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Technological and Environmental Behavior of Coal Fly Ash in Lime-Based Materials

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Abstract

Coal fly ash is considered an industrial by-product derived from coal combustion in thermal power plant. It is one of the most complex anthropogenic materials. Its improper disposal has become an environmental concern and resulted in a waste of recoverable resources. The aim of this paper is to study the physico-chemical characteristics of binders based on coal fly ash and lime in order to develop an eco-cement. The various characterization tests carried out are X-ray fluorescence, X-ray diffraction, compressive strengths, thermophysical properties and setting time. X-ray fluorescence and X-ray diffraction were used to determine the chemical composition and phases of fly ash, lime and binders. This allowed us to see that the chemical composition of fly ash is similar to that of cement. Compressive strengths of mortars containing 20%, 40%, 60% and 80% of fly ash have shown that fly ash has a long-term positive effect which might be related to a pozzolanic activity. The L_3 binder consisting of 60% of coal fly ash and 40% lime has a higher compressive strength than the others. The binder setting start time is greater than that of cement but shorter than that of lime. The study of the thermophysical properties of the L_3 binder shows that it has a higher thermal resistance than cement mortar. Moreover, it heats up less quickly because of its low effusivity compared to that of the latter. This analysis highlighted the principal characteristics that must be taken into account to use coal fly correctly in lime-based materials.

Keywords

Coal Fly Ash, Lime, Binder, X-Ray Fluorescence, X-Ray Diffraction, Thermomechanical Behavior

1. Introduction

Cement plants are one of the main industries that emit toxic gases that pollute the atmosphere and contribute to global warming. 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 [1] [2] [3] [4]. 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 [5] [6] [7].

Coal fly ash is considered as an industrial by-product derived from coal combustion in thermal power plants. It is one of the most complex anthropogenic materials. Its improper disposal has become an enviromental concern and resulted in a waste of recoverable resources.

The main application fields of Coal Fly Ash are currently attributed to construction, ceramic, environmental and agricultural sectors. According to statistics, the industrialized countries such as 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 [8]. The reference [9] shows how to use fly as a brake lining ingredient.

The construction industry is a great consumer of resources and materials, which makes it a sector with an enormous potential for the use of waste materials generated by its own activities and those from other sectors. The use of such waste materials allows decrease energy consumption, preserve non-renewable natural resources, and reduce 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, in order to improve some of their properties or confer special characteristics [10].

The most common artificial pozzolana is the fly ash (FA) which is precipitated electrostatically or mechanically from the exhaust gases of coal-fired power stations [11]. ASTM C 618 classifies FA into two groups as Class F and Class C, where Class F has pozzolanic properties and Class C in addition to having pozzolanic properties, also has some cementitious properties [12].

Regarding the problems related to the cost of construction in the building, air pollution such as greenhouse gases and energy expenditure for the production of cement, we propose to study the effect of the integration of the coal fly ash of the Senegalese Chemical Industries plant in lime, to produce an environmentally-friendly cement. Indeed, lime can react with silica in the presence of water to produce a hydrated calcium silicate responsible for the resistance of cementitious materials. The large amounts of solid waste such as coal fly ash produced can be recovered and used as building materials [13] [14] [15] [16].

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

2. Materials and Methods

2.1. Origin of the Materials Used

The fly ash used in this work comes from the thermal power of the Chemical Industries of Senegal in Thies region (**Figure 1**). Fly ash is a fine grey powder and is the principal by-product generated during coal combustion process. It has an absolute density of 2.4 g/cm^3.

The lime, as an important material used in the building construction, has an apparent density varying between 0.6 and 0.75 g/cm^3 while its absolute density is in the range of 2.6 and 2.9 g/cm^3.

2.2. Analytical Procedures

XRF Chemical Characterization

Samples were weighed and then introduced in oven at 105˚C for 24 h to remove moisture prior to the preparation of pellets made by mixing them with 10 wt% of a binder called Licowax [17] [18].

