

Article

Development of Flash-Calcined Sediment and Blast Furnace Slag Ternary Binders

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Abstract: Partial cement replacement by low-carbon-impact additions has the potential to reduce CO₂ emissions. The aim of this study is the development of a ternary binder that includes ordinary Portland cement (OPC), ground granulated blast furnace slag (GGBS), and flash-calcined sediment (FCS). To upgrade dredged mineral material into FCS, a new heat treatment, i.e., flash calcination, was used. The used materials were physically, chemically, and mineralogically characterized. The mixture design method was used to optimize the design of the ternary blended binders. A model was developed and validated for the prediction of the 90-day compressive strength for mortars composed of OPC (C), GGBS (S), and FCS (F). Five mixes, reference RM (100% OPC), binary mix (50% OPC and 50% GGBS), and three ternary mixes with FCS rates of 10%, 15%, and 20% were characterized in fresh and hardened states. The results show that the incorporation of FCS reduced the workability of the mixes and increased their densities. Moreover, the initial setting time of the mix was delayed, and the heat of the hydration peak was decreased. The 90-day compressive strengths of the mix containing 10% FCS were higher than those of RM. In conclusion, the use of 10% FCS and 40% GGBS was an efficient substitute for 50% OPC.

Keywords: sediments; circular economy; cement; ternary eco-binders; flash calcination method; mixture design



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1. Introduction

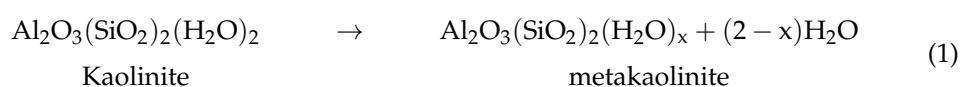
The alarming climate change in recent decades is primarily due to global warming, which is linked to an increase in greenhouse gas (CO₂) emissions. Cement production contributes heavily to CO₂ emissions. It is estimated that 5 to 8% of world CO₂ emissions are due to cement production [1–4]. The tools and strategies to meet the environmental challenges should involve the substitution of ordinary Portland cement (OPC) [5].

To achieve this goal, the use of mineral additions, particularly those derived from industrial byproducts such as ground granulated blast furnace slag (GGBS), as partial substitutes for cement (i.e., supplementary cementitious materials, SCMs) is an interesting alternative. In addition to reducing the environmental impact of cement, SCMs enhance the mechanical properties and the durability of concretes [6,7]. Other types of waste such as dredged sediments can also be used after undergoing adequate treatment.

Dredging operations generate large amounts of sediments every year. For example, the volume of dredged sediments generated in France, Europe, and in the USA is about 50 Mm³, 300 Mm³, and 300 Mm³, respectively [8,9]. European Directive 2008/98/EC dated 19 November 2008 considers dredged sediments as waste, and their management is associated with major technical, economic, and environmental issues. Thus, the recycling of dredged sediments as supplementary cementitious materials represents an interesting alternative [10]. The potential of using dredged sediments as SCMs is strongly linked to their mineralogical composition (calcite, quartz, kaolinite, etc.) and their physical characteristics (granulometry, BET-specific surface area, etc.). The type and proportion of clay are an

important parameters for sediment activation. Heat treatment by calcination of dredged sediments is essential for the vaporization of their free water at a temperature of 100 °C, the elimination of their organic compounds at 300 °C, the activation of their clay minerals by the transformation of kaolin into metakaolin at 530 °C, and the decomposition of their calcium carbonates at around 730 °C. TGA analysis performed on dredged sediments showed that the calcination process induces a decarbonation of calcite at temperatures between 600 °C and 750 °C [9]. Moreover, the calcination process eliminates the volatile components and destroys the crystalline structure of gangue minerals.

Moreover, the calcination of dredged sediments leads to the dihydroxylation of the crystallized and non-reactive kaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) and the evaporation of moisture, resulting in metakaolinite, an amorphous and reactive aluminosilicate ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ or AS_2) according to Equation (1) [11]. Thus, the reactivity of the sediments in the cement matrix depends on their calcination temperature and duration.



Sediment can be calcined by two different methods: conventional calcination or flash calcination. The latter is a relatively new calcination technique. It was initially developed for clays (kaolin) [12–14]. Flash calcination involves heating the material for about 1/10 s at a temperature of 700 °C to 1000 °C before rapid cooling. Thanks to this partial destruction, new amorphous phases with potential activation of the sediment are formed. This method has several advantages compared to the conventional calcination method, namely a quick calcination process, better temperature control, decreased energy consumption, and increased activation potential [15].

The pozzolanic activity of calcined sediments using the conventional method has been extensively studied in the literature [16–22]. Numerous authors have reported that binary binders containing 8% calcined sediments improved the hydration cement-sediment blends and resulted in mechanical strengths equivalent to those obtained with a CEM I Portland cement (e.g., see Dang et al. [19] and Diouri et al. [20]). This is generally associated with portlandite consumption [21]. Additionally, an improvement in concrete resistance to sulfuric acid attack was reported by Safer et al. [22] when 10%, 20%, and 30% of cement was substituted with sediments calcined at 750 °C. This is because the calcined sediments led to the formation of supplementary C-S-H, which in turn reduced the concrete porosity, improved its durability, and increased its resistance to sulfate attacks and freezing and thawing [7,19].

When it comes to the use of flash-calcined sediment to substitute OPC, earlier studies dealt only with binary binders [23,24]. More recently, binary binders containing flash-calcined sediment used for 3D-printable mortar development [25]. The results showed that 10% FCD in printable mortar led to similar or higher compressive strength relative to that of the reference mortar. In addition, Amar [26] showed that sediments treated by flash calcination exhibited pozzolanic activity superior to those calcined in a traditional kiln when used in binary binders. Moreover, the energy consumed by flash calcination was about 2 GJ per ton of sediment [12,27]. On the other hand, 8 GJ of energy is needed for a ton of pure clinker [5,26], with an energy requirement of about 1.3 GJ per ton for GGBS [28]. Besides improving the mechanical properties of cementitious materials, flash-calcined sediments reduce the carbon footprint by limiting the quantity of clinker used [29,30]. The development of ternary binders containing flash-calcined sediment with GGBS and OPC has not been addressed in the literature. The objective of this contribution was to recycle dredged sediments to formulate a ternary eco-binder containing OPC, GGBS, and flash-calcined sediment.

2. Materials, Mixture Design, and Test Methods

2.1. Materials and Methods

2.1.1. Constituent Materials

The ternary binders were composed of OPC, GGBS, and FCS. The contents of clinker and secondary components in OPC (CEM I 52.5 N cement) accounted for more than 90% and 5%, respectively. The OPC used in this study, which was compliant with European standard NF EN 197-1, 2012 [31], was characterized by a low gypsum content. The compressive strength of a normalized mortar obtained with this type of cement is 52.5 MPa after 28 days according to European standard NF EN 196-1 [32]. Moreover, its setting time was under 240 min.

The GGBS used in this study was class A slag (Ecocem), compliant with NF EN 15167-1, 2006 [33], with a vitrification rate of more than 90% and a 20% lower energy consumption and 10% lower CO₂ emissions than OPC [34].

Marine dredged sediments from the Grand Port Maritime of Dunkerque (GPMD) harbor in Northern France were used in this study. After dredging, the sediments were transported to the laboratory, homogenized, and dried at 60 °C to decrease their water content.

2.1.2. Sediment Grinding and Flash Calcination Treatments

The treatment of sediments is a necessary step to reduce their heterogeneity in terms of organic matter content and to optimize their use as SCMs in a cementitious matrix. Sediment grinding yields fine particles to enhance the specific surface area and reactivity of the sediment [17,27]. The sediment pozzolanic potential also depends on the calcination temperature and duration, as the mineral phases, and especially clayey phases, are activated during this step [35–37]. The choice of an optimal calcination temperature is crucial because the reactivity of the calcined sediment is strongly linked to the treatment temperature. During calcination, the degradation of the clay minerals begins with the loss of the intralayer and adsorbed water between 100 °C and 250 °C. The dihydroxylation of the kaolinite begins between 300 °C and 400 °C and accelerates between 500 °C and 600 °C to generate metakaolin.

The effect of calcination on the physicochemical properties of sediments was studied by Benzerzour et al. [9], who showed that calcination induces a decrease in the BET surface area and an increase in the density. The abovementioned study reported a drop in the occurrence of crystalline phases such as calcite due to the decarbonation phase, whereas clay phases such as kaolinite must be transformed into reactive metakaolin.

A flash calcination treatment was chosen in this study.

The sediments used in this study were ground, sieved at 120 µm, and flash-calcined at a temperature of 820 °C [9].

2.1.3. Material Characterization Methods

All materials were characterized using physical and chemical methods. The particle size distribution was determined using a laser diffraction particle size analyzer (LS 13320, Beckman Coulter). The absolute density was measured according to NF EN 1097-7 (2008) [38] using a helium pycnometer (AccuPyc 1330, Micromeritics). The particle-specific surface area was assessed by measuring nitrogen adsorption using the BET method (Brunauer–Emmett–Teller) according to the NF EN ISO 18757 standard [39]. A water demand test was performed according to NF EN 196-3 (2017) [40] using a Vicat Vicatronic I06 091 apparatus equipped with a 10 mm diameter needle. Mineralogical characterization was carried out by X-ray diffraction (D2 Phaser diffractometer, Brucker Co., Ettlingen, Germany) using CuK α radiation ($K\alpha = 1.78 \text{ \AA}$) in a 2Θ acquisition range of 1–60° set at 40 kV and 40 mA.

2.1.4. Materials Properties

The physicochemical characteristics of the powders are summarized in Table 1. The particle size distribution of GGBS (Figure 1) shows D_{10} and D_{50} values closer to those

of the cement (Table 1). However, FCS shows a finer granulometry, with $D_{50} = 5.75 \mu\text{m}$, which is about half that of OPC. This fineness can be explained by the densification of the sediment grains during the heat treatment process [17]. Moreover, a change to a more rounded shape of the sediment particles is likely to occur due to cyclonic air movement during flash calcination [36,41]. These physical modifications concerning fineness and shape contribute to the improvement of the compactness and mechanical properties of binders containing FCS. The BET-specific surface area of FCS is more significant than those of OPC and GGBS, whereas the density of FCS is lower than those of OPC and GGBS. The reactivity of FCS, when used as an SCM, is enhanced with higher fineness and BET-specific surface area [42,43]. Moreover, the water demand of FCS is higher than that of OPC and GGBS. The increase in the fineness and BET-specific surface area leads to increased water demand [44,45].

