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Abstract: Cementitious mortars face durability problems in aggressive environments, which reduces the lifespan of cement buildings. The objective of this study is to investigate the physicochemical and durability properties of cementitious mortars with added sugarcane bagasse ash (CBCS). The ashes were produced by calcining sugarcane bagasse at temperature ranging from 600 to 750 °C for 2 or 3 hours. The microstructure and the pozzolanic activity of the ash were investigated. Mortars were then manufactured by partially replacing 0-25 wt % of the cement with ash. Their physicochemical properties, such as bulk density, setting time, expansion, and durability in an acidic environment, were studied. The results show that the ash has irregularly shaped grains and high porosity. They are rich in amorphous silica (58.37 to 75.91 wt.%) and have pozzolanic indices above the minimum value of 75 % required by ASTM C618 for pozzolanic materials. A decrease in density and an increase in the initial and final setting times of cement pastes modified with ash are observed. The addition of ash did not cause significant expansion of the pastes. Durability studies in aggressive environments have shown that mortars modified with bagasse ash are more resistant to acid action due to the increased presence of calcium silicate hydrates (CSH), resulting from the pozzolanic reactivity between portlandite (CH) produced during cement hydration and the amorphous silica in bagasse ash. The use of these ashes as cement additives is a solution to improving the lifespan of cement buildings.

Keywords: sugarcane bagasse ash, cement mortars, pozzolanic reactivity, durability, aggressive environments, hydrated calcium silicates

Propriétés physico-chimiques et de durabilité des mortiers cimentaires amendés aux cendres de bagasses de canne à sucre

Résumé : Les mortiers cimentaires sont confrontés à des problèmes de durabilité en milieu agressif ce qui réduit la durée de vie des bâtiments issus. Ainsi, L'objectif de ce travail est d'étudier la durabilité des mortiers cimentaires amendés aux cendres de bagasses de canne à sucre (CBCS). Pour ce faire, les cendres ont été produites par calcination de bagasses de canne à sucre à des températures allant de 600°C à 750°C avec des paliers de 2 et 3 heures. Les cendres ainsi obtenues ont été caractérisées puis leur indice d'activité pouzzolanique déterminé. Ensuite, les mortiers ont été élaborés par substitution partielle de 0 à 25% de ciment par les cendres puis leur durabilité en milieu acide a été étudiée. Il ressort des résultats obtenus que les cendres sont riches en silice amorphe (58,37 à 75,91%) et présentent des indices pouzzolaniques supérieurs à la valeur minimale de 75% fixée par la norme ASTMC618. Par ailleurs, la présence des CBCS au sein de la matrice cimentaire engendre la consommation de la portlandite (CH) et favorise la formation de l'ettringite ($C_6A\bar{S}_3H_{32}$) et des silicates de calcium hydratés (CSH). Aussi, la présence des CBCS diminue la densité et augmente le temps de prise des pâtes de ciment. Les études de la durabilité des mortiers en milieu agressif ont montré que les mortiers amendés aux cendres de bagasse résistent mieux à l'action de l'acide due à la présence des CSH résultant de la réactivité pouzzolanique entre la portlandite produite lors de l'hydratation du ciment et la silice amorphe des CBCS. L'utilisation de ces cendres en tant qu'additif cimentaire constitue une solution à l'amélioration de la durée de vie des pièces cimentaires.

Mots clés : cendre de bagasse de canne à sucre, mortiers cimentaires, réactivité pouzzolanique, durabilité en milieu agressif, silicates de calcium hydratés et ettringite.

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1. Introduction

Developing countries in sub-Saharan Africa are experiencing rapid growth in the construction sector. This expansion encompasses infrastructure development, including roads, hospitals, and schools. To meet the needs of this vast program, these countries, particularly Burkina Faso, have become major consumers of cement, one of the most widely used construction materials after steel and aluminum [1]. However, cement production is not without consequences; it is energy-intensive and polluting. The production of one ton of cement releases approximately one ton of carbon dioxide and consumes 4.9 GJ of energy [2][3]. Cement-based products also face sustainability issues. The lifespan of a structure exposed to a harsh industrial or natural environment can be significantly reduced. The penetration of chloride ions (Cl^-), sulfate ions (SO_4^{2-}), and carbon dioxide (CO_2) into the cement matrix reduces the pH of the environment, leading to deterioration of mortar or concrete. Additionally, the widely used Portland cement triggers alkali reactions that are detrimental to the structure [4].

Furthermore, the hydration of cement releases portlandite, which affects the mechanical performance and durability of mortars [1]. To overcome the difficulties associated with cement use, pozzolanic materials are commonly used as partial substitutes for cement [5]. Pozzolan is an aluminosilicate composed of fine particles, whose presence in a material reduces its porosity and makes it more compact. Its incorporation into cement causes a reaction known as the pozzolanic reaction between the portlandite in the cement and the amorphous silica in the glassy phase of the pozzolan [6]. This reaction promotes the formation of new CSH hydrates and reduces the amount of Portlandite. The use of pozzolan in mortar or concrete results in low hydration heat release, high long-term compressive strength, low permeability, high sulfate resistance, and low alkali-silica reactivity [7][8]. Thus, the use of plant-based raw materials as pozzolanic materials has been little explored in the literature compared to mineral raw materials, to the best of our knowledge [9]. However, these biomasses are most often waste products whose storage poses real problems. Therefore, the use of such waste could be an alternative way of combating environmental pollution. In addition, the use of natural plant resources as building materials can be part of a sustainable development approach, as it addresses the economic and environmental challenges of the 21st century. Among these plant-based raw materials is sugarcane bagasse, which is readily available in Burkina Faso. It is an industrial by-product used to fertilize agricultural soils and as a medium for wastewater treatment. Its use as a building material has also been the subject of scientific research [10]. According to Ouedraogo et al. [10], the addition of sugarcane bagasse to the clay matrix significantly enhances the mechanical strength of adobe bricks by improving the microstructure. These authors believe that the presence of bagasse fibers induces hydrogen bonding between bagasse molecules and clay minerals, which could explain the improvement in physical and mechanical properties. This work is a continuation of our previous research on the use of sugarcane bagasse as a cement additive. Previously, we demonstrated that ash obtained by calcining sugarcane bagasse at 600-750 °C for 2 or 3 hours exhibited pozzolanic properties and could therefore be used as a partial cement substitute in construction [11].

The main objective of this work is, on the one hand, to investigate the impact of sugarcane bagasse ash on the physicochemical properties of cement paste. In particular, we will monitor the density, setting time, and hardening time of the cement, and its expansion with the addition of ash. On the other hand, this work aims to evaluate the durability of cement mortars modified with sugarcane bagasse ash in acidic environments (sulfuric and hydrochloric).

2. Materials

The ashes were produced by calcining sugarcane bagasse from the New Sugar Company of Burkina Faso (SN–SOSUCO–BF) at temperatures ranging from 600 to 750 °C. The physical, chemical, and mineralogical characteristics of ashes were studied in our previous work [11]. We present the main findings of these characterizations here.

