic properties.

So the use of such waste materials allows decrease energy consumption, preserves non-renewable natural resources, and reduces the high amount of material that goes to landfills. Mineral additions are defined as inorganic materials, pozzolanic materials or latent hydraulic materials that finely divided can be added to concrete and/or to Portland cement-based mortars, to improve some of their properties or confer special characteristics [17]. The large amounts of solid waste produced can be recovered and used as building materials [14]-[21].

To better understand the characteristics between hydrated lime and FSSA or CFA we also used a comparison between these samples.

Then this paper focuses on a comparative study of using coal fly ash (CFA) and fine sewage sludge ash (FSSA) as an additive to hydrated lime, to develop a new type of binder. The physical, chemical and thermomechanical properties of the systems were also compared. Multiple techniques including X-ray fluorescence (XRF) and X-ray diffraction (XRD) were used to assess the chemical composition and mineralogical characterization of samples. The FTIR, the density, the standardized consistency tests, the setting time, the compression tests and the thermal properties of ashes were also investigated.

2. Materials and Methods

Two types of ash CFA and FSSA mixed with hydrated lime were used as binding materials. The FSSA was collected from the Senegalese sewage treatment plant of Camberene located in the Dakar region while for the CFA comes from the thermal power of the Chemical Industries of Senegal in the Thies region. Both are fine grey powder and CFA is the principal by-product generated during the coal combustion process while for the FSSA, sludge is dewatered, dried and burnt in a fluidized bed incinerator at 850˚C. The FSSA particles were porous with a relatively rough surface while the CFA particles were largely spherical in shape [22]. They have approximately the same absolute density of 2.4 g·cm^{-3}. The lime was commercially available from local suppliers and its absolute density is in the range 2.6 to 2.9 g·cm^{-3}.

A portable X-ray fluorescence (P-XRF) device Niton XLT900s was used to analyze the chemical composition of the ashes in terms of major and minor elements with a measurement time of 350 s. XRF was performed with 100% normalization and full fundamental parameter quantification techniques [23] [24] [25] [26]. The mineralogical characterization of ashes and mixtures lime-ash-water was carried out by X-ray powder diffraction (XRD) using a PW1840 diffractometer with Cu Kα radiation operating at 30 mA and 40 kV. The analysis was done in the continuous scanning mode with a speed of 0.05˚ per s within the range of $10^\circ \le 2\theta \le 70^\circ$ and acquisition time of 1560 s. The diffractometer is linked to a computer equipped with APD software. For data processing we used X Pert High Score software [22] [25] [26]. The Fourier transform infrared FTIR measurements were carried out by spectrometer Perkin Elmer FTIR System Spectrum X in the range 400 - 4000 cm^{-1}.

For the setting time, the Vicat apparatus (NF EN 196-3) was used. The powder was dry mixed for one minute at a low speed by a standard mechanical drum mixer before adding water, and then another three minutes of mixing (1 min at low speed, 2 mins at high speed) was carried out to obtain the homogenous pastes. After mixing, the fresh mixture was cast into prisms steel moulds with a size of 4 × 4 × 16 cm and then subjected to vibration for about one minute to remove entrapped air in the samples. Compressive strength was then investigated after hydration times: 3, 7, 28 days and each value is the average of 6 tests (NF EN 196-1). All thermophysical experiments were performed using samples prepared in a mould of dimensions of 10 cm × 10 cm × 2 cm for the thermal test. Then the thermal conductivity and effusivity of samples were determined simultaneously using a transient method [25] [27].

3. Results and Discussion

The chemical compositions of FSSA and CFA determined by using a portable X-ray fluorescence (P-XRF) device Niton XLT900s, are shown in **Table 1**. The major constituents of FSSA are SiO_2, Al_2O_3, CaO, Fe_2O_3 and P_2O_5 and the FA is mainly consisted of SiO_2, Fe_2O_3 and Al_2O_3.