Table 1. Physicochemical characteristics of OPC, GGBS, and FCS.

	Characteristic	OPC	GGBS	FCS
Physical properties	Density (g/cm^3)	3.21	2.91	2.64
	Surface area BET (cm^2/g)	9194	16,102	59,930
	Water demand (%)	32	33	53
	D_{10} (μm)	1.01	1.04	0.95
	D_{50} (μm)	10.7	9.82	5.75
Major oxides (wt%)	Al_2O_3	5.10	10.8	8.00
	CaO	60.9	40.7	21.6
	Fe_2O_3	4.00	0.53	9.00
	K_2O	0.72	0.46	1.88
	MgO	1.16	6.23	2.00
	MnO	-	0.20	0.15
	Na_2O	0.67	0.33	2.00
	P_2O_5	0.46	-	0.41
	SO_3	4.49	4.69	0.20
	SiO_2	16.3	31.7	52.8
	TiO_2	0.33	0.66	0.45
	ZnO	0.25	-	0.12

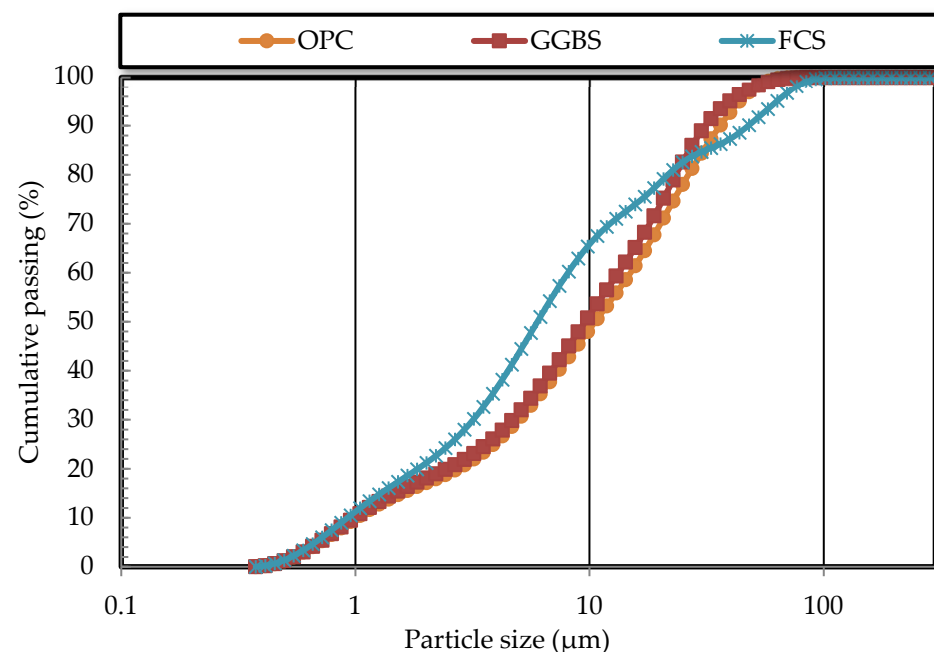


Figure 1. Grain size distribution of OPC, GGBS, and FCS as determined by a laser diffraction particle size analyzer.

X-ray fluorescence analysis results show that the major elements of these materials are silica, alumina, and calcium. The silicon dioxide concentrations in FCS, OPC, and GGBS were 52.8%, 16.3%, and 31.7%, respectively. Moreover, the FCS concentration of SO_3 , which is factor affecting delayed ettringite formation and durability enhancement, is lower than that in OPC and GGBS.

XRD mineralogical analysis results are summarized in Figure 2. FCS is composed of two major oxides, i.e., quartz (SiO_2) and calcite (CaCO_3), as well as minor oxides such as hematite (FeO_3) and anhydrite (CaSO_4). Similar findings were previously reported by Benzerzour et al. [9]. Oxides such as anhydrite are initiated by the calcination process and may enhance the cement hydration reaction.

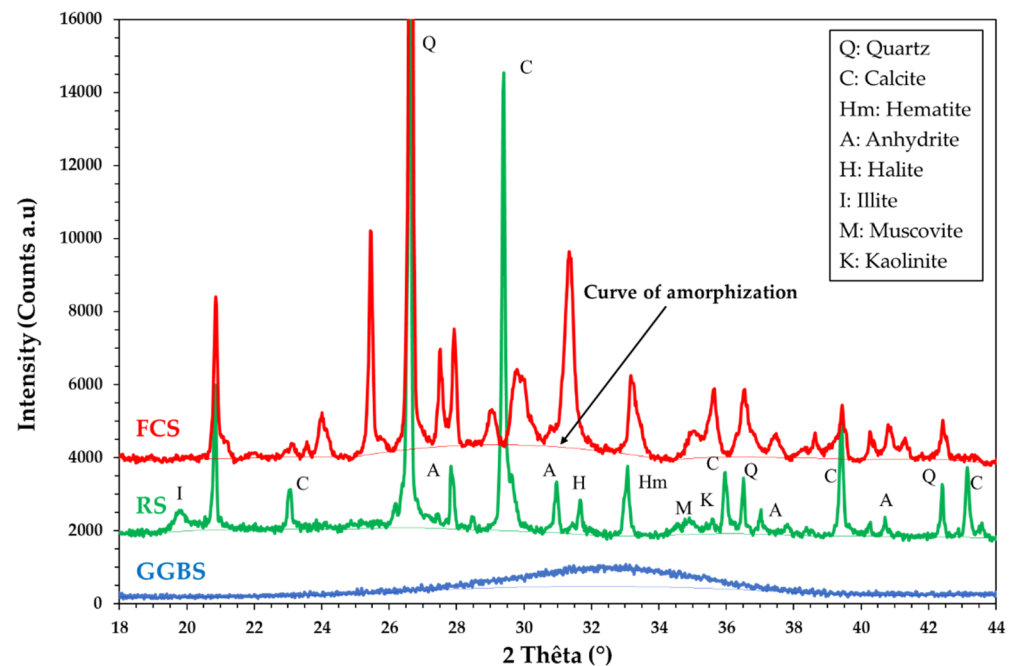


Figure 2. XRD pattern of ground granulated blast furnace slag (GGBS), raw sediment (RS), and flash-calcined sediment (FCS).

Moreover, the percentages of amorphous oxides in the raw sediment (RS) and FCS were estimated using Diffrac.Eva analysis software and found to be equal to 30% and 49%, respectively. The flash calcination process increased the percentage of amorphous oxides by almost 20%. During the pozzolanic reaction, amorphous silica reacts with portlandite CH to form additional C-S-H, which takes part in the densification of the cementitious matrix.

2.2. Mixture Design

2.2.1. Experimental Design and Mixture Model

The design of experiments (DoE) consists of using a set of statistical methods for the mathematical modelling of the system behavior after carrying out a limited number of selected tests to maximize the amount of collected information. Briefly, the influence of the input variables, called factors, (and their possible interaction) on the system is quantified by the assessment of the output variables or responses.

Several DoE methods, such as full factorial, Plackett–Burman, Box–Behnken, and Taguchi designs, have been used in the literature. However, the mixture design method was used herein because of its formulation optimization when the constituent proportions are considered [46–50]. It was considered the most appropriate method to use because the focus of this study was on determining the optimum replacement proportions of OPC by GGBS and FCS in the mix.

The mixture design method is applied to mixes for which the constituent amounts are interdependent. The three factors (X_1 , X_2 , and X_3) considered in this study are the volume

proportions of OPC, GGBS, and FCS, respectively. Therefore, the sum of the proportions of OPC, GGBS, and FCS is equal to 1 or 100% (i.e., $X_1 + X_2 + X_3 = 1$), as shown in the following equation:

$$\sum_{i=1}^n X_i = \text{OPC} + \text{GGBS} + \text{FCS} = 1 \text{ (or 100\%)} \quad (2)$$

The aim is to replace up to 50% of OPC with GGBS and FCS. The studied response, i.e., the 90-day compressive strength, was modelled using the Scheffé quadratic regression model equation [51]:

$$\hat{Y} = \sum_{i=1}^q \beta_i X_i + \sum_{i=1}^q \sum_{i < j}^q \beta_{ij} X_i X_j \quad (3)$$

where $\hat{Y}$ is the compressive strength after 90 days, q is the number of constituents ($q = 3$: OPC, GGBS, and FCS), β_i and β_{ij} are the model coefficients, and X_i is the volume proportion of constituent i .

To define the experimental domain, the amount of cement was varied between 50% and 100%, that of slag was varied from 0 to 50%, and that of FCS was varied from 0 to 20%. Table 2 represents the variation range of the three factors, i.e., X_1 , X_2 , and X_3 .

Table 2. X_1 , X_2 , and X_3 Lower and upper bounds.

Mixture Factor	X_1 –OPC	X_2 –GGBS	X_3 –FCS
Lower bound	0.5	0	0
Upper bound	1	0.5	0.2

As the experimental domain was irregular, no generic method such as a simplex lattice design or a simplex centroid design could be used to determine the formulation points to be tested. Therefore, the points were computer-generated according to the D-optimality criterion, which minimizes the uncertainty of the resulting model coefficients. Using Design Expert software (StatEase), the D-optimal design included 9 experimental points labelled as 1 to 9, as detailed in Figure 3 and Table 3. The points were distributed evenly to ensure a homogeneous coverage of the experimental domain. The results of the mixture design were also analyzed using Design Expert software.

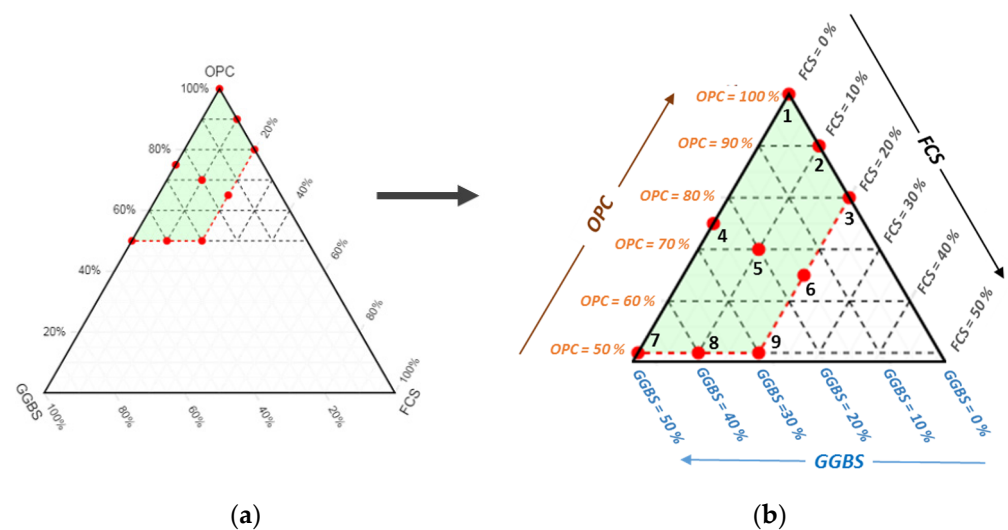


Figure 3. (a) Studied experimental mix designs. (b) Selected experimental mix designs.

