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Effect of flash-calcined sediment substitution in sulfoaluminate cement mortar

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Calcium sulfoaluminate cement is a relatively new type of cement with environmental advantages linked to its low carbon emission. However, there is a lack of knowledge on its use with supplementary cementitious materials. This article aims to study the effect of utilizing the activated flash-calcined sediment from Noyelles-sous-Lens (SC) as a supplementary cementitious material in calcium sulfoaluminate mortar. Therefore, four mixes are prepared by replacing calcium sulfoaluminate cement with 5%, 10%, and 20% of flash-calcined sediment. The reactivity of the cement pastes with and without the sediment is studied by isothermal calorimetry and by measuring the setting time. Then, the mechanical performance is tested at 1, 7, and 28 days. The results show that adding the flash-calcined sediment has an accelerator effect on cement hydration and decreases the induction period. The cumulative heat release and the compressive strength at 28 days are almost the same up to 10% of sediment substitution.

KEYWORDS

calcium sulfoaluminate cement, sediment, mortar, recycling, flash calcination

Introduction

The cement industry emits more than 5% of the total man-made global CO₂ emissions (Gartner and Hirao, 2015). Therefore, after the Kyoto Protocol (1997), industries were constrained to find durable solutions to reduce carbon emissions (Shi et al., 2011; Ghezloun et al., 2017; Scrivener et al., 2018). Recently, the International Conference on Climate Change (COP 25, 2019) was held in Madrid and has tried to regulate the global carbon market, climate change, global temperature increase, and water supply problems. For this purpose, several approaches have been envisaged in the construction sectors of supplementary cementitious materials (SCMs) as a substitution for ordinary Portland cement (OPC). Some solutions have been tried to use alternative fuels rather than those commonly used for material combustion in cement kilns and also the reduction of decarbonization by the replacement of some raw materials (such as calcium carbonate) by other products—already decarbonated—that confer the same chemical components. Another solution has also been considered, which consists in elaborating new types of

cement, like calcium sulfoaluminate (CSA) cement, as an interesting alternative to Portland cement. CSA cement is part of the “Third Cement Series” (TCS) developed in China in the early 90s and is increasingly used in several types of applications where early high strength or shrinkage control is needed. It was also used by Kleib et al. (2018) to mitigate alkali-silica reactions in mortars. The CSA cement, also known as “green cement” is environmentally friendly due to its low-carbon footprint, and its production consumes less energy than the OPC cement (Tang et al., 2015). In fact, the clinkerization temperature of the CSA clinker is 200–300°C (Gartner and Hirao, 2015; Gartner, 2004) less than that of the Portland clinker. This calcination temperature difference represents a 25%–35% reduction in carbon dioxide emission (Hanein et al., 2018). In addition, the CSA clinker is friable, reducing the energy consumption needed for grinding compared to OPC cement, and the amount of limestone required for the CSA clinker production is lower than that for OPC clinker production, consequently reducing carbon emissions related to the calcination process (Quillin, 2001; Zhang et al., 2012; Berger et al., 2013; Su et al., 2019). The main phase in the CSA clinker is ye’elite, also called Klein’s compound $C_4A_3\dot{S}$, in addition to other secondary phases such as C_2S , C_4AF , and $C\dot{S}$.

