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Physicochemical and Mechanical Characterization of Unconventional Fly Ash from the Bargny-Sendou Coal-Fired Power Plant in Senegal

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Abstract: The aim of this work is to characterize unconventional fly ash from the Bargny coal-fired power plant, with a view to its use in the cement matrix as a substitute by-product, either directly in cement, or from its composition as a substitute for clinker at different contents (0%, 5%, 10% and 20%). To this end, we will carry out a physicochemical and mechanical characterization of the ash. On the physical side, we will carry out laser particle size analysis, and determine bulk and absolute densities, Blaine specific surface area and loss on ignition. Chemical and mineralogical characterization was carried out using SEM-EDS scanning electron microscopy technology to determine fly ash reactivation. And finally, the influence of the fly ash effect on the fresh and hardened mechanical behavior of the mortar was studied. The results of the physicochemical characterization showed that the ashes are spherical siliceous with a Blaine specific surface area equal to 4126 cm2 /g. The results also show that the reactivity of the ash, obtained by measuring the activity index, is equal to 77% at 28 days. A study of the fresh and hardened behavior of mortar in which LAFARGE's CEM II/B-LL 32.5 cement is substituted by fly ash at different contents (0%, 5%, 10% and 20%) was also carried out, and the results showed an increase in the workability of the mortar as the ash content increased, and acceptable mechanical performance at 28 days up to 10% substitution.

Keywords: Fly ash; Physicochemical characterization; Mortars; Clinker; Activity index; LAFARGE cement

1. Introduction

In Senegal, there is potential for the valorization of as yet untapped by-products. This is particularly true of fly ash from the Bargny coal-fired power plant. Given the large quantity of ash produced, in the long term there will be a storage problem that could have negative impacts on the environment [1]. It is in this context that we propose to valorize the non-conventional fly ash from the Bargny coal-fired power plant (Senegal), with a view to its use either in the manufacture of concrete and mortar, or as a raw material in the composition of cement. This research involves characterizing this coal by-product, with a view to studying its effects when used in mortar or concrete, as a cement substitute at different levels (0%, 5%, 10% and 20%). To do so, we will determine its physical, chemical or mineralogical characteristics, its reactivity, as well as its medium-term mechanical performance (28 days). To achieve this, we'll perform a laser particle size analysis, then experimentally determine the physical characteristics, i.e. apparent and absolute densities, loss on ignition and Blaine's specific surface area. After the physical analysis, the chemical and mineralogical characteristics will be determined, and finally we will study the effects of fly ash on the behavior of mortar in the fresh and hardened states.

Materials and Methods

To determine physical, chemical and mineralogical characteristics, reactivity and medium-term mechanical performance (28 days), we will perform a laser particle size analysis, determine apparent and absolute densities, loss on ignition, Blaine's specific surface, chemical and mineralogical characteristics, and finally study the effects of fly ash on mortar behavior in the fresh and hardened states.

Physical characterization

Laser particle size analysis

The aim of particle size analysis is to study the size distribution of the various particles in a sample. This rapid, reliable technique was used to determine the particle size distribution of fly ash. The instrument used for our analyses was the FRITSCH ANALYSETTE 22, type NanoTec plus, with a measuring range from 0.01 to 2000 µm (Figure 1). This is a state-of-the-art instrument for measurements in the nanometric range, with very high accuracy and detection of the smallest particles by measurement of the light reflected by a third, rear-facing laser beam (Figure 2). Measurement time with the ANALYSETTE 22 is in excess of one minute in most cases. The instrument comprises four dispersion modules:

• Liquid dispersion module;
• Liquid dispersion module for small quantities;
• Dry dispersion module;
• Dry droplet dispersal module.

In this work, we have chosen the liquid dispersion module, and the liquid used is alcohol. Liquid dispersion is the ideal solution for almost 80% of samples. The sample is introduced into a liquid in a closed circuit. An ultrasonic probe ensures rapid and complete dispersion of agglomerates. This FRITSCH laser particle sizer (Figure 1) meets and exceeds ISO 13320 specifications for repeatability, reproducibility and accuracy.

Figure 1: FRITSCH laser granulometry

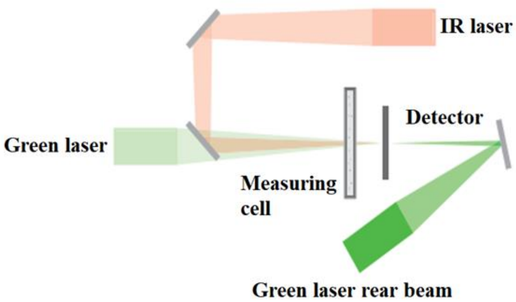

Figure 2: Measuring device for particle size classes in the nanometric range

Before proceeding with the analysis, we must ensure that the material in question is dry, or else place it in an oven at 40°C until a constant mass is obtained. The material must also be homogeneous, with grain size less than or equal to 3.8 mm. In the following, we will analyze the particle size of fly ash from the Bargny-Sendou coal-fired power plant in Senegal and cements synthesized in the laboratory of the DANGOTE CEMENT, whose clinker has been substituted by the same ash at different contents (0%, 5%, 10% and 20%).