2.2.2. Mix Preparation

Based on the selected mix designs summarized in Table 3, 9 normalized mortar mixes were prepared based on European standard NF EN 196-1. A normalized sand, which

was compliant with ISO 679:2009, was used with a maximum diameter ($D_{\max}$) of 2 mm. Mortars were composed of binder (450 g), normalized sand (1350 g), and an effective water-to-binder ratio (w/b) of 0.5. The replacement of OPC by mineral additions was volumetric.

Table 3. Selected experimental mix designs.

Exp. N°	X ₁ -OPC	X ₂ -GGBS	X ₃ -FCS
F1	100	0	0
F2	90	0	10
F3	80	0	20
F4	75	25	0
F5	70	20	10
F6	65	15	20
F7	50	50	0
F8	50	40	10
F9	50	30	20

Compressive strength tests were performed in triplicate on $4 \times 4 \times 16$ cm prismatic test samples (NF EN 196-1 [32]). After casting, the specimens were stored at a relative humidity of 90% for 24 h before demolding, then completely immersed in lime-saturated water at a constant temperature of 20 °C until testing.

2.3. Test Methods

2.3.1. Fresh State Characterization

The mortar workability was estimated using a flow table test with a $70 \times 100 \times 60$ mm flow mold [52]. After mixing, the mortar was introduced into the flow mold in two equal layers, then rodded without excessive settlement using a rod. The flow table was then lifted up 40 mm, then dropped 15 times, causing the concrete to flow. Then, the diameter of flow of the concrete was measured. Three samples were tested for each formulation. The maximum slump was measured in two perpendicular directions, and the average of these two values was recorded. Measurements were carried out 5 min after mixing the mortar.

The fresh density was measured according to European standard NF EN 1015-6 (1999) [53], and the air content was measured according to NF EN 1015-7 [54] using the pressure method and device equipped with a manometer. Moreover, the initial setting time was measured using an automatic Vicat apparatus (Vicatronic) according to NF 196-3 (2017) [40].

2.3.2. Heat of Hydration

The reaction of cement with water (hydration) is exothermic. Hydration heat monitoring in fresh mortar provides information about the reactivity of different binding matrix components with water. The hydration heat was determined using the semi-adiabatic Langavant method in compliance with NF EN 196-9 (2010) [55]. A fresh mortar sample was introduced into a semi-adiabatic calorimeter (CERILH), and the released heat was monitored over time. The mortar heat was then compared to the temperature of an inert sample in a reference calorimeter.

The heat of hydration (Q) dissipated into the environment can thus be determined using the following equation:

$$Q = \frac{C}{m_C} \theta_t \quad (4)$$

where C is the total heat capacity of the calorimeter (J/K), m_C is the weight of the binder (g), and θ_t is the difference in temperature between the mortar and the inert samples.

2.3.3. Mechanical Characterization

The compressive and flexural strengths were determined after 7, 28, and 90 days of curing in three ($4 \times 4 \times 16$ cm) prismatic test samples according to NF EN 196-1 [32].

Loading rates of 2400 ± 200 N/s and 50 ± 10 N/s were used for the compressive and flexural strength tests, respectively.

2.3.4. Dynamic Elastic Modulus

The dynamic elastic modulus was determined by measuring the fundamental resonance frequency according to NF EN 14146 (2004) [56]. In this test, the mortar samples were subjected to instant and longitudinal mechanical impulses. For each formulation, three samples were tested, and the average of the obtained values was considered. The dynamic modulus was obtained using the following equation:

$$Ed_L(\text{MPa}) = 4 \times 10^{-6} \cdot l^2 \cdot F_L^2 \cdot \rho \quad (5)$$

where Ed_L is the dynamic elastic modulus (MPa), F_L is the fundamental resonance frequency (Hz), ρ is the density (kg/m^3), and l is the length of the sample (m).

2.3.5. Mercury Porosity

The mortar porosity was determined using a Micromeritics AutoPore IV 9505 mercury porosimeter according to French standard NF P 94-410-3. This instrument enables the analysis of a wide range of porosities between 3 nm and 360 μm in diameter by applying a pressure of 0.5 psi to 30,000 psi (206 MPa). Measurements are conducted at low and high pressures for large and nanopore sizes, respectively. The test was carried out on 1 cm cubic mortar fragments. The porosity was measured 14, 28, and 90 days after discontinuing hydration to monitor its evolution in terms of the setting time for cement pastes.

2.3.6. SEM (Scanning Electron Microscopy) Analysis

SEM was used to view the surface of platinum metallized fragments based on a ternary binder mortar at high resolution after 90 days of curing. Metallization makes the observed material more conductive and capable of resisting an e-beam high-vacuum exposure. In this study, SEM analyses were performed using a Hitachi S-4300SE/N tabletop SEM with an accelerating voltage of 15 kV.

2.3.7. Leaching Test

The environmental impact of using sediment as an SCM with GGBS in a ternary binder was evaluated by a leaching test according to European standard EN 12457-2 [57]. The tested samples were prepared from a ground fragment taken from the investigated mortar mixes with a liquid-to-solid ratio of 10. The samples were agitated for 24 h, then vacuum-filtered with a 0.45 μm pore filter before leaching.

3. Results and Discussion

3.1. Mixture Design

3.1.1. Mortar Strength Prediction Model

The compressive strengths of the nine selected mortar mixes (Table 3) were determined in triplicate using $4 \times 4 \times 16$ cm prismatic test samples after 90 days of curing. Table 4 summarizes the average compressive strengths of the selected mortar mixes. The results show that the compressive strengths decreased from 66.45 MPa for the control mortar (F1) to 61.50 MPa for the mortar with 50% GGBS (F7) and to 55.56 MPa for the mortar with 20% FCS (F3). This compressive reduction can be explained by the cement dilution effect. On the other hand, the compressive strength increased for ternary mortars, even with 50% OPC substituted. In fact, the compressive strength of mix F8 (40% GGBS and 10% FCS) exhibited a 3.34% increase compared to F1 (100% OPC) and an 11.6% increase compared to F7 (50% OPC and 50% GGBS). Increased due to a synergy between two SCMs is well documented [58–62]. The improvement in compressive strength a ternary binder composed of OPC, GGBS (finer size), and FCS (even finer size) may be due to a better packing density in the mix.

Table 4. Compressive strength results of the selected mixes.

Mix N°	C (OPC) (100%)	S (GGBS) (100%)	F (FCS) (100%)	Compressive Strength after 90 Days (R_{c90}) (MPa)
F1	100	0	0	66.45 (± 0.47)
F2	90	0	10	62.72 (± 0.85)
F3	80	0	20	55.56 (± 1.24)
F4	75	25	0	66.75 (± 0.41)
F5	70	20	10	64.24 (± 1.12)
F6	65	15	20	57.02 (± 0.9)
F7	50	50	0	61.50 (± 0.09)
F8	50	40	10	68.67 (± 0.33)
F9	50	30	20	58.67 (± 0.27)

A model was developed in this study for the prediction of the 90-day compressive strength for mortars composed of OPC (C), GGBS (S), and FCS (F). The prediction model was based on the quadratic regression equation that defined by Scheffé (Equation (3)), which can be rewritten using the following equation:

$$R_{c90} \text{ (MPa)} = (C \cdot \beta_1 + L \cdot \beta_2 + A \cdot \beta_3) + (C \cdot L \cdot \beta_{12} + C \cdot A \cdot \beta_{13} + L \cdot C \cdot \beta_{23}) \quad (6)$$

where R_{c90} is the mortar compressive strength after 90 days; C, S, and F are the volume proportions of OPC, GGBS, and FCS, respectively; and β_1 , β_2 , β_3 , β_{12} , β_{13} , and β_{23} are the model coefficients. Design Expert software (StatEase) was used to determine the values of the coefficients of the model by inputting into the program the values of C, S, F, and R_{c90} as summarized in Table 4. Table 5 summarizes the obtained model coefficients.

Table 5. Scheffé quadratic model coefficients.

Coefficient	β_1	β_2	β_3	β_{12}	β_{13}	β_{23}
	66.48	51.87	−247.47	12.43	315.42	451.91

The compressive strength at 90 days (R_{c90}) for the mortars composed of OPC (C), GGBS (S), and FCS (F) can be predicted using the following equation:

$$R_{c90} \text{ (MPa)} = (66.48 C + 51.87 S - 247.47 F) + (12.43 C \cdot S + 315.42 C \cdot F + 451.91 S \cdot C). \quad (7)$$

3.1.2. Prediction Model Validation

The model described above was validated by comparing the 90-day compressive strengths predicted by the model to those experimentally measured at the same age. For this purpose, a new design mix was prepared solely for model validation. Three specimens from the new mix were tested at 90 days based on the experimental protocol defined in Section 3.2. The design mix is summarized in Table 6. It is worth noting that the new mix composition was not used in the determination of the prediction model. The compressive strengths of the new mix specimens at 90 days are summarized in Table 7. Design Expert software was used to determine the 95% confidence prediction interval for the 90-day compressive strength, which was found to be between 60.75 MPa and 66.91 MPa. The 90-day compressive strengths of the three new mix specimens were experimentally measured, and the mean and standard deviation of these strengths were found to be equal to 64.90 and 0.70, respectively.

The results show that the experimental compressive strengths of the new mixes lay within the prediction model 95% strength confidence interval set by the Design Expert software. The mean compressive strength value at 90 days is within the 95% confidence interval centered on the mean predicted strength value. This shows that the model was able to accurately predict the 90-day compressive strength for a mix with a composition unknown to the software. In other words, the mix was not used in the development of the

prediction model. This is an indication that the prediction model can be used to accurately predict the 90-day compressive strength for any mix given composed of C, S, and F.

Table 6. Mix composition for model validation.

Material	Composition (%)
OPC	50
GGBS	35
FCS	15

Table 7. The 90-day compressive strengths of the mixes for model validation.