Figure 1. Fly ash.

We use several mixtures by combining fly ash and lime at different proportion (Table 1).

The obtained mixture was homogenized in a mortar and the technology press VANEOX FLUXANA considering a force of 10 N on a surface of a disk of a radius of 11 mm was used to form the pellets. After obtaining the pellets, we used an X-ray portable fluorescence Niton XLT900s (P-XRF) for our X-ray analyzes with a measurement time of 350 s.

XRF was performed with 100% normalization and full fundamental parameter quantification techniques: see Table 2 for specification and operating conditions.

The experimental methods used for the physical and chemical characterization of ashes are shown in Table 3 [14] [19]. It also gives the experimental program concerning the study of the activity of FA in lime-based materials.

For the thermal test, the hot plate transient method was used in an asymmetrical configuration to determine simultaneously the thermal conductivity and thermal effusivity of samples [20]. The different elements that make up the experimental device are represented in Figure 2 as follows.

Table 1. Designation of the mixed Fly ash and lime powders at different proportions.

Mix combinations	Designation
Fly ash	FA100
Lime	CH100
Fly ash + 10 wt% de lime	CH10FA90
Fly ash + 20 wt% de lime	CH20FA90
Fly ash + 30 wt% de lime	CH30FA90

Table 2. Spectrometer specification and operating conditions.

Resolution	**178 eV at Mn Kα**
Window Thickness	12.7 µm Be
Rating	50 kV, 40 µA maximum power of the tube 2 W
Beam diameter	7 mm
Filter	Element analysis
Ag excitation source	Sb, Sn, Cd, Pd, Ag, Mo, Nb, Zr, Sr, Rh, Bi, As, Se, Au, Pb, W, Zn, Cu, Re, Ta, Hf, Ni, Co, Fe, Mn, Cr, V, Ti, Th, and U
Sandwich of Al, Ti and Mo	Ba, Sb, Sn, Cd, Pd, Ag
Cu Filter	Cr, V, Ti, Ca, K
No Filter	Al, P, Si, Cl, S, Mg

Table 3. Experimental methods for the physical and chemical characterization of FA, and for the study of the activity of FA in lime-based materials.

Property	Test methods/Standard
Chemical analysis	X-ray fluorescence (XRF) X-ray portable fluorescence Niton XLT900s (P-XRF) for our X-ray analyses with a measurement time of 350 s. XRF was performed with 100% normalization and full fundamental parameter quantification techniques
Mineralogy	X-ray diffraction (XRD); Cu Kα radiation (λ = 1.54060 Å) 2θ step interval of 0.04˚ (10˚ - 70˚) and acquisition time of 1560 s
Effect on binder hydration: Mineralogical study Setting time	-XRD of mixtures lime-ash-water -Setting time using Vicat apparatus (NF EN 196-3)
Effect on mechanical properties of mortars	Compressive strength of 4 × 4 × 16 cm prisms (NF EN 196-1) Hydration times: 3, 7, 28 days; Each value is the average of 6 tests
Effect on thermal properties of mortars	The thermal test samples were also prepared in a mould of dimensions 10 cm × 10 cm × 2 cm. All thermophysical experiments were performed using samples prepared in a mould of dimensions of 10 cm × 10 cm × 2 cm. The thermal conductivity and effusivity of samples were determined simultaneously using a transient method [20]