Determination of apparent and absolute densities

Density is an important parameter, especially when it comes to determining the composition of concrete, as it can be used to determine either the mass or the volume of the different granular classes of a compound, depending on the desired characteristics. Knowing the absolute density of fly ash is necessary for determining Blaine's specific surface area, in accordance with current regulatory requirements [2]. The apparent and absolute densities of fly ash were determined in accordance with [3] and found to be 814.5 kg/m^3 and 2,484.06 kg/m^3 respectively (according to equations 1 and 2). The densities found are of the same order of magnitude as those found in the literature.

$$\rho_{app} = \frac{m2-m1}{V} \quad (1)$$

With:

m_1: Empty container mass in grams

m_2: Mass of sample container in grams

V: Vacuum container volume

ρ_{app} apparent density in g/cm^3

$$\rho_{abs} = \frac{mp2-mp1}{(mp4-mp1)-(mp3-mp2)}\rho_{ethanol} \quad (2)$$

With:

m_{p1}: Vacuum pycnometer mass in grams

m_{p2}: Filled pycnometer mass in grams

m_{p3}: Filled pycnometer mass (sample and ethanol) in grams

m_{p4}: Ethanol-filled pycnometer mass in grams

ρ_{abs}: Absolute density in g/cm^3

Blaine's specific surface

It is used to measure the fineness of grind of a cement (Figure 3). It is characterized by the specific surface or total developed surface of all the grains contained in one gram of cement [4]. The specific surface expressed in cm^2 /g is calculated using the following equation 3:

$$S = \frac{10\times k\times\sqrt[3]{e}\times\sqrt{t}}{\rho(1-e)\times\sqrt{\eta}} = (K\times\sqrt{t})/(\sqrt{\eta}) \quad (3)$$

With

S: Specific surface area (cm^2 /g)

k: Device constant

e: Porosity of the packed layer

t: Time measured in seconds

ρ: Density (g/cm3)

η : Air viscosity at test temperature in Pa.s

K =$(10\times k\times\sqrt[3]{e})/(\rho(1-e))$

Figure 3: Blaine fineness meter

Determination of the specific surface area of fly ash follows the same procedure as for cement. To do this, we used a standard cement of type CEM I 52.5N SN4C of known specific surface and absolute density, from which the constant K of the device was deduced. The latter was then used to calculate the Blaine specific surface of the fly ash, whose mean value is given in Table 1 and is equal to 4126 cm^2/g.

Table 1: Blaine surface area values for fly ash after 3 measurements (cm^2/g)

SSB1	SSB 2	SSB 3	**SSB MOY**
4128	4223	4028	**4126**

Determining specific surface area using the BET method

The BET (Brunauer, Emmett, Teller) method determines specific surface area, and is based on the phenomenon of physical adsorption. It consists of measuring the vapor likely to condense on the exposed solid surface of the sample. The quantity of helium (He) or nitrogen (N_2) gas adsorbed at a given pressure is measured to calculate the sample's specific surface area. The BJH (Barrett, Joyner, Halenda) method uses adsorption data to retrieve intrinsic solid data such as pore volume and size. The BJH method thus links thermodynamic data, sorption and desorption isotherms, with geometric data, intrinsic properties of the solid, pore size distribution.

The "VacPrep 061 from Micromeritics" has 2 sample compartments (Figure 4), the first in which the sample is heated under vacuum to dry and release the material, and the second in which the sample is cooled so that it is ready for use.

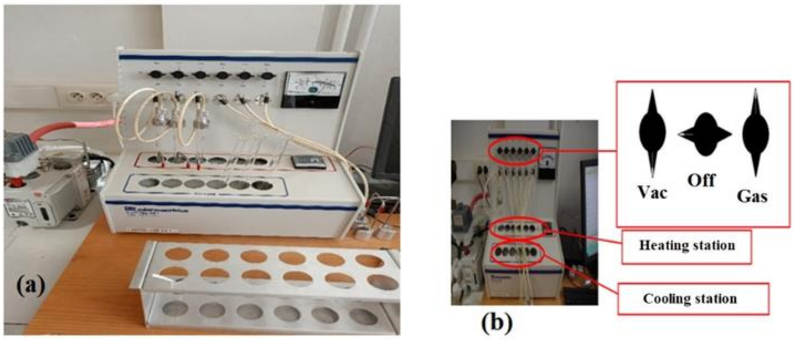

Figure 4: a°) Micromeritics VacPrep 061 and b°) Sample preparation locations and valve descriptions

The instrument also features a valve to control gas inlet and pressure (Figure 4b). A flexible hose connects each valve to a test tube. This hose is attached to the tube by means of a system comprising a coupling, a nut, a ferrule and an O-ring (Figure 5).