Strength Prediction Model 95% Confidence Interval Set by Design Expert Software (StatEase) (MPa)	Experimentally Measured 90-Day Compressive Strength (MPa)
60.75–66.91	64.90 (± 0.70)

3.2. Fresh State Properties

To investigate the effect of the ternary structure (OPC + GGBS + FCS), five formulations including three ternary formulations based on GGBS and FCS (TSM), one binary formulation (BSM), and one reference formulation (RM) were chosen for a more thorough study. The compositions of the blended cements are presented in Table 8.

Table 8. Blended cement compositions (%).

Index	OPC	GGBS	FCS
RM	100	–	–
BSM 50-0	50	50	–
TSM 40-10	50	40	10
TSM 35-15	50	35	15
TSM 30-20	50	30	20

The fresh state properties of ternary binders based on GGBS and FCS (i.e., density, air content, flow, and initial setting time), are summarized in Table 9.

Table 9. Blended cement mortar properties.

	RM	BSM 50-0	TSM 40-10	TSM 35-15	TSM 30-20
Fresh density (kg/m ³)	2 188	2 151	2 207	2 212	2 215
Air content (%)	7.2	6.6	4.0	4.1	4.5
Flow (cm)	22.4	22.0	21.5	21.0	18.7
Initial setting time (min)	256	338	318	311	308

The flow results show that the use of FCS leads to a loss of workability, which depends on the percentage of FCS in the ternary matrix. This is because the specific surface area of FCS, which differs from that of GGBS and OPC, causes divergent water absorption behaviors. Moreover, material characterization shows that the water demand of FCS is far superior to that of OPC and GGBS, which leads to higher retention of the mixing water, which is no longer available to act as an intergranular lubricant in the mix fresh state.

An improvement in the mix density was also noted, which is attributed to the fineness of FCS and its rounded shape due to its treatment [41]. Thus, the use of FCS enhances the granular compactness and lowers the air content, leading to a better density in the fresh state.

The air content results agree with these findings. The results show that the use of a ternary binder based on FCS and GGBS causes reduced air content. The use of fine SCMs causes intergranular pores to be filled and increases the compactness of mortars [34].

Both BSM and TSM presented a delay in terms of the initial setting time compared to RM, which is attributed to the cement dilution effect when 50% of OPC is substituted [63–65]. Furthermore, the incorporation of FCS in the binder reduced this delay. An acceleration of the setting time proportional to the percentage of FCS in TSM was observed. This acceleration might be due to the fineness of FCS, which induces a fast hydration rate due to the nucleation effect [9,66].

3.3. Hydration Heat

The binder cumulative hydration heat results, which are summarized in Figure 4, show a lower heat release for the mixes containing GGBS and FCS compared to the reference mix, with decreases of 37% and 51% for TSM and BSM, respectively, compared to RM. It is worth noting that the maximum hydration heats of RM, TSM, and BSM were found to be equal to 231.07 J/g, 146.29 J/g, and 112.25 J/g, respectively. The slow hydration of GGBS, which forms an acidic layer of aluminum hydroxide $\text{Al}(\text{OH})_3$, prevents water from accessing and dissolving the grains [67]. Moreover, the addition of FCS leads to higher heat release compared to the binary binder (BSM), equal to 26.29 J/g, 35.87 J/g, and 39.85 J/g for TMS 40-10, TMS 35-15, and TMS 20-30, respectively. The fineness of the calcined sediment ($D_{50} = 5.75 \mu\text{m}$) and the higher specific surface area accelerate the hydration via a nucleation effect [66,68,69].

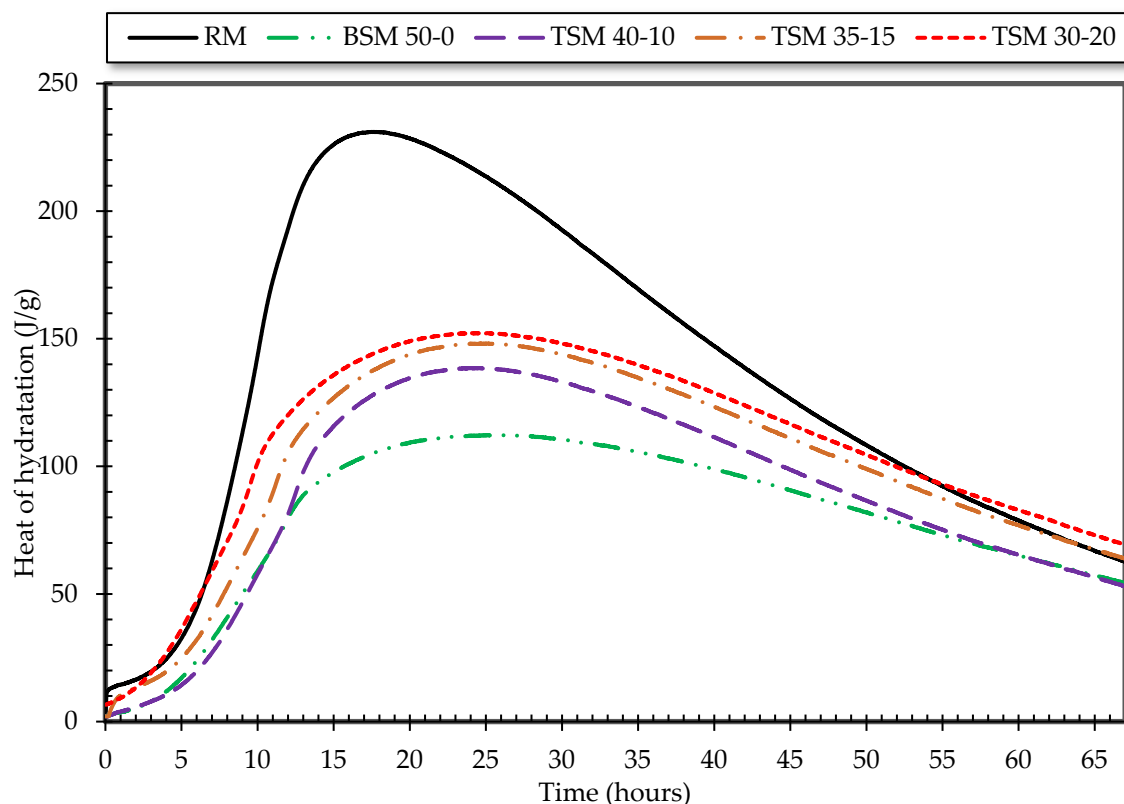


Figure 4. Heat hydration versus time as determined by semi-adiabatic Langavant calorimetry.

3.4. Hardened State Properties

3.4.1. Compressive and Bending Strengths

The mechanical strengths were determined at 14, 28, 60, and 90 days. Figures 5 and 6 show the binder compressive and flexural strength results, respectively.

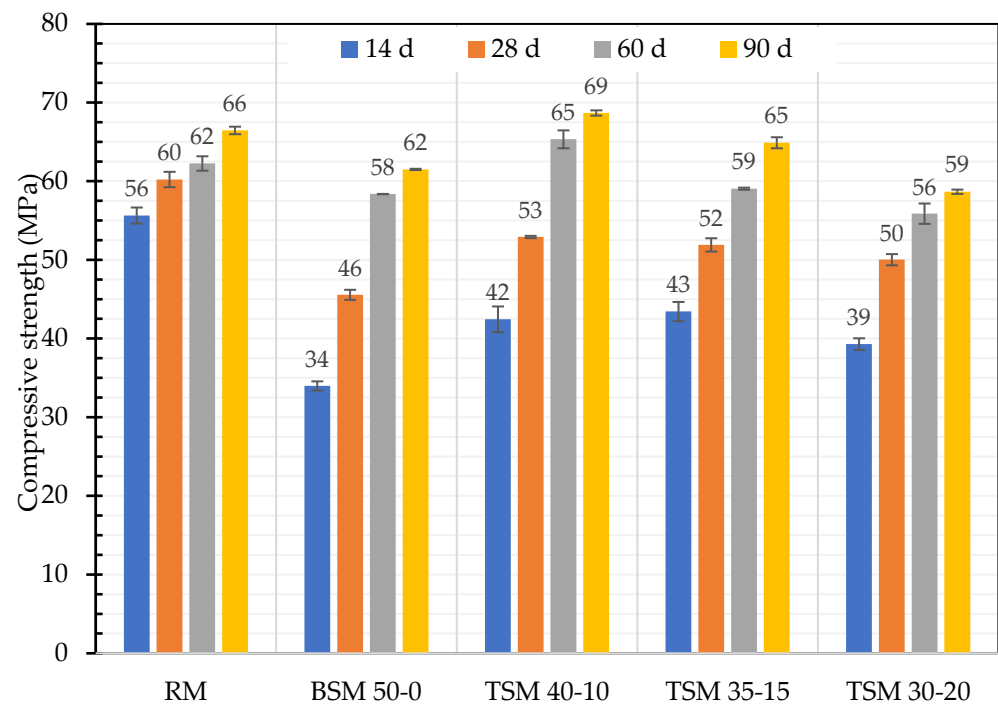


Figure 5. Compressive strength of RM, BSM 50-0, TSM 40-10, TSM 35-15, and TSM 30-20 mortars at 14, 28, 60, and 90 days.

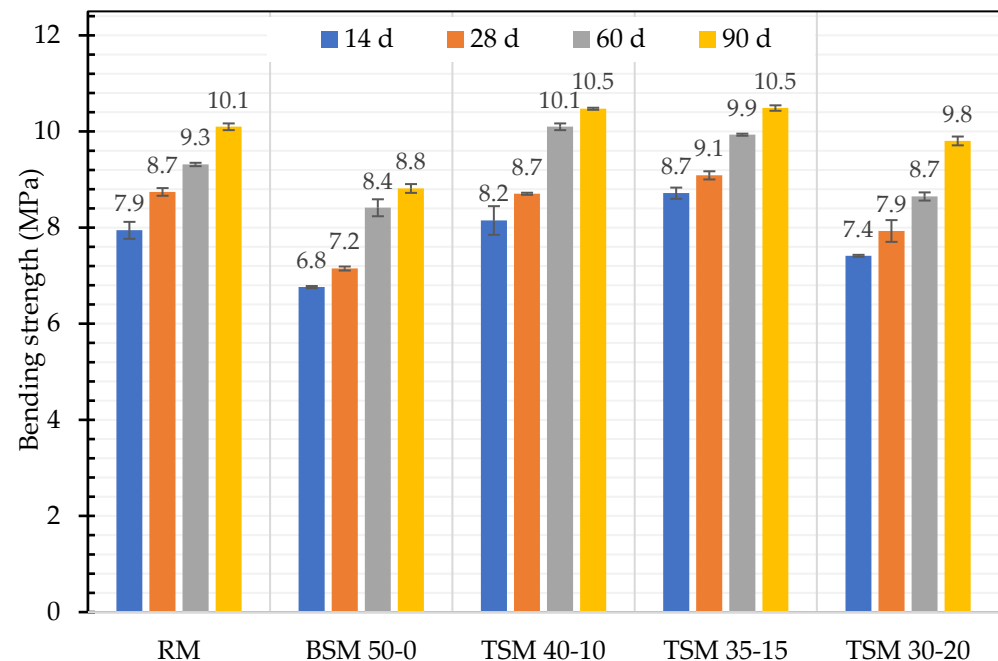
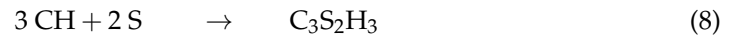


Figure 6. Bending strength of RM, BSM 50-0, TSM 40-10, TSM 35-15, and TSM 30-20 mortars at 14, 28, 60, and 90 days.