We find that concentrations of SiO_2, TiO_2, and Al_2O_3 are higher in CFA. The amount of CaO and P_2O_5 is high in FSSA compared to CFA and others minerals admixtures. These minerals are combined to form a calcium phosphate mineral weakly soluble in a basic environment [19]. It is known that the occurrence of phosphorus affects the setting time [28]. As shown in **Table 1** the amount of Mg is 3.90% in FSSA while no Mg has been identified in the CFA or their concentration was below the detection level. The concentration of CaO in FSSA is instead 4.7 times higher than that in CFA while for SiO_2 the concentration is 2.2 times in CFA compared to FSSA. For the Al_2O_3 the concentration in the former is instead 1.8 times higher than that in the latter. The chemical composition of FSSA and CFA is closer to that of cement produced by industries in Senegal having the following chemical composition: SiO_2 (19.53), Al_2O_3 (7.12), Fe_2O_3 (2.22), and CaO (65.5) [29]. These oxides SiO_2, Al_2O_3 and Fe_2O_3 compose the reactive part of pozzolanic materials [30] [31]. In the CFA the sum of SiO_2, Al_2O_3 and Fe_2O_3 represent more than 70% so this fly ash is in Class F Fly ashes based on the

Table 1. Results of major (%) and minor (mg/kg) elements using XRF technique of sewage sludge ash contents.

Elements	FSSA	CFA
SiO_2	24.1%	52.1 wt%
Al_2O_3	6.69%	11.8 wt%
Fe_2O_3	12.6%	7.59 wt%
CaO	22.2%	4.68 wt%
MgO	3.90%	LOD
K_2O	3.22%	1.72 wt%
TiO_2	0.66%	1.52 wt%
MnO	0.10%	0.05 wt%
P_2O_5	5.92%	1.15 wt%
Pb	357 ppm	29 ppm
Cu	1264 ppm	47 ppm
Cr	212 ppm	157 ppm
Zn	3632 ppm	54 ppm
As	16 ppm	25 ppm

standard classification [32]. So both may be used in civil engineering. As for heavy metals they are more important in FSSA than CFA except for the As. For example, the concentration of Pb is about twelve times higher in FSSA than in CFA, while that of Zn is 67.2 times greater in the former than that of Zn in the latter.

3.1. Mineralogical Characterization

Figure 1 shows the diffractogram of lime. The phases identified in lime are portlandite and calcite. Portlandite comes from the slaking of lime. The presence of calcite could be due to the carbonation of porlandite due to its exposure to air. The carbonation of lime is due to the reaction of two compounds: 1) CO_2 from the atmosphere, and 2) calcium hydroxide ($Ca(OH)_2$). First, CO_2 dissolves in the water of the pores forming carbonate ions. Then, these carbonate ions can react with the Ca ions of the pore solution leading to calcium carbonate ($CaCO_3$) precipitation. The $CaCO_3$ coming from the carbonation of other hydration products and/or anhydrous silicates and aluminates phases [8].

Diffractograms of FSSA (**Figure 2**) and CFA (**Figure 3**) show the existence of a complex polyphasic material composed of several crystalline phases and a glass phase [1] [14] [25] [26].

Besides glass, the main minerals identified in these ashes are quartz (SiO_2), and anhydrite ($CaSO_4$) but in CFA we detected calcium aluminiumoxide ($Ca_3Al_2O_6$) while for the FSSA a phosphorus calcium silicate (Ca_2SiO_4-$Ca_3(PO_4)_2$) was found and minor phases (**Figure 2**).