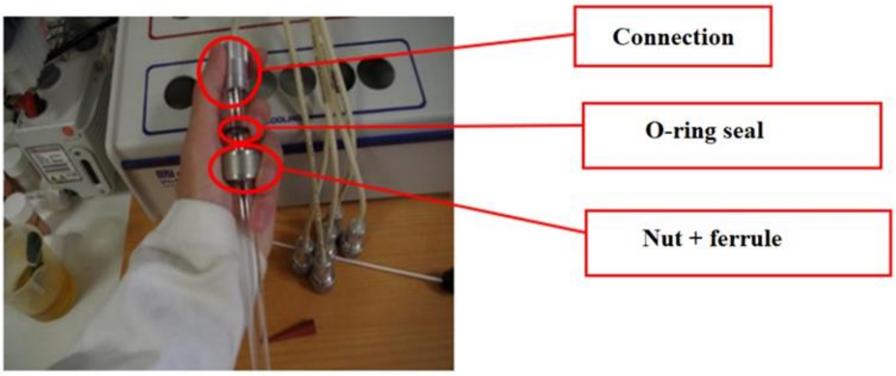

Figure 5: Presentation of the test tube attachment hose

Fire loss

Loss on ignition is determined in accordance with [4]. It corresponds to the bound water content of the concrete, the organic matter content and the CO_2 of the carbonates. The test consists in taking a mass m_3 equal to 10.3g of product dried at 80°C in the previously calcined crucible, then placing it in an oven at 975°C ± 25°C for 30mn, then weighing the product obtained to obtain a mass m_4 equal to 9.98g. The loss on ignition (LOI) is obtained using equation 4 and is equal to 3%:

$$PAF = \frac{m3-m4}{m3} \times 100 \quad (4)$$

Chemical and mineralogical characterization

Chemical characterization of the fly ash was carried out to determine either its chemical composition using ***x-ray*** fluorescence or to determine free and bound chemical elements using a simple anion-cation assay.

Chemical composition

Table 2 gives the chemical composition of fly ash in mass percentage of the oxides that are present in the majority and in mg/kg of the elements that are in the minority (Mg, Cd, Pd and S). From Table 2 we deduce the overall mass percentage of silica, aluminum and iron oxides, which is equal to 81.83%. This value provides information on the pozzolanic properties of fly ash in accordance with ASTM C618. Table 2 also provides information on the silico-aluminous nature (SiO_2 = 48.94 and Al_2O_3 = 29.65) of the fly ash from the Bargny-Sendou coal-fired power plant.

Table 2: Chemical composition of fly ash

Majority items in % by mass		**Minority interests in mg/kg**	
SiO_2	48,94	Mg	0
Al_2O_3	29,65	Cd	0,08
Fe_2O_3	3,24	Pb	0,182
CaO	9,29	S	2,24
MgO	1,55		
TiO_2	1,55		
P_2O_5	2,19		
Na_2O	0,1		
K_2O	0,46		
SO_3	2,67		
SiO2+ Al2O3+ Fe2O3	**81.83**		

Analysis using SEM-EDS scanning electron microscopy technology

Scanning Electron Microscopy SEM-EDS is a laboratory microscopic analysis technique using a field-effect gun (electron-matter interaction). This technique produces very high-resolution images of the surface of a sample, and is therefore used to study the chemical composition and morphology of solid materials. SEM analyses were carried out using a JOEL IT 300 SEM (Figure 6) from the CMEBA platform at the University of Rennes 1. Samples were gold-plated prior to each analysis to ensure good surface conductivity.