The results show that the compressive strength increases with the curing age for all binders. The compressive strength of RM is higher than that of BSM and TMS at 14 days. The BSM and TMS binders contains 50% less OPC than RM. Thus, they contain a reduced amount of C_3S , which is responsible for the formation of a resistant C-S-H gel upon its quick hydration in the short term. On the other hand, the compressive strengths of BSM and TSM significantly increased between 14 and 90 days, representing an increase of 80% and 53.3% for BSM 50-0 and TSM, respectively. On the other hand, the compressive strength of RM increased by only 18%. This may be due to the pozzolanic reaction of the GGBS and

calcined sediment additions [17,70,71]. Indeed, during the hydration of OPC, portlandite $\text{Ca}(\text{OH})_2$ is formed by the combination of Ca^{2+} and OH^- released by alite (C_3S) and belite (C_2S). The portlandite reacts with silica SiO_2 and alumina Al_2O_3 contributed by FCS and forms more C-S-H according to the following equation [72]:



where CH = portlandite, and S = SiO_2 .

TSM 40-10 binder, which is composed of 40% GGBS and 10% FCS, had a compressive strength that exceeded that of RM after 60 days of curing by 4.8%. Other works also mentioned an improvement in mechanical strength after 60 days of curing using 10% FCS [26]. This is an indication of the high level of reactivity of FCS, which is linked to the physical and chemical aspects of sediment, namely granulometry and shape. The fine spherical particles of sediment, which are obtained by flash calcination treatment, constitute nucleation zones that are favorable to the formation of hydrates. Moreover, the chemical elements of sediment such as sodium sulfate Na_2SO_4 can activate the slag [73,74]. The activation of certain sediment clayey phases (including kaolinite) by flash calcination also contributes to an increased pozzolanic reaction.

The bending strength results (Figure 6) show that binders based on FCS have higher bending strengths than those of control mortars after 60 days of curing for sediment percentages up to 15%.

According to these results, TSM 40-10 is the optimal formulation. The same result was also reported by Hadj Sadok et al. [28], who investigated ternary binders based on calcined sediments and GGBS.

3.4.2. Dynamic Elastic Modulus

Figure 7 shows the variation of the binder dynamic moduli over time. The dynamic moduli of all the binders increased with curing time. This can be explained by the pozzolanic activity, which contributes to the densification of the cementitious matrix and therefore the improvement of the dynamic modulus. It is worth noting that the dynamic moduli of TSM 40-10 and TSM 35-15 binder at 90 days were higher than that of RM binder. This seems to confirm the obtained mechanical results. FCS improves the mortar elasticity by physical and chemical effects.

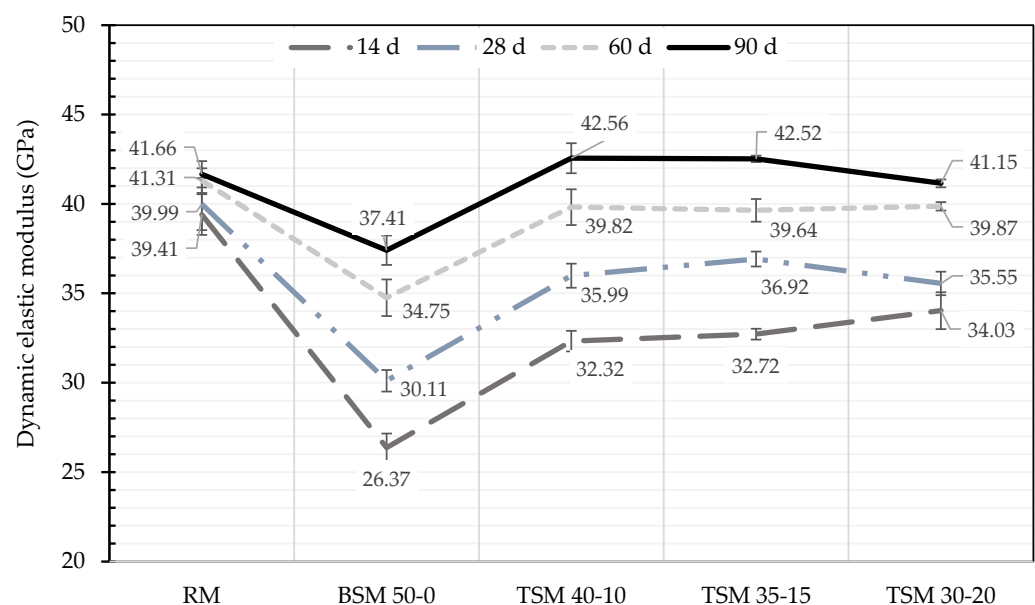


Figure 7. Dynamic elastic moduli of RM, BSM 50-0, TSM 40-10, TSM 35-15, and TSM 30-20 mortars at 14, 28, 60, and 90 days.

3.5. Microstructural Characterization

3.5.1. Mercury Porosity

The mortar porosity results at the ages of 14, 28, and 90 days are shown in Figures 8 and 9. The total binder porosity values, which ranged between 7.12 and 12.55%, decreased with age. At 14 days, the TSM and RM porosity values were similar (12% on average). However, at 90 days, the porosity values of the mortars containing FCS were lower than those of the control mortar. This is due to the pozzolanic activity of flash-calcined sediments and slag, which generates new hydrates that fill the pores and improve the density of the binder. Moreover, pozzolanic activity is linked to other factors such as the type of the formed hydration phases and the gelation degree of the semi-crystallized hydrates (e.g., C-S-H), the Ca/Si ratio, the hardening temperature, and the nature of the mineral additives [75,76].

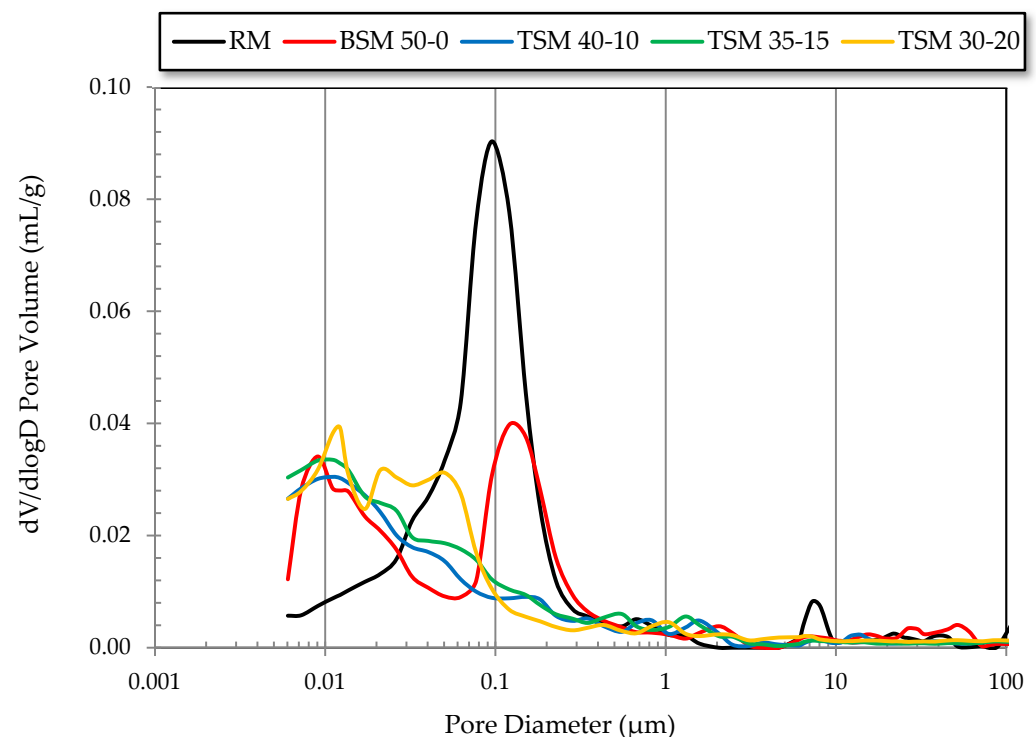


Figure 8. Pore distribution of RM, BSM 50-0, TSM 40-10, TSM 35-15, and TSM 30-20 mortars at 90 days.

Figure 9 displays the pore size distribution in each binder at 14, 28, and 90 days. The results show that the RM pore diameters are larger than those of ternary binders containing FCS. At 90 days, more than 63% of the pores in ternary matrices had a size smaller than 50 nm. On the other hand, less than 20% of pores in RM had a size smaller than 50 nm. Moreover, the highest concentration of TSM porosity was observed for a diameter smaller than 20 nm. Thus, the use of ternary binders leads to smaller pore sizes, which is beneficial in terms of durability. According to the literature [77–79], the following four classes of structural porosity can be defined:

- Class A corresponds to typical mesogel porosities, with pore sizes between 1 and 25 nm. This class comprises porosities between the C-S-H chains in the matrix. The higher the porosity value in this class, the more C-S-H gel in the matrix;
- Class B corresponds to typical microcapillary and mesocapillary porosities (pore size between 25 and 50 nm) between the groups of C-S-H chains;
- Class C corresponds to a typical macrocapillary porosity (pore size between 50 nm and 1 μm) in the structure of long C-S-H chains;
- Class D: corresponds to a macrocapillary porosity (pore size between 1 and 10 μm) linked to wall effects and the morphology of glass powder.

As shown in Figure 9, the use of ternary binders results in an increase in class A and class B porosities. However, RM results in higher concentrations of class C porosities.