The ashes are composed (Tables 1 and 2) mainly of silica (SiO_2) and alumina (Al_2O_3) with relatively low contents of iron oxide (Fe_2O_3), potassium (K_2O), calcium (CaO), magnesium (MgO), and sodium (Na_2O). The presence of silica in the ashes is due to the absorption of silicic acid from the soil and its deposition, in amorphous form, in all parts of the plant [12]. The sum of the ash ($SiO_2 + Al_2O_3 + Fe_2O_3$) ranges from 79 % to 87 %, which is higher than the minimum value of 70 % required for classification as a pozzolan [13]. It should also be noted that the ash obtained is low in lime (CaO), with an average CaO content of 1.59 %, well below 10 %. Therefore, based on their composition and in accordance with

ASTM C 618, we can conclude that the ashes are a type F pozzolan. The K_2O content is high and could affect mortar durability through alkali-silica reactions. However, K_2O can accelerate the dissolution of amorphous silica by increasing the pH of the interstitial solution [14]. In addition, the sum of the mass percentages of the alkali oxides Na_2O and K_2O exceeds the minimum value of 0.95% reported in the literature, indicating that the ashes are rich in alkali oxides [15][16][17]. The ashes as a whole showed high potassium oxide (K_2O) contents, averaging 6.09 % for 2 hours and 4.57 % for stage 3 hours. These results were expected, as potassium ions are the most abundant cations in plant tissues. This presence may be due to the use of fertilizers in sugarcane production [18]. Sodium and potassium oxides, when present at high concentrations in a cementitious matrix, can react with certain reactive forms of silica leading to material degradation, such as alkali-aggregate reactions [19][20]. However, some authors have already demonstrated that sugarcane bagasse ash with a high K_2O content (9 % by mass) can be utilized as a mineral additive in cementitious materials [21]. The loss on ignition (LOI) values indicate that all calcination temperatures adopted are sufficient to remove organic matter and volatile compounds that can have a detrimental effect on the durability of cementitious minerals [22][23].

Figure 1: Sugar cane bagasse

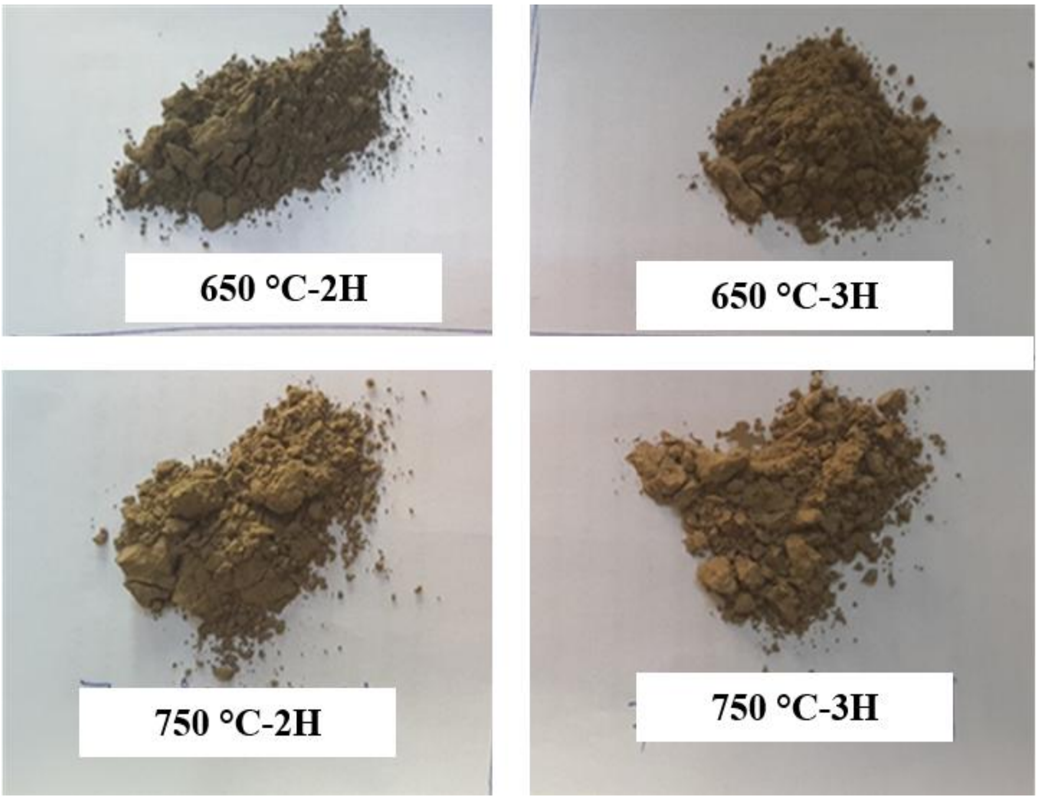


Figure 2: Sugarcane bagasse ash

Table 1: Chemical composition of ash for 2 hours of leveling

Oxides (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	PF
Temperature (°C)								
600	64.14	6.82	5.12	1.78	1.75	6.32	0.22	5.83
650	64.00	8.16	5.04	1.80	1.90	6.55	0.25	3.90
700	63.86	9.50	4.96	1.81	2.04	6.78	0.27	3.22
750	66.32	12.97	5.27	1.35	1.60	4.70	0.36	3.12

Table 2: Chemical composition of ash for 3 hours of leveling

Oxides (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	PF
Temperature (°C)								
600	69.79	9.86	3.02	1.92	1.78	5.57	0.05	5.61
650	72.50	10.63	4.04	1.14	1.34	3.56	0.08	3.57
700	74.22	9.87	4.13	1.72	0.96	2.70	0.03	3.13
750	75.91	10.49	2.99	0.84	1.31	3.30	0.07	2.46

3. Experimental methods

3.1. Physicochemical and mineralogical characterization of the cement used

The elemental chemical composition of the cement was determined by X-ray fluorescence (XRF). The mineral phases of the cement and formulated mortars were identified by combining X-ray diffraction (XRD) and thermogravimetric analysis/derivative thermogravimetry (TGA/DTG). The diffractograms were recorded with a Bruker AXS diffractometer using CuKα radiation and a rear graphite monochromator. The TGA curves were recorded with a Linseis device operating in an air atmosphere with a heating rate of 10 °C/min. The DTG curves derived from the TGA curves in relation to temperature were deduced from these TGA curves.

The absolute density of ashes was measured using a turpentine pycnometer. The specific surface area was measured using a Controlab Vision 1090 permeameter. The microstructure of the ash and mortars produced was observed using a JEOL 6380 LV scanning electron microscope equipped with a backscattered electron detector.

3.2. Microstructural characterization and pozzolanic activity index of ash

Pozzolanic activity index (PAI) is a parameter that quantitatively describes the degree of reaction over time or the reaction rate between a pozzolanic material and $Ca(OH)_2$ in the presence of water [24]. It is obtained according to **Equation 1** as the ratio between the 28-day compressive strengths of mortar containing 25 % mineral admixture and that of the control mortar (C) without mineral admixture.

$$\text{PAI} = \frac{R_{C_{28}}}{R_{C_{28}}(C)} \qquad \textbf{Equation 1}$$

Thus, according to ASTM C618, a material with a minimum pozzolanic activity index of 0.75 at 28 days can be used as a pozzolan.

To determine this, mortars and mechanical tests were conducted in accordance with Standard NF-P-15-403.

3.3. Influence of ash on the density and stability of test specimens

The apparent density of the mortar specimens (ρd) was determined using **Equation 2** in accordance with the standard (NF EN 18-459) [25]:

$$\rho_d = \frac{M_{dry}}{M_{air} - M_{water}} \times \rho \qquad \textbf{Equation 2}$$

Mdry: specimen dry mass in air. Mwater: Mass of the water-saturated sample obtained by hydrostatic weighing, and Mair: Mass of the water-saturated sample weighed in air.

The stability test is carried out in accordance with standard EN196-3 [26]. The procedure involves creating a paste of standardized consistency and placing it in the Chatelier mold. After filling, the mold is kept for 24 hours in a room with 95 ± 5% humidity and approximately 20 °C. After this time, the distance A between the tips of the needles is measured to the nearest 0.5 mm. The mold is then placed in a water bath at 20 °C. which is brought to a boil for 3 hours ± 5 minutes. Let B be the distance between the tips of the needles when the mold, after cooling, has returned to a temperature of 20 °C. Stability S is characterized by the difference between B and A given by **Equation 3** and expressed in mm to the nearest 0.5 mm.

$$S(mm) = B - A \qquad \textbf{Equation 3}$$

3.4. Preparation of specimens

The test specimens used were made in accordance with Standard NF EN 197-1 in prismatic molds measuring 40 x 40 x 160 mm^3. They were mechanically compacted in two layers using an impact table with 60 blows at a drop height of 15 mm. The molds containing the test specimens were kept at 20 ± 1 °C and approximately 55 ± 5% relative humidity. Demolding is performed after 24 hours, and the specimens are kept in water at 20 ± 1 °C until the test period (7, 14, 28 and 56 days). Each formulation yielded a series of test specimens, which were used for mechanical testing and mineralogical analysis by XRD, ATD/TG, and microstructural analysis by SEM.