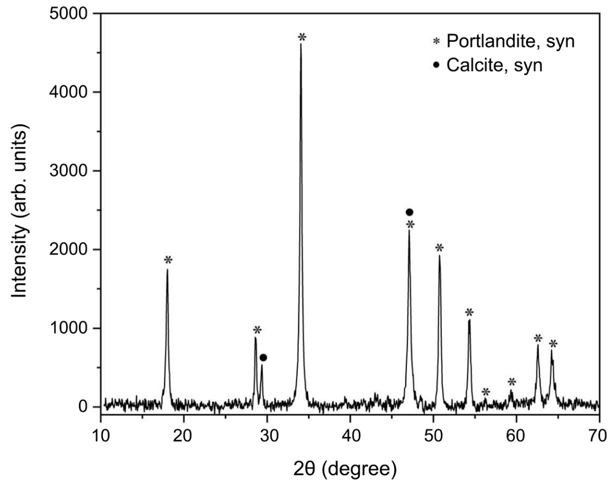

Figure 1. Diffractogram of lime.

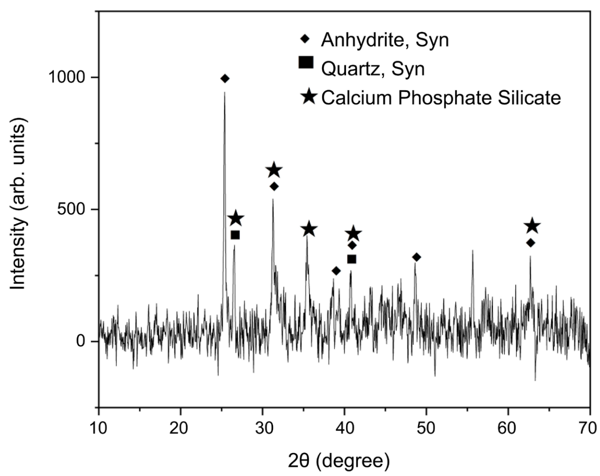

Figure 2. Diffractogram of the sewage sludge ash.

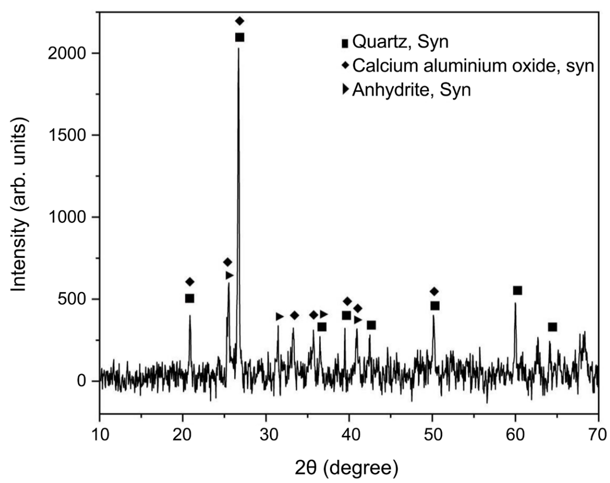

Figure 3. Diffractogram of coal fly ash.

Both have a high background due to the amorphous phase. Then elements in the amorphous phase can react with lime and produce new phases. The intensity of the peaks of FSSA is low compared to CFA because the crystallinity of the fine sewage sludge ash is weak [33] [34].

Gypsum, hydrated anhydrite is a mineral added to the clinker to form cement Portland. It is used as a retardant in Portland cement clinker. It reacts with calcium aluminates to regulate the setting by forming ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}·26H_2O$). Its presence in the ashes is beneficial because we have more need to add it to form a binder based on ash and hydrated lime. Calcium reacted with aluminum and silica to form new phases of hydrated aluminum and aluminum silica like Gehlenite and Calcium Silicate Hydrate for certain formulations (**Figure 4** and **Figure 5**). Aluminum and silica came probably from the partial decomposition of the vitreous phases of ashes. In the presence of hydrated lime, phosphate can cause precipitation of the poorly soluble calcium phosphate on the surface of cement grains. The compounds form a fine crystalline and poorly permeable layer, which limit the hydration process. Hence poorly soluble phosphate can crystallize in the pores of the paste [28].