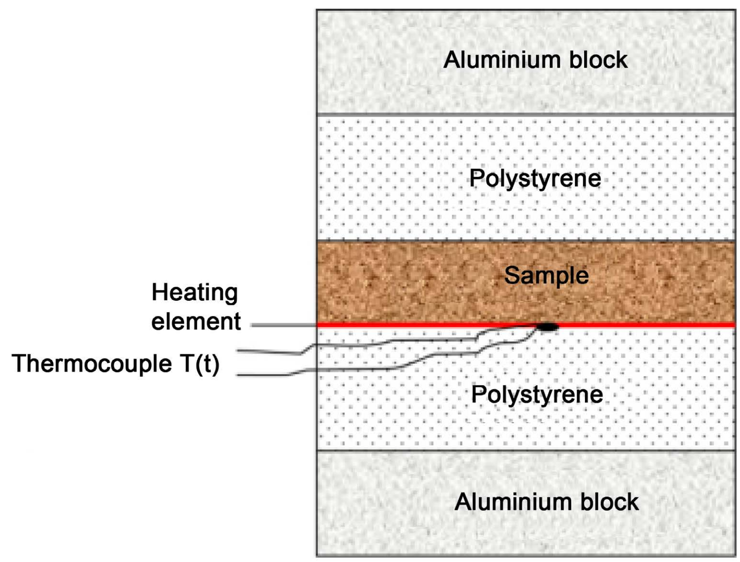

Figure 2. Schema of the experimental hot plate device.

3. Results and Discussion

3.1. X-Ray Fluorescence Analysis

A portable XRF device Niton XLT900s was used to analyze the chemical composition of the ashes in terms of major and minor elements (Table 4). Oxides are the major elements and are expressed in%, while the minor elements are given in mg/kg (ppm).

The chemical analysis of the fly ash of the Chemical Industries of Senegal using X-ray fluorescence spectroscopy showed that it contained the pozzolanic material SiO_2, Al_2O_3 and Fe_2O_3. It is known that these oxides compose the reactive part of pozzolanic materials [21] [22]. The sum of SiO_2, Al_2O_3 and Fe_2O_3 represent more than 70% of FA. So this fly ash is in Class F Fly ashes based on the standard classification [23]. ASTM 618 C classifies fly ash chemically and by coal rank. Class F fly ash content is at least 70% by weight of SiO_2 + Al_2O_3 + Fe_2O_3 and are typically the product of burning high rank coals (bituminous and anthracite).

Class C Fly ashes contain a minimum of 50% by weight of SiO_2 + Al_2O_3 + Fe_2O_3 and a cementitious component, and are normally a product of burning low rank coal (lignite and sub bituminous).

So the Fly ash used in this study is bituminous and we list the work from other different authors in order to give a comparative asset in term of elemental composition with the samples we have used Table 5.

From the chemical analysis of the coal ashes, it is obvious that some elements fingerprint the different regional geological settings between coal fly ash. Thus, the ash of bituminous coal from the Chemical Industries of Senegal shows lower

Table 4. Results of major and minor elements using XRF technique fly ash contents.

Elements	Fly ash
SiO_2	52.1 wt%
Al_2O_3	11.8 wt%
Fe_2O_3	7.59 wt%
CaO	4.68 wt%
MgO	LOD
K_2O	1.72 wt%
TiO_2	1.52 wt%
MnO	0.05 wt%
P_2O_5	1.15 wt%
Pb	29 ppm
Cu	47 ppm
Cr	157 ppm
Zn	54 ppm
As	25 ppm

Table 5. Comparison of major (wt%) element contents measured by other authors working with the same materials.

Element	Fly ash PW (wt%)	[21] (wt%)	[22] (wt%)	[24] (wt%)	[25] (wt%)
SiO_2	52.1	52.7	42.64	55.89	44.42
Al_2O_3	11.8	26	20.49	23.06	32.56
Fe_2O_3	7.59	12.8	11.48	6.66	6.49
CaO	4.68	3.2	14.27	1.64	6.67
MgO	LOD	1.4	2.62	2.57	1.86
K_2O	1.72	0.79	2.84	0.99	1.81
TiO_2	1.52				1.24
P_2O_5	1.15				0.44

concentrations of Al than the other ashes. The chemical composition of bituminous coal ashes from literature data compared in Table 5 shows that the other elements are in the same range. Clearly, the results of the present work are in agreement with these data. As concerns the concentrations of the main ash oxides in the mixtures, they vary between the values corresponding to the individual ashes, but not proportionally [26]. The similarity of these ashes with ours is important to highlight as they are both used in the processing of cement and concrete. Indeed, our ashes show some similarities with one type of cement in Senegal having the following chemical composition: SiO_2 (19.53), Al_2O_3 (7.12), Fe_2O_3 (2.22), CaO (65.5) [27]. This suggests that the coal fly ash presented in this work may be used in civil engineering.