3.5. Durability testing of mortars in aggressive environments

For the durability tests in an acidic environment, test specimens measuring 50 x 50 x 50 mm^3 were prepared in accordance with the procedures set out in standard NF EN196-1. The mixing procedure was that recommended in standard NF P15-403. The formulated test specimens were kept in water at 20 ± 2 °C for 28 days before removal from their molds. They were weighed before and after being placed in the various acid solutions (sulfuric and hydrochloric). Moreover, the microstructure of the specimen after immersion was observed using SEM/EDS.

4. Results and discussion

4.1. Physicochemical and mineralogical characterization of cement

The cement used in the mortar formulation is a CEM II 42.5/B-L Portland cement manufactured by Heidelberg CIMBURKINA. Its elemental chemical composition (Table 3) indicates that it is primarily composed of calcium oxide and silica (61.08 % CaO and 21.94 % SiO_2), as well as a non-negligeable amount of alumina and iron oxide (5.68 % Al_2O_3 and 3.18 % Fe_2O_3). The sulfate content of 2.53 % is consistent with that typically found for this category of cement. Based on these results, the overall chemical composition of the cement is typical of a Portland cement and complies with the applicable standards NF EN 196-1 [27] and NF P 15-317 [28]. The alkali equivalent (% Na_2Oeq = Na_2O + 0.658 x % K_2O) of less than 0.6 indicates a good alkali content in the clinker, which is likely to prevent alkali-aggregate reactions. The CaO/SiO_2 ratio = 2.16 is higher than the value of 2 required by standard EN 196-2 [29]. The sum of CaO and SiO_2 exceeds 50 %, the minimum required by the standard NF EN 197-1. The cement could therefore be used to formulate test specimens.

The diffractogram of anhydrous cement (Figure 3) revealed the presence of minerals such as tricalcium silicate (C_3S), known as alite, dicalcium silicate or belite (C_2S), tricalcium aluminate C_3A or celite, and tetracalcium ferroaluminate or ferrite (C_4AF). These mineral phases are responsible for the setting and hardening of cement during hydration. Calcite ($CaCO_3$) and gypsum ($CaSO_4 \cdot 2H_2O$), which regulate setting, are also present. The results of the cement mineralogy are consistent with those in the literature [29]. The mineralogical analysis and some physicochemical parameters of the cement are shown in Table 4.

Table 3: Chemical composition of CEMII/42.5 B-L cement

Oxides	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**SO_3**	**K_2O**	**Na_2O**	**Total**
Contents (%)	21.94	5.68	3.18	61.08	1.46	2.53	0.56	0.35	96.78

Table 4: Mineralogical composition and some physicochemical parameters of cement

Minerals	**C_3S**	**C_2S**	**C_3A**	**C_4AF**	
Composition (%)	46.24	20.76	8.17	9.13	
Physicochemical parameters	Specific Surface Area Blaine (cm^2/g)	Density	Initial setting (min)	End setting (min)	Expansion (mm)
CEM II 42.5/B-L	4405	3.2	192	285	0.5

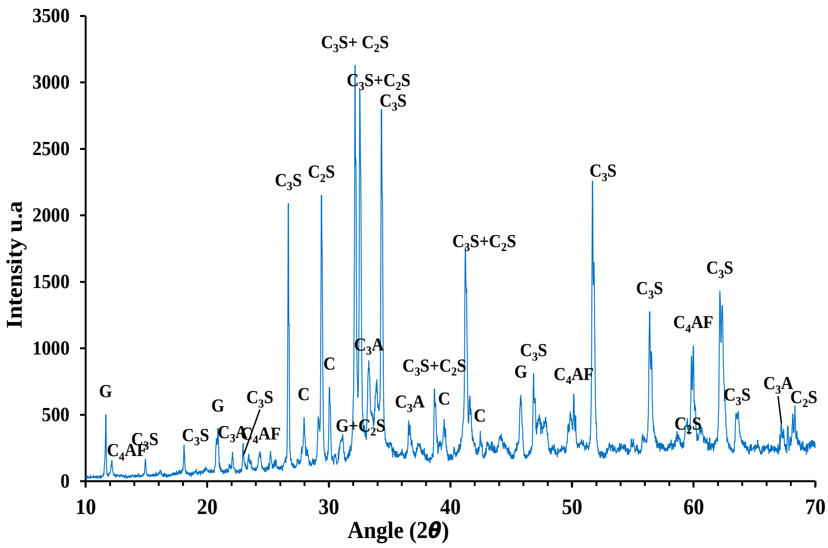


Figure 3: Diffractogram of cement powder

C_3S: Alite; C_2S: Belite;(C_3A): Celite;(C_4AF): Ferrite; C: Calcite ($CaCO_3$); G: gypsum ($CaSO_4.2H_2O$)

4.2. Microstructure and pozzolanic activity index of ash

Figure 4 below shows SEM images of CBCS obtained at 650 °C and 750 °C with dwell times of 2 and 3 hours, respectively.

SEM images show that the ash has irregularly shaped grains and intrinsic porosity. These particles are elongated and angular. At a temperature of 650°C, there is a significant particle agglomeration, indicating an amorphous phase. The large platelets are attributable to crystalline phases. The ash produced at 750 °C exhibits more platelet structures, which can be attributed to the recrystallization of the abundant amorphous silica originally present in the material. The increased porosity could explain the low density of the ash, indicating greater water absorption during mixing. The difference in morphology between CBCS 650°C and CBCS 750°C shows that particle size increases significantly during calcination [30].

Figures 5.a) and b) show the pozzolanic activity indices of the ash produced after 2 and 3 hours of stabilization, respectively.

According to these results, for over 2 hours, most of the index values exceed 75 %, except for the ash mixture at 750 °C. For mixtures with ash obtained at 3 hours, only the pozzolanic activity indices of ash obtained at 700 °C and 750 °C are below the minimum requirement of 75 %. Regardless of the calcining time and temperature used for ash production, the pozzolanic activity indices are consistently below 100 %. This could be explained by the slowness of the pozzolanic reaction. In addition, as the percentage (25 %) of cement replaced by ash is high, it takes longer for the strength of the mortars to reach that of mortars without additives[31][32][33]. Table shows the percentages of amorphous phase.

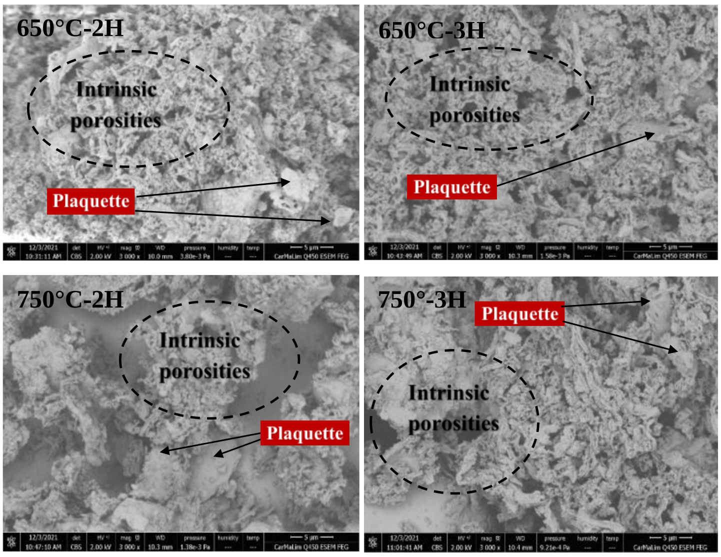


Figure 4: SEM images of ashes obtained at 650 °C and 750 °C for 2 hours and 3 hours exposure periods