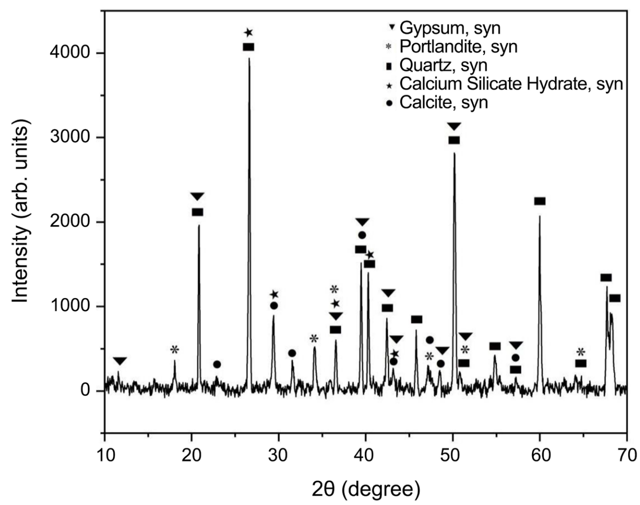

Figure 4. Diffractogram of L328JCM.

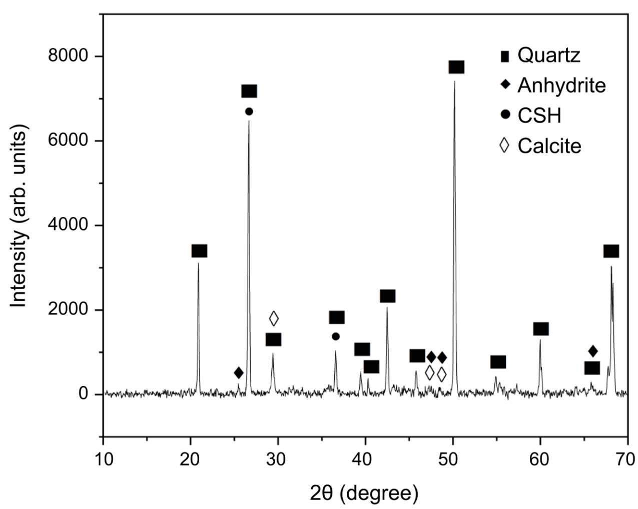

Figure 5. Diffractogram of L428JCB.

The hydration reaction allows the transformation of the binder paste from the liquid state to the solid state. The hydration mechanisms occur simultaneously and it is a complex process in which the main binder compounds react to form new insoluble compounds which cause the material to set and gradually harden. Thus these results show the reactivity of the ash in the presence of lime, which mechanically results in the hardening of the paste figure.

3.2. FTIR Results

Results of FTIR show that these materials tend to report small bands located between 600 and 650 cm^{-1} and bands between 790 and 800 cm^{-1}, which are attributed to the bonds present in the fly ash source (quartz and mullite) [35] (**Figure 6**). In both ashes, SiO_2 (quartz) was identified by the absorption bands at 773 cm^{-1} and 788 cm^{-1} while for the pure SiO_2 the absorption bands were found at 797 and 778 cm^{-1} [36]. In the reference [37] the absorption bands of silica appear at 785 cm^{-1}. Tantawy *et al.* showed that incineration of sewage sludge at 800˚C leads to a decrease in the intensity of the absorption bands of silica. So this is an indication of increasing the degree of polymerization of silica network as a result of the crystallization of silica into quartz. Bands observed around 3200 and 1601 cm^{-1} are due to vibrations of hydroxyl groups.

The absorption bands at 1027, 1103 and 1129 cm^{-1} in FSSA (**Figure 7**) show the P-O stretching region and this could identify the $Ca_3(PO_4)_2$ found in XRD results of FSSA [36].

The absorption band of P-containing compounds appears at 567 cm^{-1} due to P-O bending vibration [37] [38]. In FSSA (**Figure 7**) this absorption band appears at 557 cm^{-1}. Anhydride $CaSO_4$ could be responsible for the absorption band at 1131 cm^{-1}. A shoulder at 875 accompanied by a low intensity band at 1459 is characteristic of C-O bond stretching vibration modes. These absorption bands highlight the presence of calcium carbonate which could come from a slight carbonation of these ashes [39]. This also suggests that in the minor phases of CFA diffraction there is calcium carbonate. In [37], the absorption band of carbonate appears at 1442 cm^{-1}. These results are in good agreement with XRD results.