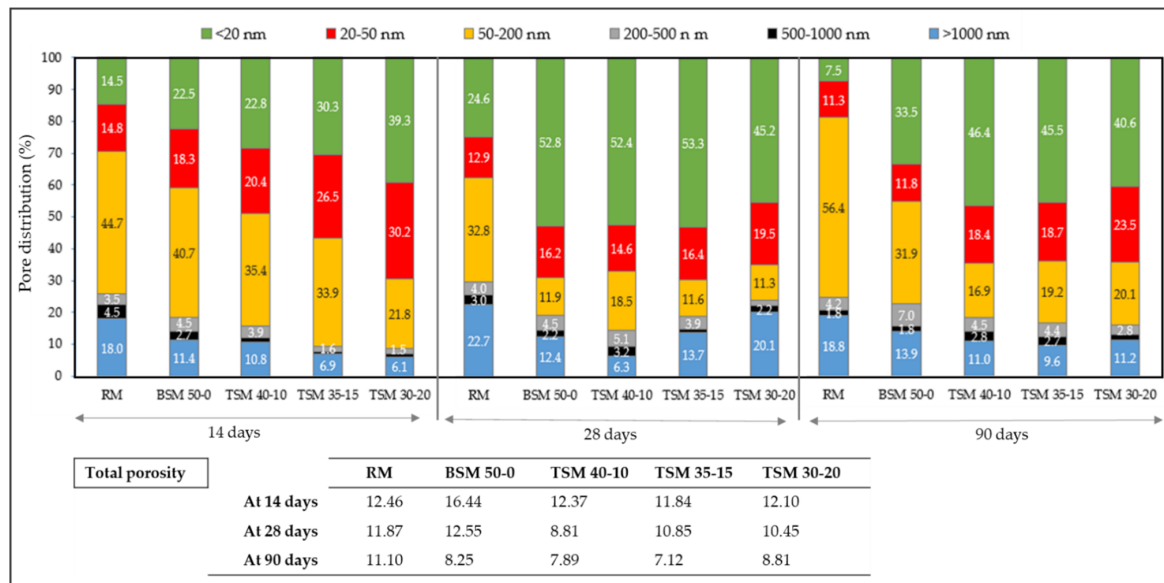


Figure 9. Total porosity and pore size distribution at 14, 28, and 90 days.

3.5.2. SEM Observation

Figure 10 shows SEM images for RM and ternary binders based on GGBS and FCS after 90 days of curing. Figure 10a shows RM microscopic elements such as the C-S-H gel and portlandite $\text{Ca}(\text{OH})_2$. These two elements result from the hydration of alite (C_2S) and belite (C_3S), which are the main components of OPC. The hydration reactions leading to the formation of these two elements are expressed as follows.

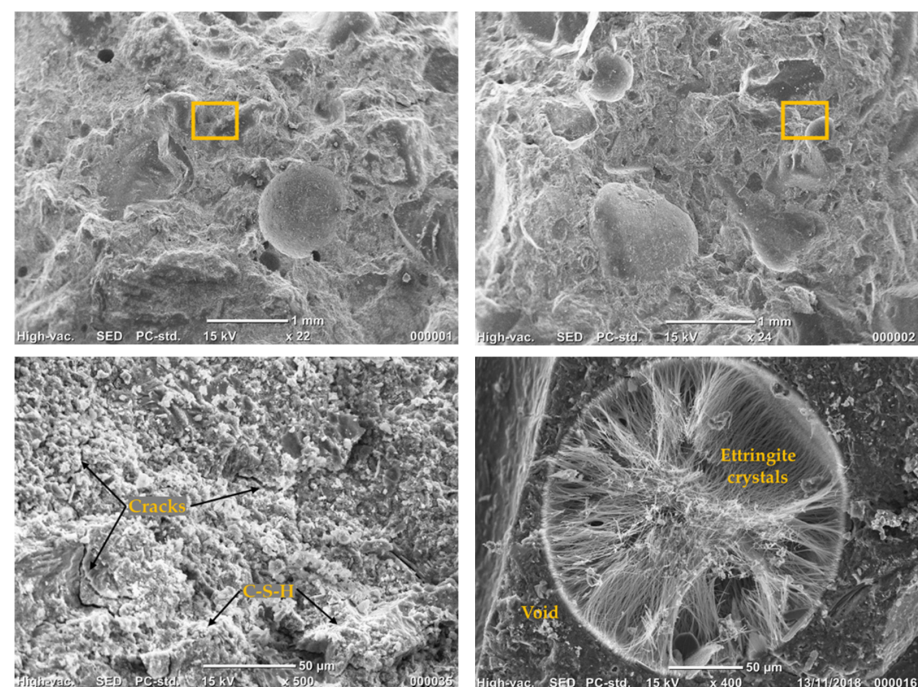
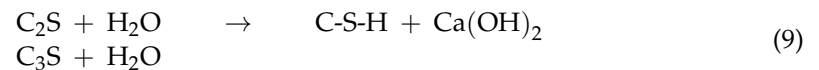


Figure 10. Cont.

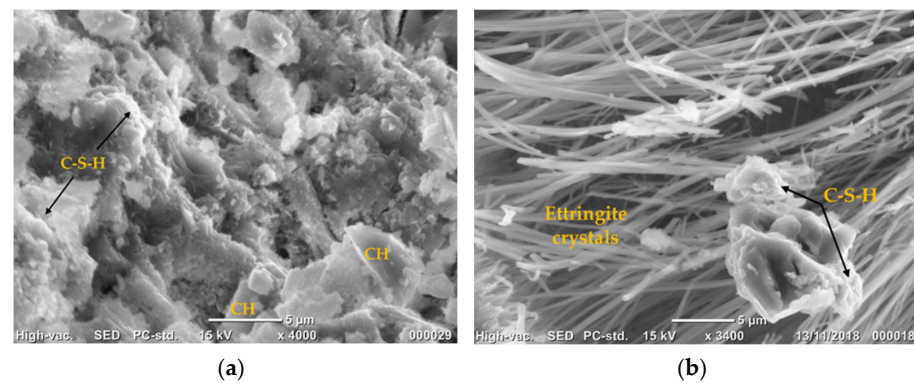


Figure 10. SEM observations of the reference mix (a) and the ternary binders based on GGBS and FCS (b) after 90 days of curing.

The SEM images for TSM 40-10 in Figure 10b show a dense binder microstructure with elements such as C-S-H and ettringite. The pores in the binder are filled with the supplementary hydration products. Hadj Sadok et al. [28] reported that the supplementary products result from the pozzolanic activity of the additions (i.e., FCS and GGBS). These observations confirm that the ternary binders based on FCS and GGBS have a denser structure and a lower porosity than those of RM.

3.6. Environmental Acceptability: Leaching Test

A leaching test was carried out to evaluate the environmental impact of the use of sediment as an SCM with GGBS in the formulation of a ternary blended binder. The results of the leaching test carried out on different cementitious matrices are presented in Table 10.

Table 10. Mobility of heavy metals in the studied formulations (mg/kg).

	RM	BSM 50-0	TSM 40-10	TSM 35-15	TSM 30-20	Limit
As	<0.11	<0.11	<0.11	<0.11	<0.11	0.50
Ba	14.33	8.23	9.82	5.44	6.86	20.0
Cd	<0.009	<0.009	<0.009	<0.009	<0.009	0.04
Cr	0.479	0.055	0.177	0.096	0.077	0.50
Cu	<0.02	<0.02	<0.02	<0.02	<0.02	2.00
Mo	<0.09	<0.09	0.091	0.126	0.112	0.50
Ni	<0.05	<0.05	<0.05	<0.05	<0.05	0.40
Pb	<0.03	<0.03	<0.03	<0.03	<0.03	0.50
Sb	<0.06	<0.06	<0.06	<0.06	<0.06	0.06
Se	<0.08	<0.08	<0.08	<0.08	<0.08	0.10
Zn	<0.01	<0.01	<0.01	<0.01	<0.01	4.00
Fluorides	5.60	4.70	4.90	5.00	5.10	10.0
Chlorides	39.0	47.0	147	220	245	800
Sulfates	216	26.0	37.0	36.0	33.0	1000

The results show that the use of ternary blended binders reduced the concentration of few pollutants within the binder. As a matter of fact, the concentrations of barium, chromium, fluorides, and sulfates in the sediment-based binders were lower than those in the RM binder. On the other hand, the concentrations of molybdenum and chlorides increased. Because the concentrations of Cd, Cu, Ni, Pb, Sb, Se, and Zn in all binders were under the detection thresholds, the use FCS as a 20% SCM substitute does not provoke a chemical change in the cement matrix. Moreover, the pollutant concentrations in all binders remained below the threshold values defined by French Directive no. 0289 for inert waste [80].

4. Conclusions

Reducing the amount of CO₂ associated with cement production has been a major issue in the last few decades. The use of SCMs as a partial replacement for cement contributes to reductions in the environmental impact of CO₂ emissions. In this study, a ternary eco-binder composed of dredged sediment and slag was developed as a partial replacement for cement (up to 50%) using a new method of sediment treatment, namely flash calcination. The results of this study highlight the positive impact of using ternary binders on the mechanical properties of mortars. Based on these results, the following conclusions can be drawn:

- The mixture design method was used to optimize the design of ternary blended binders. A model (DoE) was developed to produce a limited number of mixes, maximizing the amount of collected information. The DoE model was used to predict the 90-day compressive strength and was validated by experimental results for mortars containing OPC (C), GGBS (S), and FCS (F).
- FCS reduces the workability of mortars due to its considerable water demand. On the other hand, FCS enhances the compactness of mortar, which results in an increase in the density and a decrease in the air content proportional to its percentage in the mortar.
- The substitution of 50% OPC by FCS and GGBS results in a lower hydration heat peak and a delayed initial setting time. However, FCS reduces the impacts of this phenomenon due to its fine particles. This improvement depends on the quantity of FCS contained in the ternary blended binder.
- The use of TSM 40-10 (i.e., 10% FCS, 40% GGBS, and 50% OPC) increased the mechanical properties (compressive and bending strengths and dynamic elastic modulus) at 90 days compared to those of RM composed of 100% OPC. TSM 40-10, which is composed of 50% OPC, 40% GGBS, and 10% FCS, is the optimal formulation.
- The use of FCS reduces the total porosity of mortars and their pore sizes, which can significantly improve their durability. SEM images showed high levels of voids and portlandite in RM. However, when FCS was added, these pores were filled with supplementary hydration products resulting from the pozzolanic activity of FCS.
- The environmental impact of using ternary binders was assessed by performing leaching tests. The results show that using FCS does not imply a chemical change in the cement matrix.