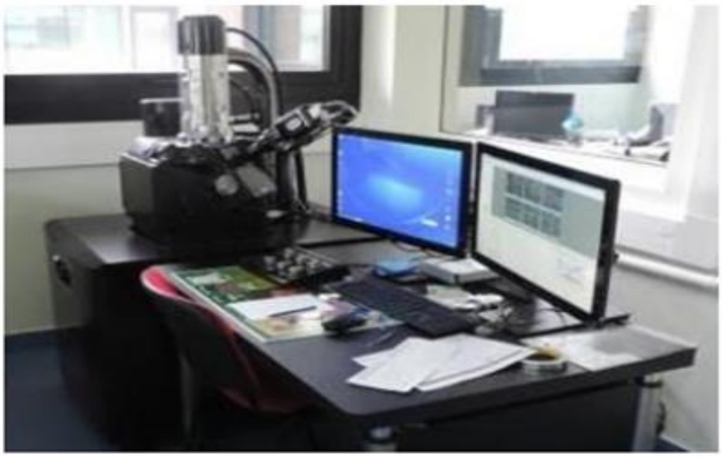

Figure 6: SEM device used

Determination of free and bound chemical elements

Determining the chloride content of fly ash by assay involves first conditioning the product to a particle size of less than 315 µm, followed by extraction of total chlorides with dilute nitric acid (HNO_3) and free chlorides with water. For total chlorides, 5 g of fly ash was removed and placed in a 250ml beaker, followed by 5ml of water. The solution was stirred for 2 minutes, then placed in a hot water bath at 80°C. Next, 100ml of dilute 2.4N nitric acid is gradually added to the solution, and the mixture is stirred for 30 min. The solution is then filtered and titrated. For free chlorides, the only difference with total chlorides is in the extraction and concentration of nitric acid used (2 ml of 6N concentrated nitric acid is added to the solution at the time of assay). The pH of the solution was measured and its value recorded in Table 8, including organic matter (OM) content and loss on ignition. Other elements were determined by ion chromatography (Figure 7).

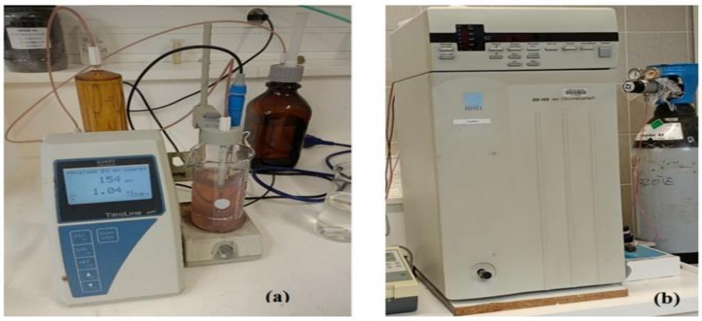

Figure 7: a°) Titrimetric apparatus for chloride determination b°) DX.100 ion chromatography apparatus from INSA Rennes

Reactivated fly ash

As part of the characterization of fly ash, its reactivity was studied by measuring its activity index (i). This measurement is required by [5] and is the ratio of the compressive strengths of standardized mortar specimens of the same age, some prepared with 75% test cement, in this case CEM I 52.5 N from LAFARGE in compliance with [6], and 25% ash addition (by mass), others prepared with 100% test cement (equation 5).

$$\boldsymbol{i} = \frac{R_p}{R_q} \qquad (5)$$

With:

I: Activity index

$\boldsymbol{R_p}$: Compressive strength of mortar with 25% ash by mass of test cement

$\boldsymbol{R_q}$: Compressive strength of control mortar with 100% test cement.

According to [7], the activity index at 28 days and 90 days must be greater than 75% and 85% respectively.

Manufacture of mortar specimens for activity index measurement.

The cement used, as previously indicated, is CEM I 52.5 N. The sand used is a standardized siliceous CEN sand. Prismatic specimens (4x4x16 cm^3) were manufactured in accordance with [8]. The three mortars used were:

- a reference mortar with 100% test cement;
- a mortar with 75% CEM I 52.5 N cement and 25% fly ash.

After manufacture, the specimens were conditioned, then demolded after 24 h and stored in water at 20°C for curing (Figure 8 b) until the following times: 28 days and 90 days.

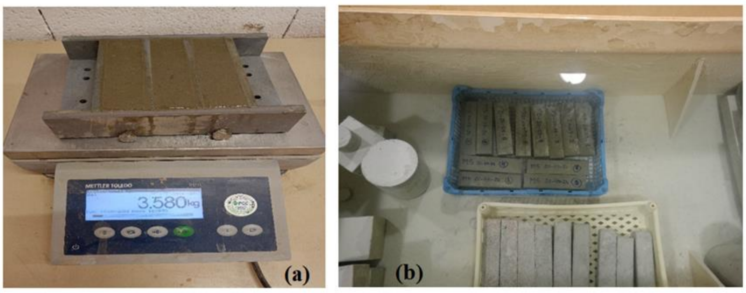

Figure 8: a°) Test tubes ready for packaging, b°) Test tubes preserved in water

Effect of fly ash on fresh and hardened mortar behavior

The effect of using fly ash was characterized by a study of the fresh and hardened behavior of the mortar. For this purpose, we adopted a mortar formulation in which LAFARGEHOLCIM's CEM II/B-LL 32.5 cement was substituted by fly ash, as shown in the following Table 3.