To assess the hydraulic and pozzolanic activity of FA, several mixtures: FA with water and FA with lime and water were prepared and analysed using XRD and mechanical behavior (Table 6).

3.2. Mineralogical Characterization

The mineralogical characterization 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 second within the range of 10˚ ≤ 2θ ≤ 70˚. The diffractometer is linked to a computer equipped with APD software. For data processing we used X Pert High Score software.

Figure 3 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 diffractogram of the fly ash shows the existence of a complex polyphasic material composed of several crystalline phases and a glass phase [28].

Besides glass, the main minerals identified are quartz (SiO_2), anhydrite ($CaSO_4$)

Table 6. Fly ash and lime at different proportion.

Mix combinations	Designation
20 wt% FA + 80 wt% lime	L_1
40 wt% FA + 60 wt% de lime	L_2
60 wt% FA + 40 wt% de lime	L_3
80 wt% FA + 20 wt% de lime	L_4
100 wt% FA	L_5

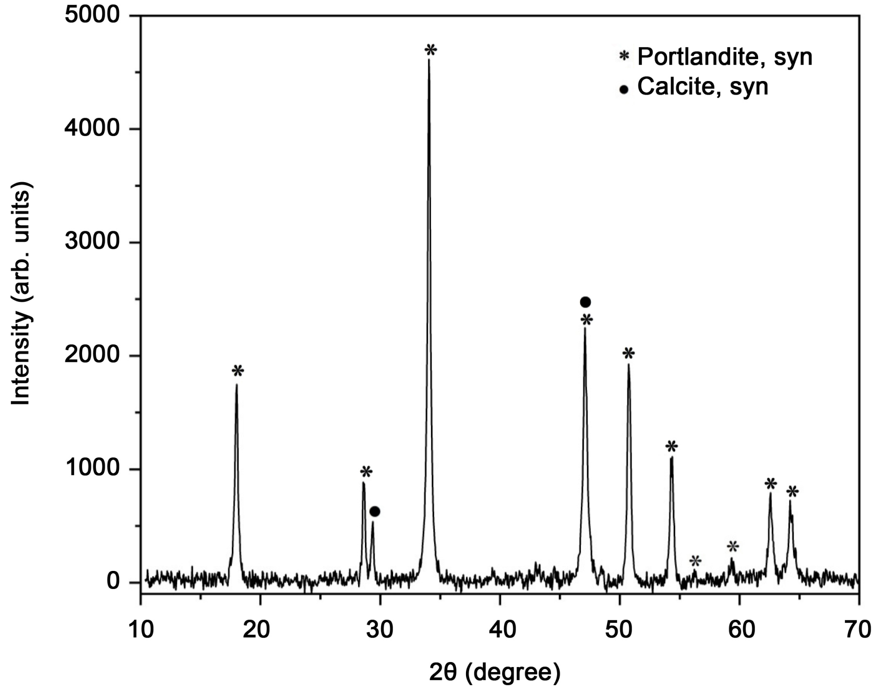

Figure 3. Diffractogram of lime.

and calcium aluminium oxide ($Ca_3Al_2O_6$) and minor phases (Figure 4).

Quartz is the most common mineral, it is found in ash, cement, and sand. $CaSO_4$ anhydrite is similar to less dense and softer gypsum. It is used as retardant in Portland cement clinker. Gypsum, hydrated anhydrite is a mineral added to clinker to form cement Portland. It reacts with calcium aluminates in order to regulate the setting by forming ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}{\cdot}26H_2O$). 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.