This study brings to light the beneficial effect of applying the flash calcination method to sediments for their valorization as SCMs in ternary blended binders. We conclude that the use of ternary binders containing flash-calcined sediments and GGBS is a feasible strategy to produce an eco-friendly material that can be used in the production of sustainable building material with adequate compressive strength. The durability of concretes based on these ternary binders was investigated and will be the subject of further publications. Last but not least, like any other secondary resource, sediment composition can be variable, so for quality control, sediments should be characterized before use.

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Conflicts of Interest: The authors declare that they have no conflict of interest.

References

1. Benhelal, E.; Zahedi, G.; Shamsaei, E.; Bahadori, A. Global strategies and potentials to curb CO₂ emissions in cement industry. *J. Clean. Prod.* **2013**, *51*, 142–161. [\[CrossRef\]](#)
2. Zhang, R.; Panesar, D.K. Sulfate resistance of carbonated ternary mortar blends: Portland cement, reactive MgO and supplementary cementitious materials. *J. Clean. Prod.* **2019**, *238*, 117933. [\[CrossRef\]](#)
3. Li, R.; Lei, L.; Sui, T.; Plank, J. Effectiveness of PCE superplasticizers in calcined clay blended cements. *Cem. Concr. Res.* **2021**, *141*, 106334. [\[CrossRef\]](#)
4. Flatt, R.J.; Roussel, N.; Cheeseman, C.R. Concrete: An eco material that needs to be improved. *J. Eur. Ceram. Soc.* **2012**, *32*, 2787–2798. [\[CrossRef\]](#)
5. Scrivener, K.L.; John, V.M.; Gartner, E.M. Eco-efficient cements: Potential economically viable solutions for a low-CO₂ cement-based materials industry. *Cem. Concr. Res.* **2018**, *114*, 2–26. [\[CrossRef\]](#)
6. Miller, S.A.; John, V.M.; Pacca, S.A.; Horvath, A. Carbon dioxide reduction potential in the global cement industry by 2050. *Cem. Concr. Res.* **2017**, *114*, 115–124. [\[CrossRef\]](#)
7. Amar, M.; Benzerzour, M.; Safhi, A.E.M.; Abriak, N.E. Durability of a cementitious matrix based on treated sediments. *Case Stud. Constr. Mater.* **2018**, *8*, 258–276. [\[CrossRef\]](#)
8. Van Bunderen, C.; Benboudjema, F.; Snellings, R.; Vandewalle, L.; Cizer, Ö. Experimental analysis and modelling of mechanical properties and shrinkage of concrete recycling flash calcined dredging sediments. *Cem. Concr. Compos.* **2021**, *115*, 103787. [\[CrossRef\]](#)
9. Benzerzour, M.; Amar, M.; Abriak, N.E. New experimental approach of the reuse of dredged sediments in a cement matrix by physical and heat treatment. *Constr. Build. Mater.* **2017**, *140*, 432–444. [\[CrossRef\]](#)
10. Zeraoui, A.; Benzerzour, M.; Maherzi, W.; Mansi, R.; Abriak, N.E. New software for the optimization of the formulation and the treatment of dredged sediments for utilization in civil engineering. *J. Soils Sediments* **2020**, *20*, 2709–2716. [\[CrossRef\]](#)
11. Siddique, R.; Khan, M.I. *Supplementary Cementing Materials*; Springer: Berlin/Heidelberg, Germany, 2011.
12. San Nicolas, R.; Cyr, M.; Escadeillas, G. Characteristics and applications of flash metakaolins. *Appl. Clay Sci.* **2013**, *83–84*, 253–262. [\[CrossRef\]](#)
13. Davies, T.W. Equipment for the study of the flash heating of particle suspensions. *High Temp. Technol.* **1984**, *2*, 141–147. [\[CrossRef\]](#)
14. Inocente, J.M.; Elyseu, F.; Jaramillo Nieves, L.J.; Jiusti, J.; Cargnin, M.; Peterson, M. Production and characterization of high-reactivity metakaolins calcined in flash reactor. *Appl. Clay Sci.* **2021**, *213*, 106247.
15. Bucher, R.; Cyr, M.; Escadeillas, G. Performance-based evaluation of flash-metakaolin as cement replacement in marine structures—Case of chloride migration and corrosion. *Constr. Build. Mater.* **2021**, *267*, 120926. [\[CrossRef\]](#)
16. Benzerzour, M.; Maherzi, W.; Amar, M.A.A.; Abriak, N.E.; Damidot, D. Formulation of mortars based on thermally treated sediments. *J. Mater. Cycles Waste Manag.* **2018**, *20*, 592–603. [\[CrossRef\]](#)
17. Amar, M.; Benzerzour, M.; Abriak, N.E.; Mamindy-Pajany, Y. Study of the pozzolanic activity of a dredged sediment from Dunkirk harbour. *Powder Technol.* **2017**, *320*, 748–764. [\[CrossRef\]](#)
18. Snellings, R.; Horckmans, L.; Van Bunderen, C.; Vandewalle, L.; Cizer, Ö. Flash-calcined dredging sediment blended cements: Effect on cement hydration and properties. *Mater. Struct. Constr.* **2017**, *50*, 241. [\[CrossRef\]](#)
19. Dang, T.A.; Kamali-Bernard, S.; Prince, W.A. Design of new blended cement based on marine dredged sediment. *Constr. Build. Mater.* **2013**, *41*, 602–611. [\[CrossRef\]](#)
20. Diouri, A.; Dang, T.A.; Sassi, O.; Bernard, F. Properties and hydration behavior of blended clinker and portland- sediment cement pastes. *Chem. Mater. Res.* **2013**, *5*, 27–30.
21. Faure, A.; Smith, A.; Coudray, C.; Anger, B.; Colina, H.; Moulin, I.; Thery, F. Ability of Two Dam Fine-Grained Sediments to be Used in Cement Industry as Raw Material for Clinker Production and as Pozzolanic Additional Constituent of Portland-Composite Cement. *Waste Biomass Valorization* **2017**, *8*, 2141–2163. [\[CrossRef\]](#)
22. Safer, O.; Belas, N.; Belaribi, O.; Belguesmia, K.; Bouhamou, N.E.; Mebrouki, A. Valorization of Dredged Sediments as a Component of Vibrated Concrete: Durability of These Concretes Against Sulfuric Acid Attack. *Int. J. Concr. Struct. Mater.* **2018**, *12*, 44. [\[CrossRef\]](#)
23. Snellings, R.; Cizer, Ö.; Horckmans, L.; Durdziński, P.T.; Dierckx, P.; Nielsen, P.; Van Balen, K.; Vandewalle, L. Properties and pozzolanic reactivity of flash calcined dredging sediments. *Appl. Clay Sci.* **2016**, *129*, 35–39. [\[CrossRef\]](#)
24. Van Bunderen, C.; Snellings, R.; Vandewalle, L.; Cizer, Ö. Early-age hydration and autogenous deformation of cement paste containing flash calcined dredging sediments. *Constr. Build. Mater.* **2019**, *200*, 104–115. [\[CrossRef\]](#)
25. Daher, J.; Kleib, J.; Benzerzour, M.; Abriak, N.-E.; Aouad, G. Recycling of Flash-Calcined Dredged Sediment for concrete 3D Printing. *Buildings* **2022**, *12*, 1400. [\[CrossRef\]](#)
26. Amar, M.A.A. *Traitement des Sédiments de Dragage Pour Une Valorisation Dans les Matrices Cimentaires*. Ph.D. Thesis, Université Lille 1, Lille, France, 2017. (In French)

27. Amar, M.; Benzerzour, M.; Kleib, J.; Abriak, N.E. From dredged sediment to supplementary cementitious material: Characterization, treatment, and reuse. *Int. J. Sediment Res.* **2021**, *36*, 92–109. [\[CrossRef\]](#)
28. Hadj Sadok, R.; Maherzi, W.; Benzerzour, M.; Lord, R.; Torrance, K.; Zambon, A.; Abriak, N.E. Mechanical properties and microstructure of low carbon binders manufactured from calcined canal sediments and ground granulated blast furnace slag (GGBS). *Sustainability* **2021**, *13*, 9057. [\[CrossRef\]](#)
29. Amar, M.; Benzerzour, M.; Abriak, N.-E. Designing Efficient Flash-Calcined Sediment-Based Ecobinders. *Materials* **2022**, *15*, 7107. [\[CrossRef\]](#)
30. Chu, D.C.; Amar, M.; Kleib, J.; Benzerzour, M.; Betrancourt, D.; Abriak, N.E.; Nadah, J. The Pozzolanic Activity of Sediments Treated by the Flash Calcination Method. *Waste Biomass Valorization* **2022**, *13*, 4963–4982. [\[CrossRef\]](#)
31. NF EN 197-1; Cement Part 1: Composition, Specifications and Conformity Criteria for Common Cements. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2012.
32. NF EN 196-1; Methods of Testing Cement—Part 1: Determination of Strength. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2016.
33. NF EN 15167-1; Ground Granulated Blast Furnace Slag for use in Concrete, Mortar and Grout—Part 1: Definitions, Specifications and Conformity Criteria. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2006.
34. LKAB Minerals. Ground Granulated Blast Furnace Slag—Ground Granulated Blast Furnace Slag (GGBS) Can Replace over 70% of the Portland Cement Content in a Concrete Mix. Available online: <https://www.lkabminerals.com/en/products/ggbs> (accessed on 7 August 2021).
35. Bouhamou, N.E.; Mostefa, F.; Mebrouki, A.; Bendani, K.; Belas, N. Influence of dredged sediment on the shrinkage behavior of self-compacting concrete. *Mater. Tehnol.* **2016**, *50*, 127–135. [\[CrossRef\]](#)
36. Safhi, A.e.M.; Rivard, P.; Yahia, A.; Benzerzour, M.; Khayat, K.H. Valorization of dredged sediments in self-consolidating concrete: Fresh, hardened, and microstructural properties. *J. Clean. Prod.* **2020**, *263*, 121472. [\[CrossRef\]](#)
37. Dixit, A.; Du, H.; Pang, S.D. Performance of mortar incorporating calcined marine clays with varying kaolinite content. *J. Clean. Prod.* **2021**, *282*, 124513. [\[CrossRef\]](#)
38. NF EN 1097-7; Tests for Mechanical and Physical Properties of Aggregates—Part 7: Determination of the Particle Density of Filler—Pycnometer Method. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2008.
39. NF EN ISO 18757; Determination of Specific Surface Area of Ceramic Powders by Gas Adsorption Using the BET Method. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2008.
40. NF EN 196-3; Methods of Testing Cement—Part 3: Determination of Setting Times and Soundness. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2017.
41. Claverie, M.; Martin, F.; Tardy, J.P.; Cyr, M.; De Parseval, P.; Grauby, O.; Le Roux, C. Structural and chemical changes in kaolinite caused by flash calcination: Formation of spherical particles. *Appl. Clay Sci.* **2015**, *114*, 247–255. [\[CrossRef\]](#)
42. Ilić, B.; Radonjanin, V.; Malešev, M.; Zdujić, M.; Mitrović, A. Effects of mechanical and thermal activation on pozzolanic activity of kaolin containing mica. *Appl. Clay Sci.* **2016**, *123*, 173–181. [\[CrossRef\]](#)
43. Yao, G.; Wang, Z.; Yao, J.; Cong, X.; Anning, C.; Lyu, X. Pozzolanic activity and hydration properties of feldspar after mechanical activation. *Powder Technol.* **2021**, *383*, 167–174. [\[CrossRef\]](#)
44. Gmür, R. Influence of aging conditions upon the properties of calcined clay and its performance as supplementary cementitious material. *Cem. Concr. Compos.* **2016**, *72*, 114–124. [\[CrossRef\]](#)
45. Neißer-Deiters, A.; Scherb, S.; Beuntner, N.; Thienel, K.C. Influence of the calcination temperature on the properties of a mica mineral as a suitability study for the use as SCM. *Appl. Clay Sci.* **2019**, *179*, 105168. [\[CrossRef\]](#)
46. Zaitri, R.; Bederina, M.; Bouziani, T.; Makhoulfi, Z.; Hadjoudja, M. Development of high performances concrete based on the addition of grinded dune sand and limestone rock using the mixture design modelling approach. *Constr. Build. Mater.* **2014**, *60*, 8–16. [\[CrossRef\]](#)
47. Akalin, O.; Akay, K.U.; Sennaroglu, B.; Tez, M. Optimization of chemical admixture for concrete on mortar performance tests using mixture experiments. *Chemom. Intell. Lab. Syst.* **2010**, *104*, 233–242. [\[CrossRef\]](#)
48. Bouzalakos, S.; Dudeney, A.W.L.; Chan, B.K.C. Formulating and optimising the compressive strength of controlled low-strength materials containing mine tailings by mixture design and response surface methods. *Miner. Eng.* **2013**, *53*, 48–56. [\[CrossRef\]](#)
49. Abouhussien, A.A.; Hassan, A.A.A. Application of statistical analysis for mixture design of high-strength self-consolidating concrete containing metakaolin. *J. Mater. Civ. Eng.* **2014**, *26*, 04014016. [\[CrossRef\]](#)
50. Ghafari, E.; Bandarabadi, M.; Costa, H.; Júlio, E. Prediction of fresh and hardened state properties of UHPC: Comparative study of statistical mixture design and an artificial neural network model. *J. Mater. Civ. Eng.* **2015**, *27*, 04015017. [\[CrossRef\]](#)
51. Scheffé, H. The simplex-centroid design for experiments with mixtures. *J. R. Stat. Soc. Ser. B Methodol.* **1963**, *25*, 235–263. [\[CrossRef\]](#)
52. NF EN 1015-3; Methods of Test for Mortar for Masonry—Part 3: Determination of Consistence of Fresh Mortar (by Flow Table). AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 1999.