Table 3: Mortar formulations where cement has been substituted by fly ash at different rates (0%, 5%, 10% and 20%).

Materials	% substitution			
	MCEMIICV0	**MCEMIICV5**	**MCEMIICV10**	**MCEMIICV20**
Cement (g)	900	855	810	720
Sand (g)	2700	2700	2700	2700
Water (g)	450	450	450	450
Ash (g)	0	45	90	180

Effect of fly ash on fresh mortar behavior

To study the fresh behavior of mortars formulated with fly ash (0%, 5%, 10% and 20%) in place of CEM II 32.5 cement, we used the Abrams mini cone (½ Abrams cone scale: h =15 cm; lower base =10 cm and upper base =5 cm) to measure mortar slump and spread. The slump and spread measurements were applied to mortars ready to be molded and intended for use in determining compressive and flexural strengths. To do this, the mortar was filled into the mini cone in two layers, each of which was pricked by a pricking rod at 25 and 15 strokes respectively for the first and second layers. Immediately after removal of the cone, slump is measured by determining the difference between the height of the cone and the highest point of the slumped sample, as shown in Figure 9, then immediately the spread is measured as the average of two perpendicular diameter measurements.

a) b)

Figure 9: a) Mini-cone slump measurement, b) Mini-cone spread measurement

Effect of fly ash on hardened behavior

Once the test bodies had been made, they were conditioned by placing the 4x4x16 specimens in water at a temperature of 20°C for 28 days, which corresponds to the hardened state. Behavior in this hardened state is assessed by mechanical bending and compression tests. For this purpose, the Controlab press shown in Figure 10 was used.

Figure 10: Controlab press for flexion-compression at INSA Rennes

3. Results and discussion

Analysis of granulometry results

The particle size analysis of fly ash from the Bargny-Sendou coal-fired power plant in Senegal and cements synthesized in the laboratory of the DANGOTE CEMENT, whose clinker was substituted by the same ash at different contents (0%, 5%, 10% and 20%), led to the following results shown in Table 4 and Figures 11 and 12.

Table 4: Granular distribution of the fly ash sample.

Sample	%	0 - 2 µm	2 - 20 µm	20 - 200 µm	0.2 - 2 mm
Fly ash	Partials	4,9	40,0	54,7	0,4
	Cumulative	4,9	44,9	99,6	100,0

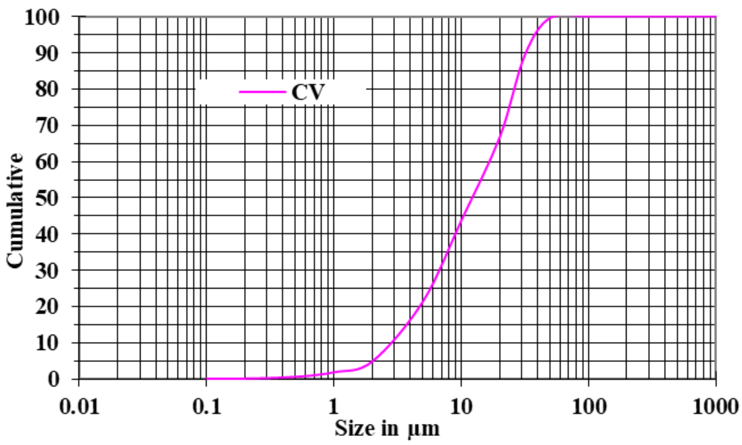

Figure 11: Cumulative percentage of ash as a function of grain size in µm

Table 5 and Figure 12 show the results of the fly ash granular analysis correlated with the cement results (C0%, C5%, C10% and C20%).

Table 5: Diameters at 25, 50, 75 and 90% fly ash and cement bypass.

EN µm	CV	C0%	C5%	C10%	C20%
25% diameter	5,6	3,7	3,8	3,6	3,4
50% diameter	12,5	6,4	6,7	11,6	11,1
Diameter at 75	13,6	17,3	16,4	11,6	11,1
90% diameter	31,9	27	25,9	22,3	20,6

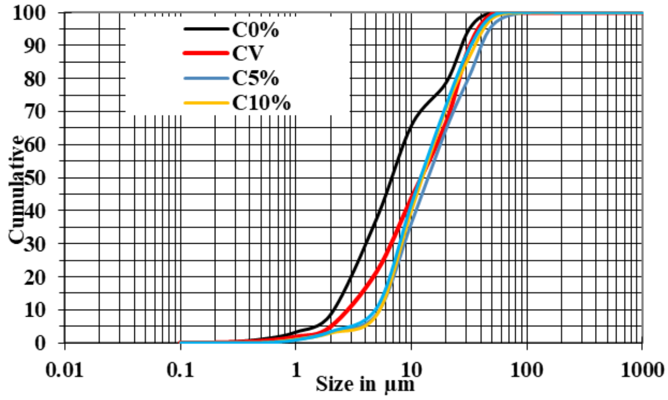

Figure 12: Cumulative percentages of ash and cement as a function of grain size in µm.