In contact with water, tricalcium silicates (Ca_3SiO_5) and dicalcium silicates (Ca_2SiO_4) dissolve in the form of ions. These interact with each other and form hydrated calcium silicates (C-S-H) and portlandite ($Ca(OH)_2$) Figure 5. These reactions give off a lot of heat, so they are exothermic and can act as a catalyst for the hydration reaction. In the case of C_2S, the hydration kinetics are slower and so is the amount of Portlandite formed. The strength of a binder is due to the entanglement of the C-S-H gel. The C-S-H develop on the surface of the

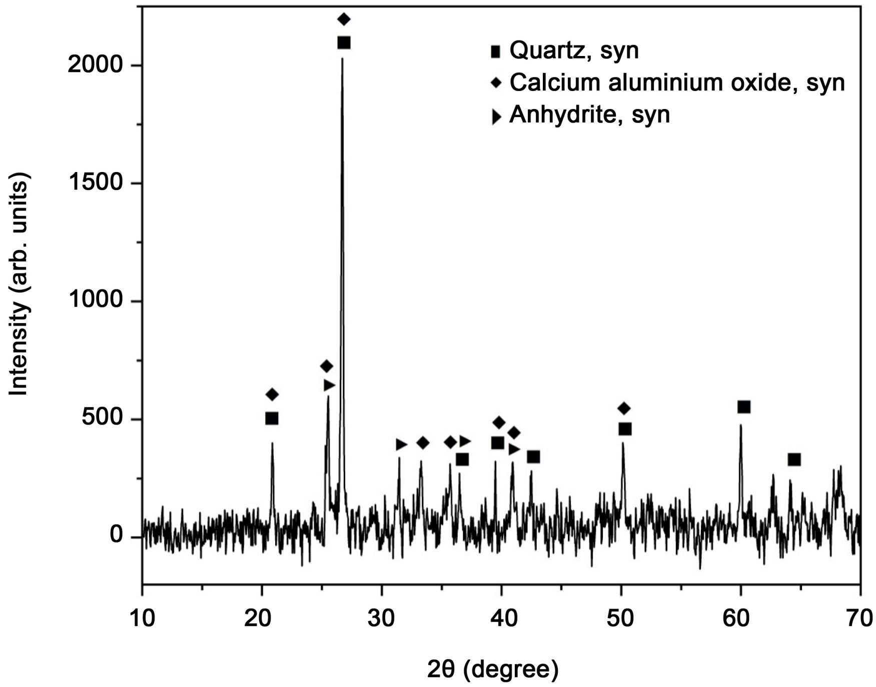

Figure 4. Diffractogram of coal fly ash.