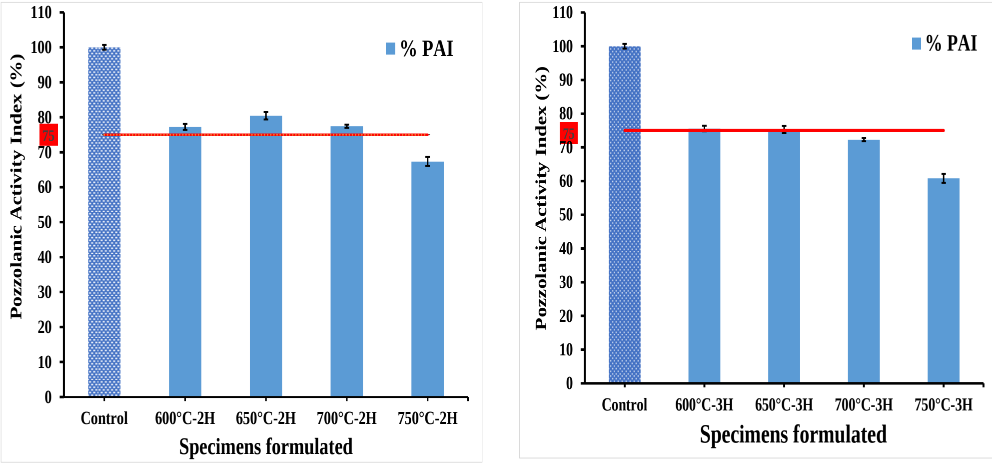


Figure 5: CBCS pozzolanic activity indices at 28 days: **a)** for 2 hours and **b)** for 3 hours

Table 5: Percentages of amorphous phase contained in ash (2 hours stages)

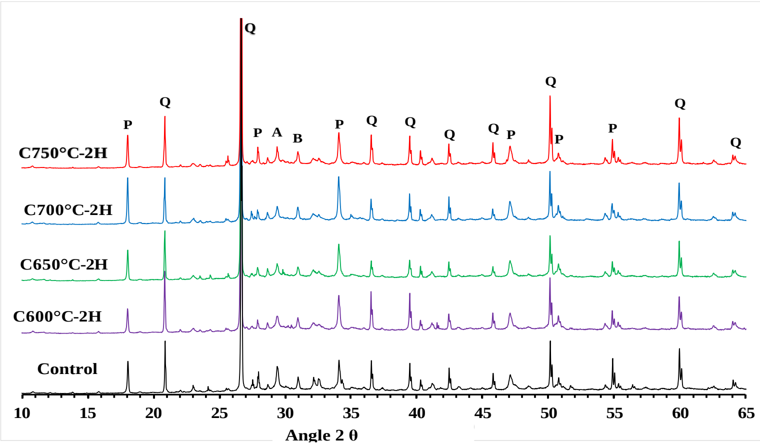
Temperature (°C)	600	650	700	750
% amorphous phase	74,69	82,91	88,50	69,85

The amount of amorphous phase increases with calcination temperature and drops sharply above 700°C. It can be seen that the amorphous phase content varies depending on the calcination temperature. The results obtained could show that at high temperatures, recrystallized phases may appear, thereby reducing the amorphous nature of the calcined materials. Generally, the index decreases after 650 °C due to the reduction in the amorphous phase resulting from the recrystallization of part of the material as the temperature increases. This effect is more pronounced with a longer calcination time. [11]

4.3. Influence of ash on the mineralogy and microstructure of mortars

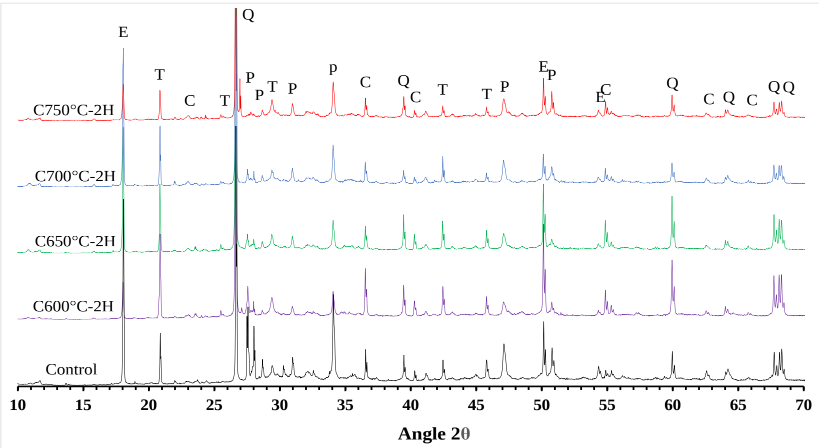
The mineralogical evolution of the cement paste upon ash addition is monitored by X-ray diffraction and thermal analysis. The ash obtained with 2 H will be used exclusively for mineralogical monitoring, as it has shown a higher pozzolanic activity index and is also more cost-effective due to lower calcination energy requirements.

The diffraction results for the mixtures at 14 and 28 days are shown in Figures 6 and 7.



E : Ettringite ; C : Calcite ; P : Portlandite ; Q : Quartz ; T : Tobermorite (CSH)

Figure 6: Diffractograms of mortars with 10 % CBCS for 14 days



E : Ettringite ; C : Calcite ; P : Portlandite ; Q : Quartz ; T : Tobermorite (CSH)

Figure 7: Diffractograms of mortars with 10 % CBCS for 28 days

The different mixtures have similar overall mineralogical compositions, with the main phases being portlandite (CH), calcite (C), ettringite (AFt), and tobermorite (CSH).

Analysis of the diffractograms of the young specimens reveals the presence of portlandite, quartz, and silicate anhydrides (alite, belite) [34]. The intensities of the peaks relating to portlandite in the diffractograms of the mixtures containing ash are not very different from the peaks in the control sample, highlighting the slow reaction of portlandite with ash [35]. As for quartz, its presence in cement-ash mixtures is attributable to non-reactive quartz [36]. Ettringite ($C_6A\bar{S}_3H_{32}$) is detected in both cement-ash mixtures and the control cement.

For the test specimens formulated at 28 days, there are significant variations in the intensity of the hydrate peaks. The intensity of the portlandite and ettringite peaks decreases in mortars modified with CBCS. However, the intensity of the tobermorite (or CSH gel) peak increases in the modified mortars. This hydrate is responsible for the internal structure of the cement paste, the bond between the paste and the aggregates in mortars, and ultimately the mechanical strength of the resulting material [37]; it is therefore an essential component [38].

The gradual increase in the intensity of the CSH peaks and the gradual decrease in the CH peaks indicate the pozzolanic reaction. This result corroborates the fact that the portlandite (CH) produced during cement hydration reacts with the amorphous silica and alumina provided by the CBCS to form additional CSH [39].

TG/DTA was performed on mortars that had been cured for 28 days to supplement the results obtained by XRD. The results are shown in Figures 8 and 9.