Analysis of Figure 12 and Tables 4 and 5 shows that fly ash is made up of fine grains, i.e. 90% passing 31.9 µm diameter and 100% passing 63µm diameter. On the other hand, fly ash contains more coarse particles than cements manufactured using ash instead of clinker, and it can also be seen that as the ash content increases, the diameters at 90% passing decrease. We can therefore assume that the substitution of fly ash for clinker would have a beneficial effect on the fineness of manufactured cement.

Results for specific surface area

Table 6 gives the BET specific surface area results for three samples of the same fly ash (CV1, CV2 and CV3) and averaging gives the BET specific surface area, which is equal to 7.3143 m^2/g.

Table 6: BET specific surface area of fly ash (m^2/g)

CV1	CV2	CV3	CV-average
7,271	6,8775	7,7944	7,3143

Exploitation of results using SEM technology

Figure 13 shows images obtained after fly ash analysis using the SEM method.

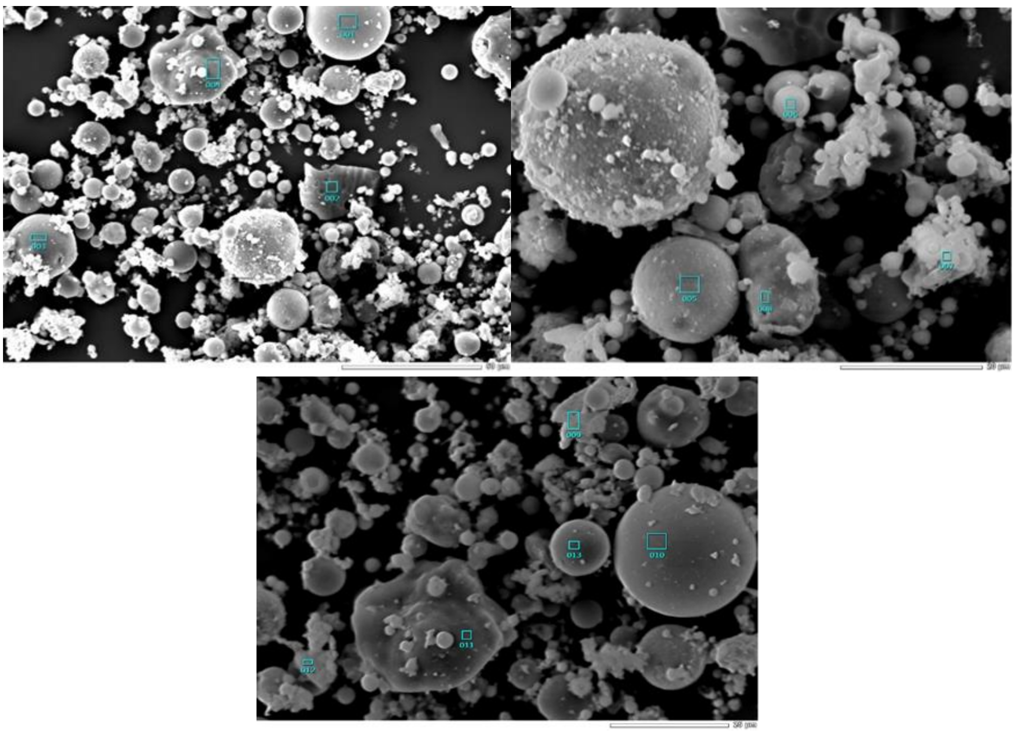

Figure 13: SEM images coupled with EDS analysis

Table 7: Chemical elements from EDS analysis of a fly ash sample.

Nature	C	O	Na	Mg	Al	If	S	K	Ca	Ti	Fe	Cu	Ba
% Atomic	32,65	39,91	0,31	0,37	7,27	16,85	0,32	0,6	0,64	0,26	0,8	0,01	0,01
% Massive	21,66	32,28	0,29	0,4	9,59	24,05	0,5	1,18	1,34	0,64	2,29	0,04	0,04

Figure 13 shows the images obtained from the SEM and the analysis of the morphology showed the presence of spherical particles of regular shapes and the presence of a few particles of irregular shapes, which may be due to the unburnt carbon content expressed as loss on ignition. At the same time, the EDS analyses in Table 7 show that the predominant chemical elements in the sample by mass percentage are oxygen (32.28%), silicon (24.05%), carbon (21.66%) and aluminum (9.59%). Analysis of these SEM-EDS results shows that the fly ash used is of a siliceous nature with a spherical shape. In fact, the spherical and regular shape of the ash will have a positive effect on its reactivation and workability when used in concrete or mortar.