3.3. Thermomechanical Behaviour

These results show that mortar containing 60% of coal fly ash and 40% of lime L_3CFA has the greatest resistance at 28 days while the L_4FSSA mortar containing 80% of FSSA and 20% lime has the greatest resistance at 3 days. Furthermore, at seven days, the resistance of the two binders is in the same order (**Figure 8**).

We find that the thermal conductivity of mortars made from ashes and lime is lower than that of mortar made from cement (**Table 2**). This shows that the ashes and lime mortars have a higher thermal resistance than the cement mortar. This is very important in the sense it helps in reducing the heat exchange between the internal and external parts of the buildings.

Also, the thermal effusively value of lime and ashes mortars is lower than that of the cement mortar. Thus, lime and ashes-based mortar absorb heat less quickly than cement mortar. These mortars when used in the construction industry will develop better thermal insulation properties than the latter. Mortar from FSSA and lime has better thermal insulation properties than the Mortar from CFA and lime.

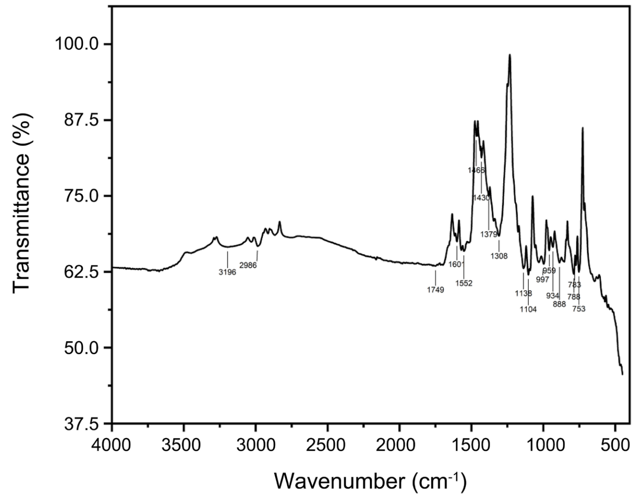

Figure 6. FTIR spectra of CFA.

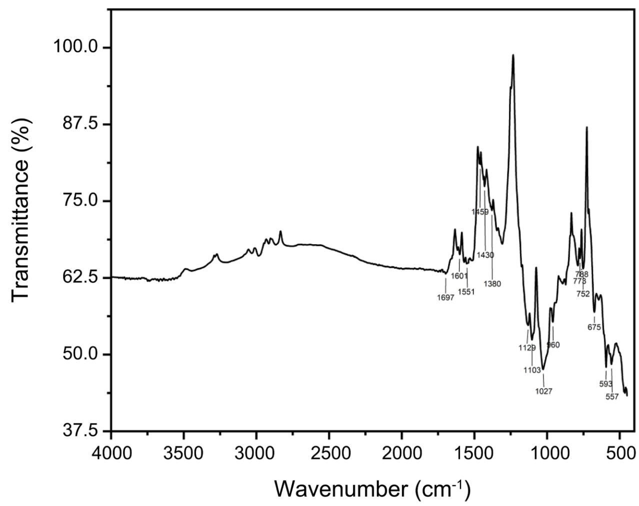

Figure 7. FTIR spectra of FSSA.

Table 2. Comparison of thermo physical properties.

Mortar	Average thermal conductivity (W/m·˚K)	Average thermal effusivity (J/m^2·S·˚K)
Based on lime and FSSA (Present work)	0.33	680.05
Cement based mortar [40]	1.40	1754. 99
Clay-based mortar [41]	0.35	705
Based on lime and CFA (Present work)	0.720	944.9

3.4. Toxicological Risk

The possible dangerousness of the ash depends essentially on its heavy metal content concerning the limits of regulated hazardous substances on one hand, and on the other hand of the overall ecotoxicity of the ash (criterion H14 of Directive 91/689/EEC).