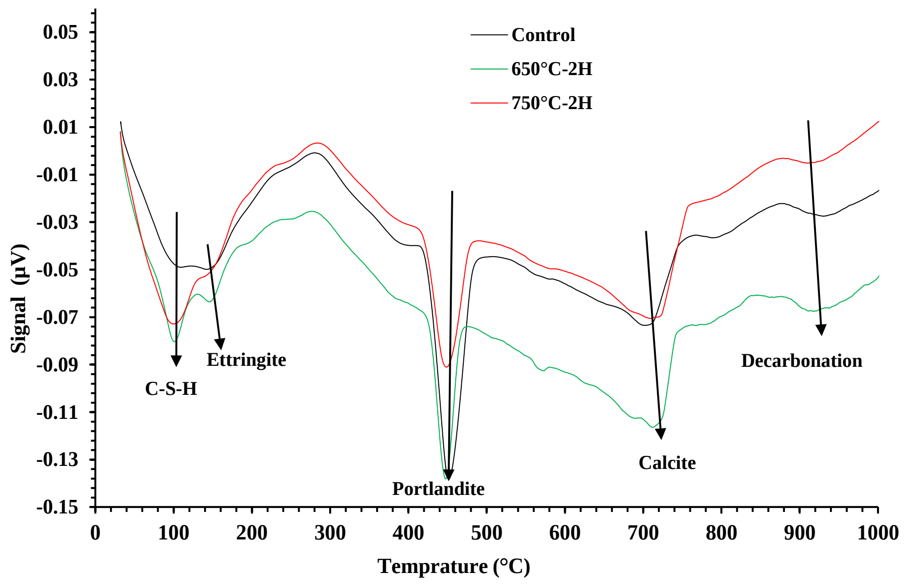


Figure 8: ATD thermograms of formulated mortars

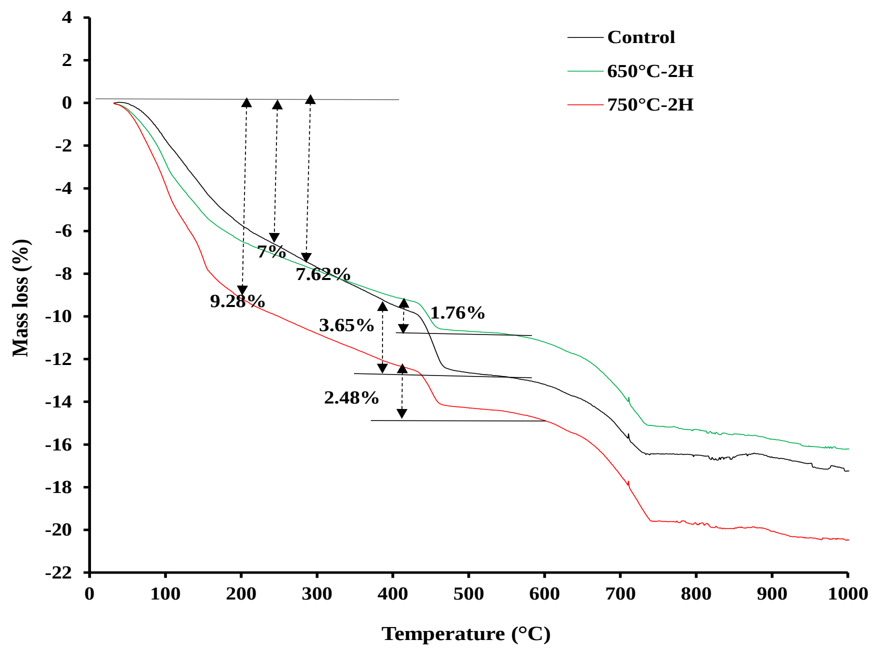


Figure 9: ATG thermogram of formulated mortars

Generally, the same behavior was observed across all samples. Endothermic peaks were observed at approximately 104 °C and 150 °C, attributable to the dehydration of ettringite, calcium silicate hydrates (CSH), and phases associated with the hydration of aluminates [40]. The various mass losses related to the different transformations are summarized in Table 5.

The mass losses associated with these phenomena are 9.28 %, 7.62 %, and 7 % for the 650 °C and 750 °C ashes and the control cement, respectively. A significant mass loss was observed between 400 and 500 °C for all mortars, due to the dehydroxylation of portlandite. The various losses show reductions in portlandite of 51.73 % and 32.02 %, respectively, with the addition of C650 and C750 °C. This significant reduction, achieved with only a 10% substitution of cement with ash, demonstrates the ash's good pozzolanic reactivity. At a temperature of 573 °C, a peak is observed, indicating the allotropic transformation of α-quartz into β-quartz, accompanied by a phenomenon of expansion.

Decarbonation of calcium carbonate ($CaCO_3$) occurred in all samples around 718 °C [41]. Changes between 600 and 750 °C may also be linked to the decomposition of CSH, which transforms into a new form of bicalcite silicates (β-C_2S). The pozzolanic reaction of CBCS reduced the amount of $Ca(OH)_2$, thereby decreasing mass loss [42]. According to Kandasamy et al. [37], a reduction in $Ca(OH)_2$ content was observed due to the pozzolanic reaction. Wongkeo et al [43] reported a decrease in CH content in ash-modified mortars compared to the control mixture due to the pozzolanic reaction, which consumes the CH content and transforms it into CSH gel.

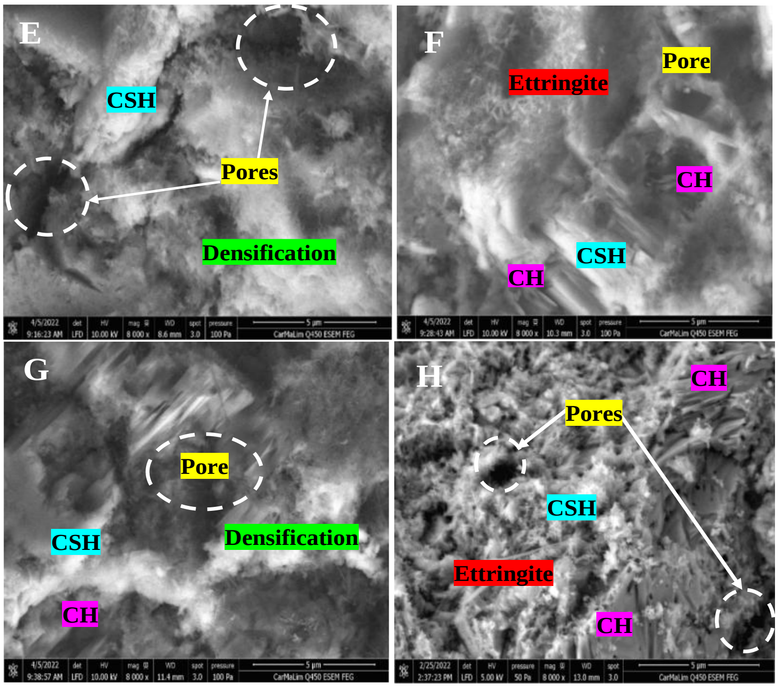
Table 6: Observed mass losses of the formulated test specimens

	Pics	450 °C	104 °C et 150 °C	718 °C
Attribution	Phases	CH	CSH et Ettringite	Calcite
Mass losses	Control	3.65	9.28	4.03
	C 650-2H	1.76	7.62	4.47
	C 750-2H	2.48	7	5.50

The microstructure of the control mortars and those obtained with 10 % cement replacement by ash was observed using a scanning electron microscope (SEM). The various images obtained are shown in Figure 10.

Analysis of SEM images enables the identification of hydration products, including CSH gels, portlandite (CH), and Aft crystals, as well as the presence of a few pores. The microstructure of the test specimens shows increased densification with the addition of ash. The ash reacted with the precipitated portlandite, resulting in CSH with a significantly modified appearance and Aft crystals. The CSH gel appears dense, composed of amorphous agglomerations. The CSH gel develops in the capillary spaces and further densifies the ash-modified mortars[36]. The aluminate products appear as fine plates or needles, mainly deposited inside the pores. In the SEM images, hydration products, especially calcium aluminosilicate hydrate plates [34], are visible.

In summary, the SEM images indicate that the addition of sugarcane bagasse ash promotes gradual densification of the cement matrix. This densification results not only from the filler effect of the fine particles, but also from the gradual precipitation of pozzolanic reaction products, which fill the capillary pores. The porous network thus becomes more closed, potentially reducing the material's permeability to aggressive agents [44]. This development may be associated with improved long-term mechanical performance and greater resistance to chemical attack. Cross-analysis of mineralogical, microstructural, and mechanical results confirms the benefits of this mineral addition.