Exploiting results for free and linked items

The results of chemical analysis of fly ash for free and bound chemical elements are reported in Table 8, together with loss on ignition, pH and organic matter content. From Table 8, the results show that the values for free element content and loss on ignition are well within the requirements of [7] for fly ash obtained from pulverized coal combustion. It can also be seen that the free calcium oxide content is well below 1.5%, which satisfies the requirements of the aforementioned standard in terms of fly ash stability.

Table 8: Free and bound chemical elements

Elements	*Shapes*	*Fly ash*	*EN 450-1*
PH		*11*	
MO %		*1,05*	
Loss between 550° and 975° %.	*CaCO3*	*1,1*	
Loss on ignition		*3*	*Category A*
[Cl^-] %	*Free*	*0,008*	*< 0.1%*
	Related	*0,032*	*-*
[SO_4^-] %	*Free*	*0,40*	*< 3%*
	Related	*0,2*	
[Na^+] %	*Free*	*0,046*	
	Related	*0,003*	
[K+] %	*Free*	*0,0067*	*Total alkali < 5%.*
	Related	*0,0173*	
[Mg_2^+] %	*Free*	*0,016*	*< 4%*
	Related	*0,0204*	
[Ca_2^+] %	*Free*	*0,29*	*< 1.5%*
	Related	*0,63*	

Analysis of fly ash reactivity results

After 28 days of curing, flexural and compressive strengths were determined by applying bending and compressive forces to the specimen surface using the automatic Controlab press as shown in Figure 14a. The dimensions of the test bodies were measured using a digital caliper as shown in Figure 14 b.

Table 9 gives the flexural and compressive strengths of the mortars as well as the fly ash activity index. In fact, the activity indices at 28 days and 90 days are determined and are equal to the values respectively 77% and 96% which remain higher than the limit values given by [7].

Figure 14: a) Controlab press for flexural-compressive strength tests and b) Specimen size measurements with calipers

Table 9: Activity index value at 28 and 90 days

Maturity	% Of ash in mortar	Flexion		Compression		Activity index (%)
		Force (KN)	σ (MPA)	Force (KN)	σ (MPa)	
28 DAYS	0%	3,503	6,991	85,282	52,451	**77**
	25%	3,326	6,390	66,492	40,146	
90 DAYS	0%	3,471	8,135	87,757	54,848	**96**
	25%	3,649	8,553	84,288	52,680	

Analysis of the influence of fresh fly ash

Table 10 and Figures 15 and 16 show the slump and mini-cone spread results obtained for mortars for which cement was substituted by ash at different contents (0%, 5%, 10% and 20%) with a water to binder ratio equal to 0.5.

Table 10: Consistency measurements for mortars with and without fly ash

Measures	Mortars			
	MCV0	MCV5	MCV10	MCV20
Settlement (cm)	8,5	9,2	9,5	9,8
Spread (cm)	13,95	13,6	13,2	12,7

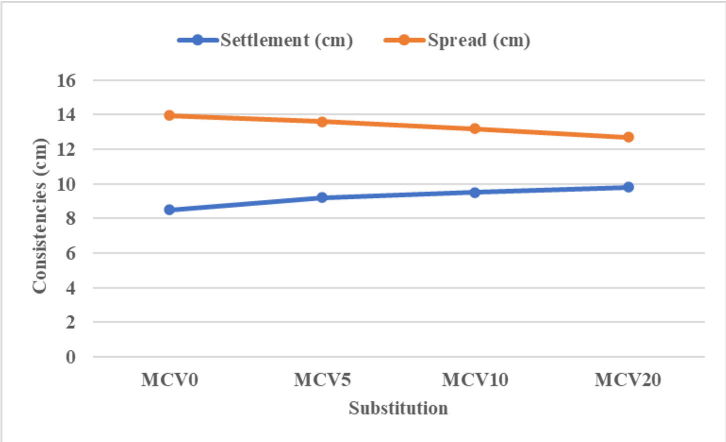

Figure 15: Measurement of mortar consistency as a function of fly ash substitution rate