53. NF EN 1015-6; Methods of Test for Mortar for Masonry—Part 6: Determination of Bulk Density of Fresh Mortar. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 1999.
54. NF EN 1015-7; Methods of Test for Mortar for Masonry—Part 7: Determination of Air Content of Fresh Mortar. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 1999.
55. NF EN 196-9; Methods of Testing Cement—Part 9: Heat of Hydration—Semi-Adiabatic Method. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2010.
56. NF EN 14146; Determination of the Dynamic Modulus of Elasticity (by Measuring the Fundamental Resonance Frequency). AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2004.
57. NF EN 12457-2; Leaching—Compliance Test for Leaching of Granular Waste Materials and Sludges—Part 2: One Stage Batch Test at a Liquid to Solid Ratio of 10 l/kg for Materials with Particle Size below 4 mm. AFNOR—French Standard Institute: La Plaine Saint-Denis, France, 2002.
58. Cardinaud, G.; Rozière, E.; Martinage, O.; Loukili, A.; Barnes-Davin, L.; Paris, M.; Deneele, D. Calcined clay—Limestone cements: Hydration processes with high and low-grade kaolinite clays. *Constr. Build. Mater.* **2021**, *277*, 122271. [\[CrossRef\]](#)
59. Gesoğlu, M.; Güneyisi, E.; Özbay, E. Properties of self-compacting concretes made with binary, ternary, and quaternary cementitious blends of fly ash, blast furnace slag, and silica fume. *Constr. Build. Mater.* **2009**, *23*, 1847–1854. [\[CrossRef\]](#)
60. Chen, J.J.; Ng, P.L.; Chu, S.H.; Guan, G.X.; Kwan, A.K.H. Ternary blending with metakaolin and silica fume to improve packing density and performance of binder paste. *Constr. Build. Mater.* **2020**, *252*, 119031. [\[CrossRef\]](#)
61. Afzali-Naniz, O.; Mazloom, M. Assessment of the influence of micro- and nano-silica on the behavior of self-compacting lightweight concrete using full factorial design. *Asian J. Civ. Eng.* **2019**, *20*, 57–70. [\[CrossRef\]](#)
62. De Weerd, K.; Ben Haha, M.; Le Saout, G.; Kjellsen, K.O.; Justnes, H.; Lothenbach, B. Hydration mechanisms of ternary Portland cements containing limestone powder and fly ash. *Cem. Concr. Res.* **2011**, *41*, 279–291. [\[CrossRef\]](#)
63. Higl, J.; Hinder, D.; Rathgeber, C.; Ramming, B.; Lindén, M. Detailed in situ ATR-FTIR spectroscopy study of the early stages of C-S-H formation during hydration of monoclinic C3S. *Cem. Concr. Res.* **2021**, *142*, 106367. [\[CrossRef\]](#)
64. Lu, L.; Xiang, C.; He, Y.; Wang, F.; Hu, S. Early hydration of C3S in the presence of Cd²⁺, Pb²⁺ and Cr³⁺ and the immobilization of heavy metals in pastes. *Constr. Build. Mater.* **2017**, *152*, 923–932. [\[CrossRef\]](#)
65. Zunino, F.; Scrivener, K. Factors influencing the sulfate balance in pure phase C3S/C3A systems. *Cem. Concr. Res.* **2020**, *133*, 106085. [\[CrossRef\]](#)
66. Zhao, Y.; Gao, J.; Liu, C.; Chen, X.; Xu, Z. The particle-size effect of waste clay brick powder on its pozzolanic activity and properties of blended cement. *J. Clean. Prod.* **2020**, *242*, 118521. [\[CrossRef\]](#)
67. Jacquemot, F. Accélération du Durcissement des Liants à Base de Laitier de Haut Fourneau pour les Produits Préfabriqués en Béton. Ph.D. Thesis, Université Lille 1, Lille, France, 2014. (In French)
68. Cyr, M.; Lawrence, P.; Ringot, E. Efficiency of mineral admixtures in mortars: Quantification of the physical and chemical effects of fine admixtures in relation with compressive strength. *Cem. Concr. Res.* **2006**, *36*, 264–277. [\[CrossRef\]](#)
69. Zairi, M.; Rouis, M.J. The influence of particle size on the pozzolanic reactivity of quartz powders. *Bull. Lab. Ponts Chaussees* **1999**, *219*, 17.
70. Skibsted, J.; Snellings, R. Reactivity of supplementary cementitious materials (SCMs) in cement blends. *Cem. Concr. Res.* **2019**, *124*, 105799. [\[CrossRef\]](#)
71. Nedunuri, S.S.S.A.; Sertse, S.G.; Muhammad, S. Microstructural study of Portland cement partially replaced with fly ash, ground granulated blast furnace slag and silica fume as determined by pozzolanic activity. *Constr. Build. Mater.* **2020**, *238*, 117561. [\[CrossRef\]](#)
72. Quang, N.D.V.; Bazhenov, Y.M.; Aleksandrova, O.V. *Effect of Quartz Powder and Mineral Admixtures on the Properties of High-Performance Concrete*; Vestnik MGSU Moscow State University of Civil Engineering: Moscow, Russia, 2019. [\[CrossRef\]](#)
73. Li, C.; Sun, H.; Li, L. A review: The comparison between alkali-activated slag (Si + Ca) and metakaolin (Si + Al) cements. *Cem. Concr. Res.* **2010**, *40*, 1341–1349. [\[CrossRef\]](#)
74. Michel, M. Accélération de Ciment au Laitier par du Ciment Sulfo-Alumineux. Ph.D. Thesis, INSA Lyon, Villeurbanne, France, 2009. (In French)
75. Bordy, A. Influence des Conditions Thermo-Hydriques de Conservation sur l'hydratation de Matériaux Cimentaires à Base d'une Fine Recyclée. Ph.D. Thesis, Université de Cergy-Pontoise, Neuville-sur-Oise, France, 2016. (In French)
76. Zingg, L. Influence de la Porosité et du Degré d'humidité Interne sur le Comportement Triaxial du Béton. Ph.D. Thesis, Université de Grenoble, Saint-Martin-d'Hères, France, 2013. (In French)
77. Bajja, Z. Influence de la Microstructure Sur le Transport Diffusif des Pâtes, Mortiers et Bétons à Base de CEM I Avec Ajout de Fumée de Silice. Ph.D. Thesis, Université Paris-Saclay, Gif-sur-Yvette, 2007. (In French)
78. Bur, N. Étude des Caractéristiques Physico-Chimiques de Nouveaux Bétons Eco-Respectueux pour leur Résistance à L'environnement Dans le Cadre du Développement Durable. Ph.D. Thesis, Université de Strasbourg, Strasbourg, France, 2013. (In French)

79. Plassais, A. Nanoporosité, Texture et Propriétés Mécaniques de Pâtes de Ciments. Ph.D. Thesis, Université Paris 6, Paris, France, 2003. (In French)
80. Ministère de L'écologie, du Développement durable et de L'énergie. *Arrêté du 12 Décembre 2014 Relatif Aux Conditions D'admission Des Déchets Inertes Dans Les Installations Relevant Des Rubriques, 2515, 2516, 2517 et Dans Les Installations De Stockage De Déchets Inertes De La Rubrique 2760 De La Nomenclature Des Installations Classées*; JORF n° 0289 du 14 décembre 2014, France, 2014; Ministère de L'écologie, du Développement durable et de L'énergie: Paris, France, 2014. Available online: <https://www.legifrance.gouv.fr/eli/arrete/2014/12/12/DEVP1412523A/jo/texte> (accessed on 30 November 2018). (In French)

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