E: C650 °C-2H (10 %); F: C650 °C-3H (10 %); G: C750 °C-2H (10 %); H: Control

Figure 10: SEM images of test specimens formulated with 10 % CBCS

4.4. Physical properties of cement mortars and pastes

4.4.1. Bulk density

Density is a crucial factor in evaluating various properties of mortar or concrete, including porosity, strength, and durability. The apparent density of mortars **(Figure 11)** was measured according to the standard [NF P 94-054] [45].

The standard NF P 94-054 specifies a cement density of 2,05-2,14. All density values for the different cement formulations are slightly below the specified range. This would result in low CBCS density. In addition, the modified mortars have lower densities than the control due to the effect of replacing cement with ash. The ash has a lower density than cement. The substitution, therefore, results in a decrease in the density of the mortars compared to the control sample. This density decreases with the rate of cement substitution by ash and further decreases as the ash production temperature increases.

4.4.2. Setting time

The initial and final setting times of the cements were determined in accordance with standard EN 196-3. The results are shown in Figure 12.

The results show variations in the setting times of the formulated pastes with varying ash percentages. Mixtures containing pozzolanic ash show increases in initial and final setting times compared to the control paste. For initial setting times, pastes with 10 % and 25 % ash show increases of 14.28 % and 21.42 %, respectively, compared to the control paste. The same behavior is observed for the end-setting times, with increases of 15 % and 20 %, respectively, for the pastes with 10% and 25% ashes compared to the control paste.

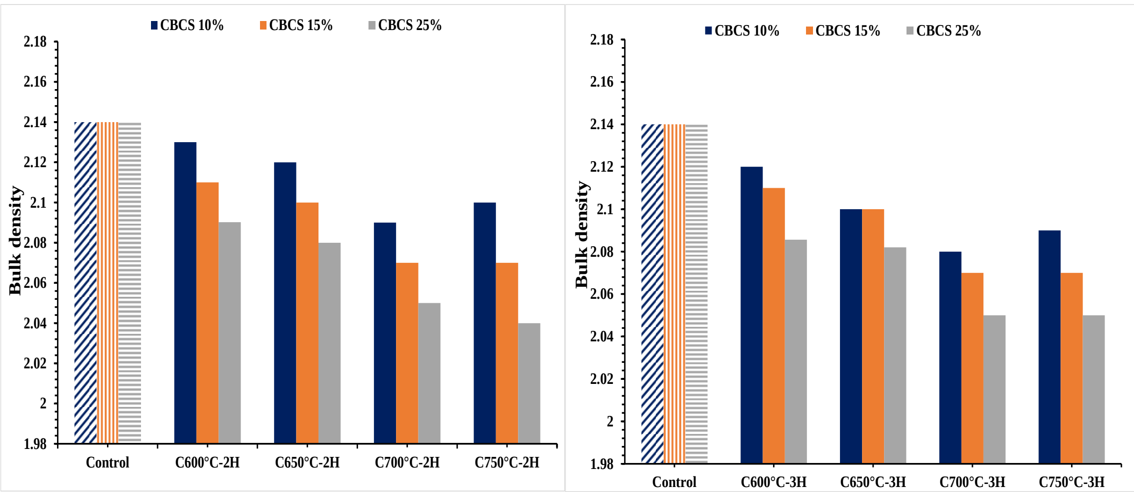


Figure 11: Evolution of the density of formulations produced at 2 hours and 3 hours

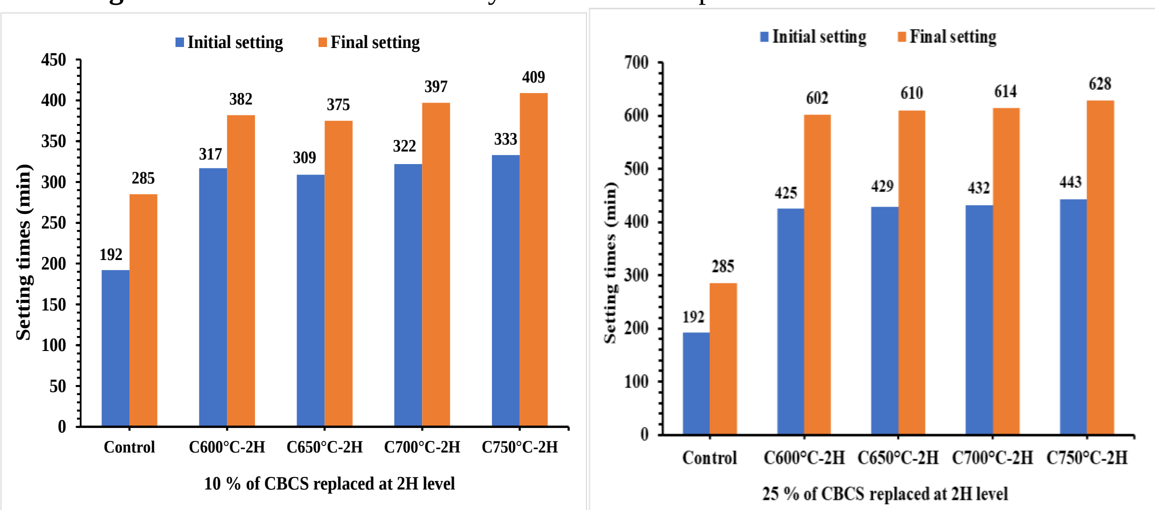


Figure 12: Setting time of pastes formulated with 10 % and 25 % CBCS

A minimal delay of approximately one hour is observed for pastes with CBCS additions in both the initial and final setting times. The setting time increases with the percentage of ash incorporated. Setting time is an essential phenomenon for hydraulic binders. Generally, it depends on several parameters, such as chemical composition, particle fineness, mixing time, dissolution rates of Si and Al, and processing temperature [46][47]. From the setting time results (Figure 12), it can be noted that the initial setting times of the cements comply with standard EN 196-3, which requires an initial setting time for type 42.5 MPa cements to be greater than or equal to 75 minutes [26]. The increase in the setting time of ash-modified pastes can be explained by the rise in water demand [48][49]. The delay in setting is mainly due to a decrease in the cement content (C3S), which is responsible for early stiffening.

Furthermore, the degree of hydration is controlled by the density and thickness of the calcium silicate hydrate (CSH) layer formed around the cement grains; the presence of ash in this layer can only delay the hydration of the mixture and, consequently, the start and end of setting [50][51]. These results are consistent with studies by Dyer et al., which demonstrate that ash significantly affects setting times and, consequently, the hydration process [52]. According to Provis et al. [53], setting time also increases with the SiO_2/Al_2O_3 ratio in aluminosilicate materials. For Diaz et al. [54], aluminosilicate compounds with an amorphous phase dissolve more easily than those with predominantly crystallized phases. Thus, the presence of amorphous silica in ash would delay setting [55][56].

4.4.3. Expansion of formulated cement pastes

The results of the expansion of mixtures incorporating different proportions of ash are presented in Table 7.

Table 7: Expansion of the pastes

Specimens / Level	Expansion (mm) 2 h	3 h
Control	0.50	0.50
600 °C	0.80	0.80
650 °C	0.80	0.80
700 °C	0.70	0.75
750 °C	0.60	0.70

The results show slightly greater expansion than the control. All expansions remain below 10 mm. This indicates that these formulated cements are highly stable, which can be attributed to their low free lime content, less than 2% according to NF EN 196-2. The results of the various expansions are compared with those required by the standard EN 196-3, with values less than 10 mm.

4.5. Durability of mortars in aggressive environments

External factors, such as exposure to aggressive agents or climatic conditions, can cause deterioration in cementitious mortars or concrete. Permeable cementitious materials are therefore vulnerable to external attacks [57]. The use of pozzolanic compounds can improve the durability of these building materials. A precise understanding of their effects is essential for the optimal use of these pozzolanic materials. This acid attack resistance test measured the mass loss of test specimens. Acid solutions (sulfuric and hydrochloric) were preferred because they are the primary acids found in acid rain.