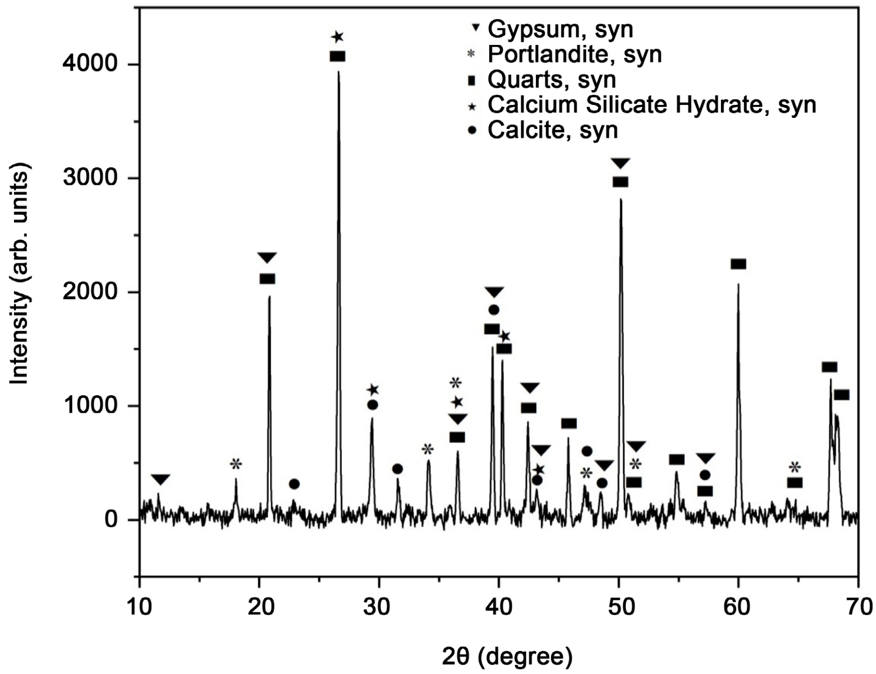

Figure 5. Diffractogram of L328JCM.

grains of the non-hydrated binder and gradually fill in the capillary interstices between the grains. This slows down, after a few hours, the diffusion of ions and water to the anhydrous components of the system. In our binder, a small amount of hydrated calcium silicate was observed on the binder L_3 at 28 days. This weakness could be due to the non-presence of these silicates in our ash [29].

The carbonation of lime is due to the reaction of two compounds: 1) CO_2 from the atmosphere, and 2) the 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 [30].

3.3. Mechanical Behavior

We show the compressive strengths of mortars according to age with the samples we have used Figure 6.

At 100% (L_5) of fly ash at 3 days (COM3d) we have very low resistance compared to others formulations. The hydration of the ash alone has not given a new phase that participates in compression resistance. In this case we can say that only ashes cannot be taken as binders even if their resistance increases over time. And they also have a weak hydraulic character.

When the percentage of ash is low, the amount of silica, alumina and gypse is not sufficient to react with calcium hydroxide. This is the origin of the low resistance observed for sample L_1.

As the percentage of ash increases, compression resistance increases, which shows that there are new phases formed that are responsible for the strength of the binders L_2, L_3 and L_4 binders of the material.

We also note that between 40% and 80% (L_3, L_2 and L_1) of lime in the mixtures with fly ash, we have a drop in resistance. This proves that the greater the quantity of lime, the less we have good resistance in this interval. So in the presence of lime, the fly ash reacts and hardens slightly so we can approve that our ashes have a power pozzolanic.

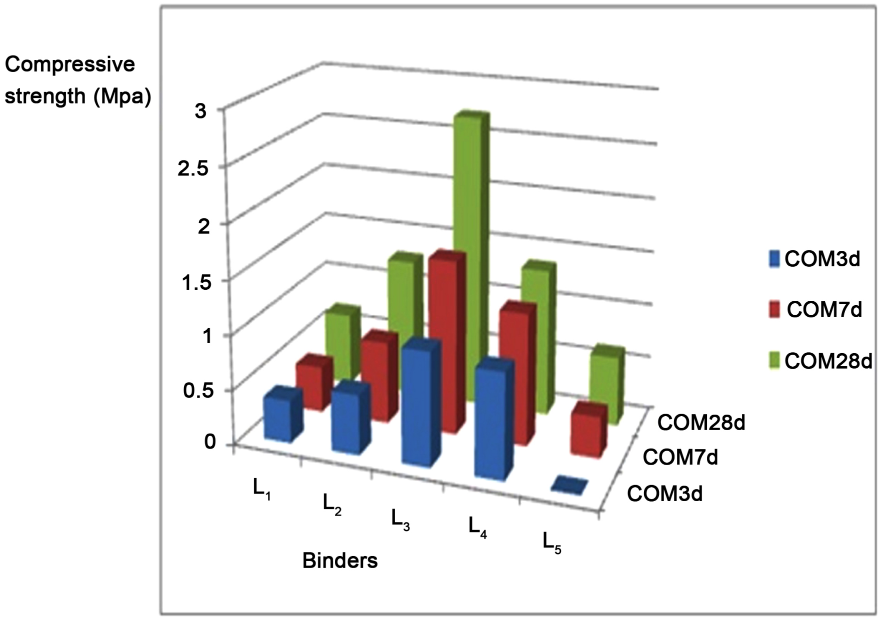

Figure 6. Compressive strengths of mortars according to age.