In this Directive the material is toxic if its content in heavy metal is higher to some thresholds as defined in Annex III (Table 3).

Except for the Cr, all the heavy metal contents of the CFA are below the threshold concentration in Table 3, while for the FSSA two heavy metals As and Pb are below the threshold concentration. So these ashes cannot therefore be a priori considered dangerous on the basis of these criteria. The classification of ashes can vary according to the criteria used, depending on the geographic location as well. However, some researchers differ on the definition of toxicity of ashes. Some will put them in landfill if there are considered dangerous while others who consider them non-dangerous will value them [42]. Thus a study of the dangerousness of the ash is necessary to see their possible compatibility with the recovery in construction.

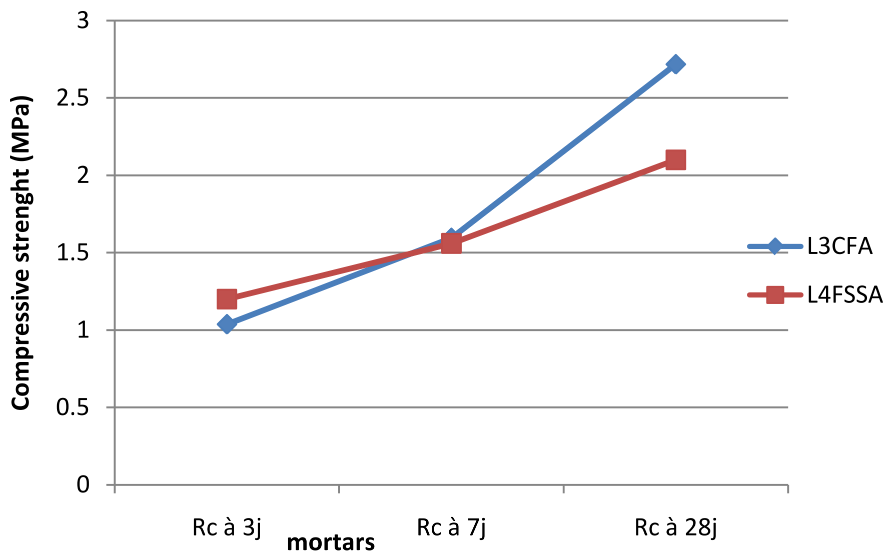

Figure 8. Compressive strength of CFA and FSSA at any age.

Table 3. Comparison of the concentration of minor elements in ashes with thresholds defined in Annex-III.

Element	Concentration (mg/kg) CFA	concentration (mg/kg) FSSA	Threshold concentration (mg/kg)
As	25	16	220
Cr	157	212	150
Cu	47	1264	340
Pb	29	357	840

4. Conclusion

A comparative study between the physico-chemical characteristics of binders based on CFA, FSSA and lime in order to develop an eco-cement was investigated. The following conclusions were derived:

- The chemical composition of ashes is similar to that of cement and the coal fly ash is in Class F.
- The main phases identified on lime are portlandite and calcite while in the coal fly ash quartz (SiO_2), anhydrite ($CaSO_4$), calcium aluminium oxide ($Ca_3Al_2O_6$) and minor phases were found.
- Binder L_3 formed from 60% fly ash and 40% lime gave the best compressive strength at 28 days than L_4 mortar containing 80% of FSSA and 20% of lime.
- Mortar from FSSA and lime have better thermal insulation properties than the Mortar from CFA and lime and these mortars when used in the construction industry will therefore present better thermal insulation properties than the cement-based materials.
- The results of FTIR are in good agreement with XRD results.
- As perspective, other basic knowledge about dimensional stability, water demand, morphology, use of setting accelerators and leaching of heavy metals should be investigated.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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