Additionally, inorganic acids are more detrimental to concrete and mortar than organic acids [58]. Only the C650 °C-2H test specimens were used for the acid attack resistance tests due to their superior mechanical strength [11]. **Figure 13** illustrates the variations in mass loss of mortars with and without ash at different percentages, depending on the immersion period (7, 14, 28, and 56 days) in 5% HCl and 5% H_2SO_4 solutions.

Firstly, it was observed that all mortars, with and without ash added, without exception, showed permanent mass losses at all periods considered. By comparing the mass losses, it was found that mortars amended with 20 % ash showed the lowest mass losses. The pozzolanic reaction fixes the portlandite, reducing capillary pores by forming second-generation CSH gels that block the absorption of the acid solution, resulting in lower mass loss for all ash mortars compared to the reference mortar [59]. In addition, the mass loss observed for all amended specimens is due to the presence of free portlandite, which can be leached when subjected to sulfuric acid attack to form gypsum [60]. These changes in mass loss are consistent with the portlandite levels determined by XRD and ATD-TG analysis. The addition of ash significantly reduces the available portlandite and improves mortars' resistance to acid attack. As the attack continues, all cement components are subsequently decomposed and leached. Additionally, the calcium sulfate formed by the first reaction reacts with the calcium aluminate phase in the cement to form hydrated calcium sulfoaluminate (ettringite), which, after crystallization, can cause the mortar to expand. The precipitated gypsum layer is easily leached, resulting in considerable mass loss [61][62].

SEM-EDS images were used to explore the microstructure of mortar samples immersed in hydrochloric acid (Figure 14) and sulfuric acid (Figure 15).

It can be seen that a large number of hydration products, including CSH gel, Aft crystals, and several pores, formed on the surfaces of the formulated specimens. The modified mortars exhibit a dense hydration product with few cracks. The use of ash as a partial substitute result in changes in the microstructural development of the mortars due to pozzolanic reactivity. It has been found that the addition of mineral materials improves the chemical resistance of mortars when exposed to acidic environments, as demonstrated by several studies [63][64][65].

The comparison of the SEM images (Figures 14A, C, and E) clearly shows the effect of the additions on the microstructure of the mortars. Between images A and C, a pronounced densification of the mortar is observed, even in an acidic environment. The same result is observed when comparing the modified mortars with the control (E). Increased porosity is evident in the control sample, confirming the mass-loss results reported above. The same observations are made with SEM images of the mortars after exposure to sulfuric acid. Sample F shows a leached material with significant porosity. Unlike the control sample, the two mortars with added ash still appear dense.

SEM-EDS analysis of samples treated with hydrochloric acid shows significant deterioration of the cement matrix characterized by considerable calcium dissolution and a notable decrease in the Ca/Si ratio. The modified surface is characterized by a substantial reduction in calcium concentration to less than 40 %, offset by a relative increase in silica and alumina from bagasse ash. At the same time, the presence of sulfur (S) remains low, contrary to what would be observed in the case of sulfate attack [66]. EDS analysis reveals a significant concentration of chlorine (10-13 %) at the surface, indicating the incorporation of chloride ions and the potential formation of soluble calcium salts ($CaCl_2$), which are readily leachable. Other chemical components, such as Ca, Si, and Al, are also present, with clearly established concentrations. Ca contents range from 27 to 37 %, Si contents range from 2.6 to 13 % and Al contents range from 1.5 to 5.1 %. These contents are similar to those of a conventional mortar enriched with pozzolan [67][68]. Thus, the presence of Cl correlates with the loss of Ca, confirming the destructive role of HCl, which causes the decalcification of CSH and the collapse of the microstructure [66].

Spot SEM-EDS analyses of mortars subjected to sulfuric acid attack reveal the predominance of oxygen (O), calcium (Ca), and silicon (Si), as well as varying amounts of aluminum (Al), iron (Fe), and alkalis (K). On surfaces treated with H_2SO_4, the available Ca decreases due to CSH decalcification. Bagasse ash can introduce significant amounts of amorphous carbon and silica, which probably increase the Si/Al ratio [69]. Mg is also present and can be detected depending on the composition of the raw materials. These proportions provide compelling evidence for the formation of ettringite and the degree of decalcification [67].

SEM-EDS analyses have demonstrated that exposure of cement mortar modified with bagasse ash to sulfuric and hydrochloric acids results in significant chemical and microstructural degradation, occurring through distinct mechanisms. Under the action of sulfuric acid, the microstructure deteriorates, leading to the formation of gypsum and ettringite, and to an increase in sulfur and a reduction in calcium, reflecting the progressive decalcification of CSH [66]. In contrast, hydrochloric acid primarily causes the intense dissolution of calcium phases, a rapid decrease in the Ca/Si ratio, and significant leaching of constituents, without significant precipitation of secondary sulfates. In both cases, the role of sugarcane bagasse ash appears ambivalent: its pozzolanic activity locally densifies the matrix and limits the formation of free portlandite; however, its porosity and combustion quality strongly influence acid penetration and, therefore, the durability of the material [66].

5. Conclusion

The main objective of this study was to investigate the effect of sugarcane bagasse ash (CBCS) on the physicochemical properties and durability of cement mortars in aggressive environments. The results obtained led to the following conclusions:

The chemical composition of CBCS showed that it contains silica (58.37 to 75.91 %), alumina (6.82 to 12.97 %), iron oxide (2.99 to 5.27 %), less than 10 % alkali oxides, and less than 6 % alkaline earth oxides when the calcination temperature increases from 600 to 750°C.