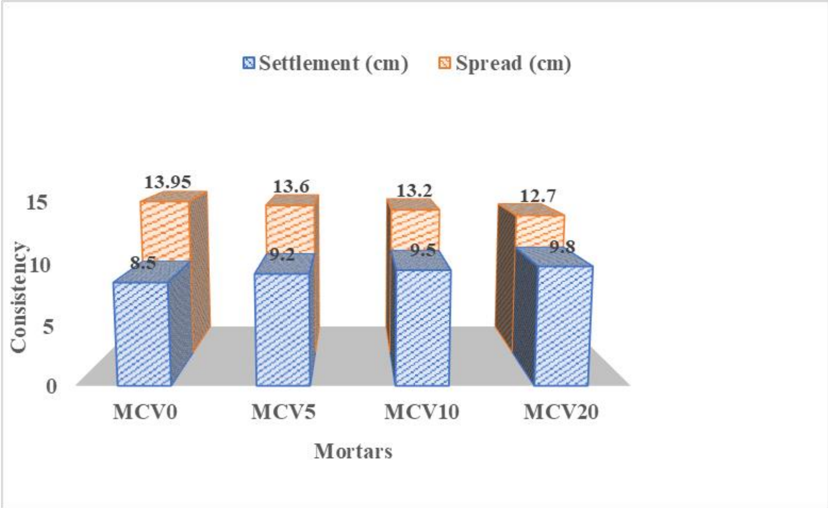

Figure 16: Measuring mortar consistency as a function of fly ash substitution rate in 3D.

Figures 15 and 16 show an increase in slump and a decrease in spread compared to the control mortar (MCV0) as the substitution rate increases. Indeed, it can be said that the substitution of cement by fly ash improves mortar consistency, and hence workability.

Analysis of the influence of fly ash in the hardened state

Table 11 and Figure 17 give the results of the mechanical performance in flexion and compression of the different types of mortar produced with or without substitution of Lafarge CEM II 32.5 cement by fly ash. Indeed, these results show that mechanical performance in compression decreases as the ash content increases, but nevertheless up to 10% substitution, the stresses found are higher than the characteristic stress of the cement used at 28 days. This confirms the results obtained with DANGOTE's CEM II 32.5 cement in Senegal.

Table 11: Flexural-compressive strength results for mortars after 28 days curing

28-day mortars	Flexion		Compression	
	Force (kN)	Stress (MPa)	Force (kN)	Stress (MPa)
MCV0	3,5	7,0	58,5	35,9
MCV5	2,8	5,6	54,5	33,1
MCV10	2,9	6,1	53,2	32,9
MCV20	2,7	5,5	48,3	29,9

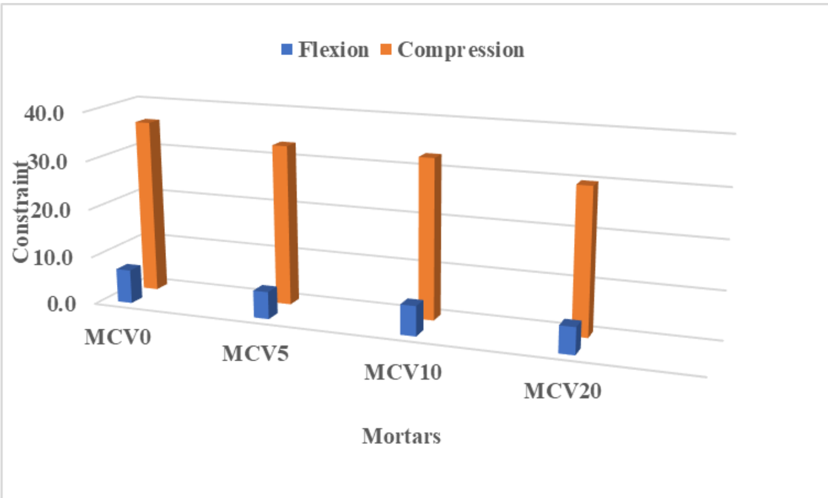

Figure 17: Evolution of normal bending and compressive stress as a function of the rate of cement substitution by fly ash.

4. Conclusion

The characterization of unconventional fly ash from the Bargny coal-fired power plant in Senegal is a fundamental step towards its valorization in the cement matrix as a substitute for cement at different contents (0%, 5%, 10% and 20%). In this context, a physico-chemical and mineralogical characterization was carried out, along with a study of the reactivity of the ash through its pozzolanic activity index, and mechanical characterization through compression and bending tests. The results obtained showed that the ashes are siliceous, spherical in shape, with a Blaine specific surface area of 4,223 cm2 /g greater than that of the cements used, which range from 3,000 to 4,200 cm2 /g. The results also show an increase in workability as the ash content of the mortar increases. And the activity index value found (77%) at 28 days tells us about the pozzolanic character of the fly ash from the Bargny coal-fired power plant, in compliance [9].

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