Mortar L_3 containing 60% of fly ash and 40% of lime has the greatest resistance at any age.

3.4. Setting Time

The setting start time measured with the Vicat apparatus is 5 h 20 min while for cement it is of the order of 2 h. There is therefore a delay in setting the binder which can be attributed to the presence of phosphorus and the non-negligible Zinc content because these elements are known as powerful cement retarders [31].

The presence of gypsum in the coal fly ash can also explain this delay in setting. In addition, the lime has a very slow start of setting which is 10 hours.

3.5. Thermal Properties

As for the tests on pure paste, the mortar containing 60% fly ash and 40% lime is used for this test.

We carried out a series of three tests on the material for more precision and we obtained the results reported in **Table 7**.

The results on thermal tests allowed us to obtain the thermal conductivity and effusivity of the binders.

3.5.1. Thermal Conductivity (λ)

The thermal conductivity reflects the ability of a material to transmit heat by conduction. The test gave an average thermal conductivity (λ) of 0.720 W/m·˚K for binder L_3.

For a cement mortar: λ = 1.4 W/m·˚K; [32].

We find that the thermal conductivity of mortar made from fly ash and lime is lower than that of mortars made from cement. This shows that the fly ash and lime mortar has a higher thermal resistant 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.

3.5.2. Thermal Effusivity (E)

It indicates the capacity of materials to absorb (or restore) more or less quickly heat.

The test results gave an average thermal effusivity of 944.9 J/m^2·s·˚K for our mortars.

For a cement mortar, the effusivity is: E = 1754.99 J/m^2·S·˚K.

Table 7. Thermal properties of the material.

Test	Effusivity	Conductivity
1	989.3	0.725
2	916.5	0.695
3	929.0	0.739

Table 8. Comparison of the concentration of minor elements in ashes with thresholds defined in Annex-III.

Element	Concentration (mg/kg) fly ash	Threshold concentration (mg/kg)
As	25	220
Cr	157	150
Cu	47	340
Pb	29	840

Also, the thermal effusivity value of lime and fly ash mortar is lower than that of the cement mortar. Thus, lime and fly ash-based mortar absorbs heat less quickly than cement mortar. This mortar when used in the construction industry will develop better thermal characteristics than the latter.

3.6. Toxicological Risk

The possible dangerousness of the ash depends essentially on their 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 8**).

Except for the Cr, all the heavy metal contents of these ashes are below the threshold concentration in **Table 8**, so the 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.

Thus a study of the dangerousness of the ash is necessary to see their possible compatibility with the recovery in construction.

4. Conclusions

The physico-chemical characteristics of binders based on coal fly ash and lime in order to develop an eco-cement were investigated. The following conclusions were derived:

- The chemical composition of fly ash is similar to that of cement and fly ash is in Class F.
- The main phases identified on lime are portlandite and calcite while in the fly ash quartz (SiO_2), anhydrite ($CaSO_4$), calcium aluminium oxyde ($Ca_3Al_2O_6$) and minor phases were found.
- Binder L_3 formed from 60% fly ash and 40% lime gave the best compressive strength at 28 days.
- Mortar made from fly ash and lime is more resistant to heat transfer by con-

duction and absorbs less heat than cement mortar. This material when used in the construction industry will therefore present better thermal characteristics than cement based materials.

- As a perspective, other basic knowledge about dimensional stability, water demand, morphology, nature of the bonds, use of setting accelerators and leaching of heavy metals should be investigated.

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Conflicts of Interest

There are no conflicts to declare.

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