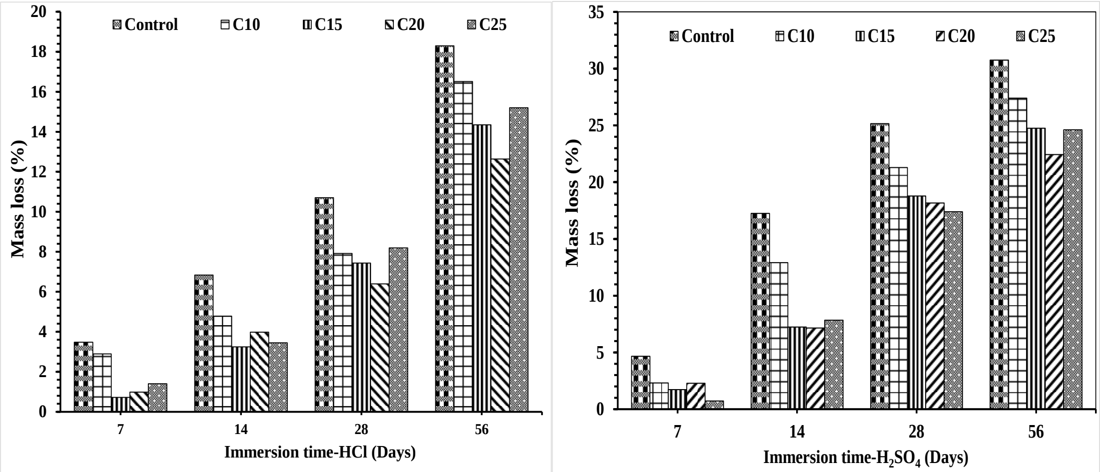
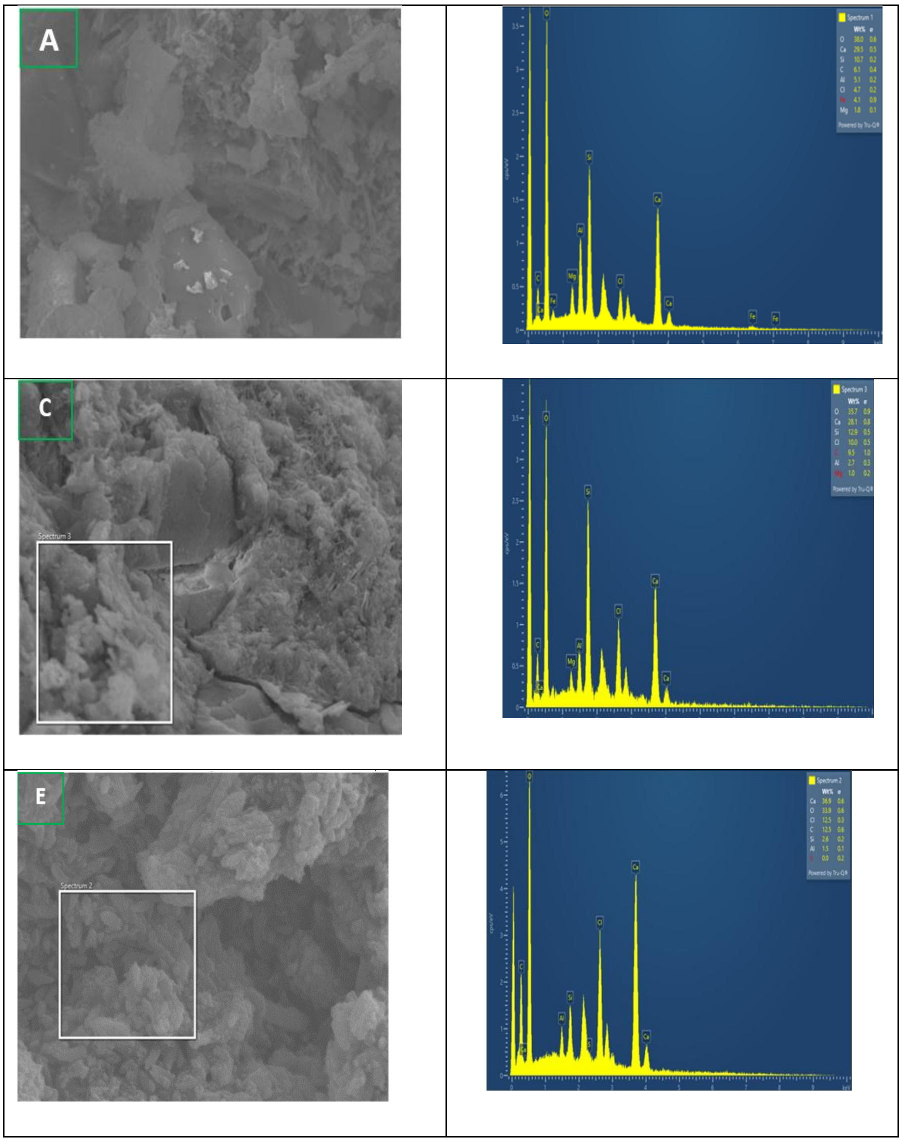
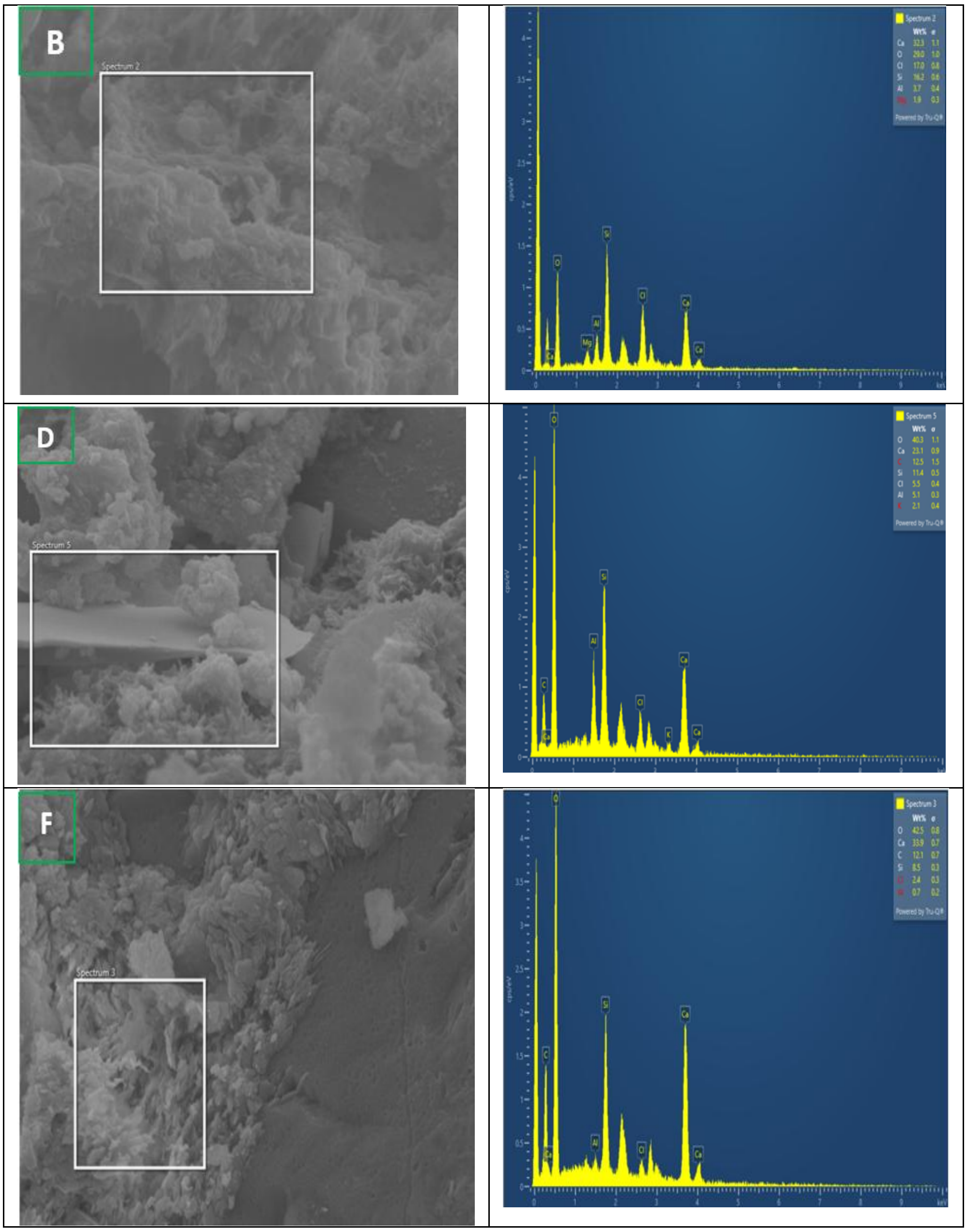


Figure 13: Mass loss of mortars immersed in 5 % HCl and 5 % H_2SO_4 solutions



A: CBCS10%-HCl. C: CBCS 20%-HCl . E: Témoin-HCl

Figure 14 : SEM-EDS images of mortars subjected to acid attacks



B: CBCS10 %-H_2SO_4 D: CBCS 20 %- H_2SO_4 F: Témoin- H_2SO_4

Figure 15 : SEM-EDS images of mortars subjected to acid attacks

This composition imparts pozzolanic properties to sugarcane bagasse ash (CBCS), making it suitable as a pozzolanic material.

The evaluation of the pozzolanicity of CBCS using the pozzolanic activity index yielded values above 75%, except for those obtained at 750 °C. All these characteristics confirm that CBCS calcined between 600 and 700 °C, are materials suitable for the formulation of pozzolanic cements.

The density of the mortars ranges from 2.04 to 2,14 which complies with NF P 94-054. The setting and final setting times of the cementitious pastes range from 65 to 185 minutes, which comply with the standard EN 196-3.

Cement mortars exposed to hydrochloric and sulfuric acid (5 %) experience mass loss, which depends on the exposure time. Thus, mass loss decreases with increasing bagasse ash content. The resistance of ash-modified mortars to acidic environments is thought to be due to the formation of CSH, which fills the capillary pores, thus limiting the penetration of acid solutions.

Despite improved acid resistance in mortars, certain durability aspects still require further exploration. They will be the subject of future work, namely the study of carbonation, gas permeability, efflorescence, and the electrical resistivity of mortars modified with sugarcane bagasse ash.

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