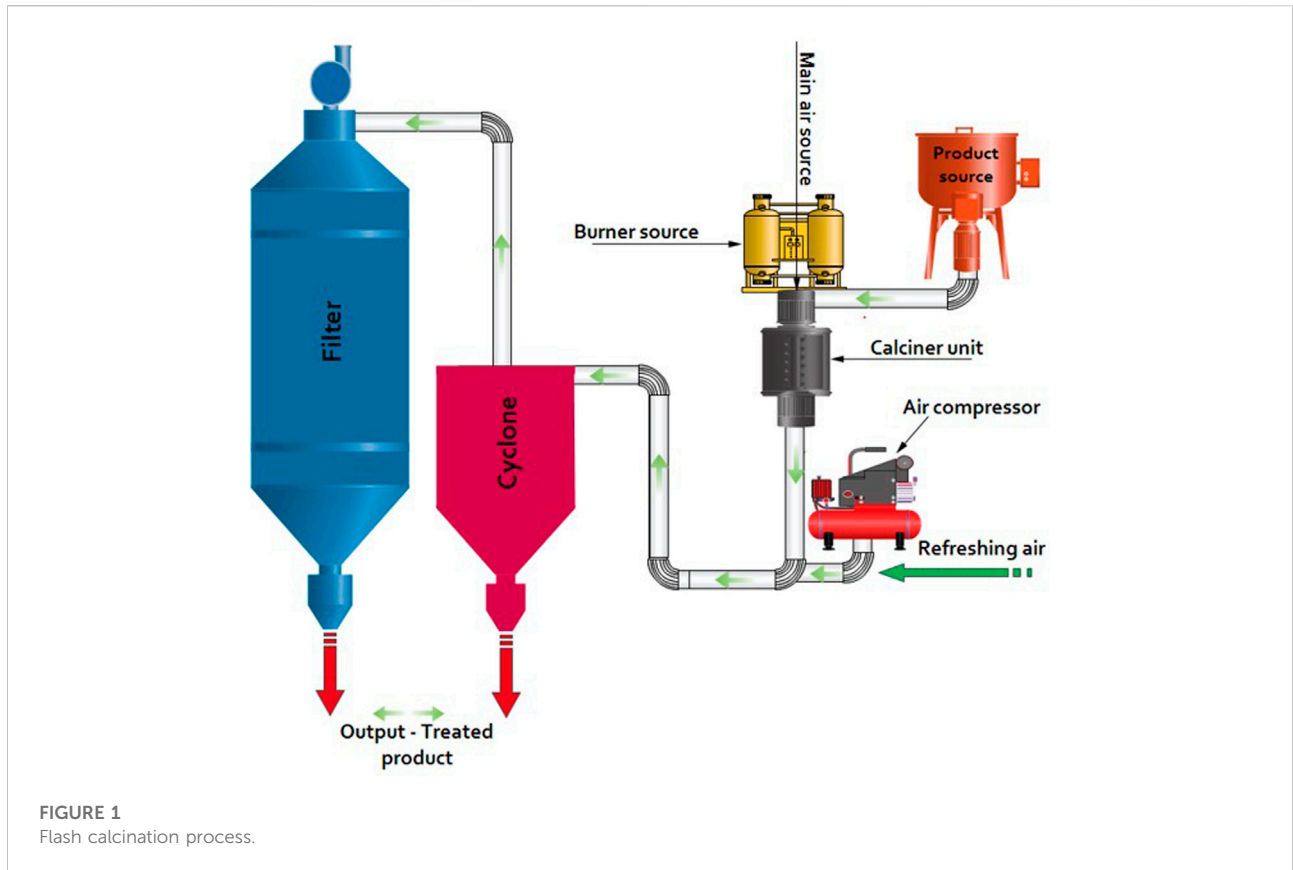
Several studies have been conducted to study the effects of SCM when the CSA cement is used. García-Maté et al. (2013) showed that the partial substitution of the CSA cement with fly ash (FA) has environmental and economic benefits. The authors showed that adding FA up to 15% increases mechanical performance; however, an excessive amount has a negative effect on these properties. The same result was obtained by Martin et al. (2017) who showed that the increase in the substitution by FA accelerates the hydration of the CSA cement and that adding up to 15% of FA does not have any effect on the compressive strength. The galvanic sludge has also been used by Luz et al. (2009) to substitute CSA cement by 25% in mortars. The results showed that the CSA cement hydration products encapsulate the chromium coming from the sludge (Luz et al., 2009).

Regarding the sediment, it was used as SCM with OPC cement in order to be recycled and to reduce OPC cement production (Snellings et al., 2017; Zhao et al., 2018; Van Bunderen et al., 2019). In fact, sediments are materials coming from dredged marine operations and continental watercourses and can also be defined as fine materials from the wear of continents (Wang and Gillot, 1991; Dubois et al., 2011). Several studies have already been conducted to use the sediment as SCM in OPC construction materials (Snellings et al., 2017; Amar et al., 2018; Van Bunderen et al., 2019). These studies have shown the presence of a pozzolanic effect when the sediment is added as a mineral addition in the cementitious matrix (Dang et al., 2013; Snellings et al., 2016; Amar et al., 2017; Snellings et al., 2017; Van Bunderen et al., 2019). Different methods, such as physical, chemical, biological, and thermal

methods, can be used to treat sediments before using them as SCM (Amar et al., 2021).

Flash calcination is a treatment technique that has been applied in some studies for the activation of clays and to transform kaolin into metakaolin (Salvador, 1995; Salvador and Pons, 2000; Nicolas et al., 2013; Claverie et al., 2015; Inocente et al., 2021; Van Bunderen et al., 2021). Danilevich et al. (2021) have thermally activated aluminum hydroxide in gibbsite using a reactor TSEFLAR. By examining the characteristics of the final product, the study has shown that process parameters such as temperature, the residence time of the particles, and raw material consumption play a major role in activation processes. Van Bunderen et al. (2021) also studied the effect of the flash-calcined sediment on the mechanical properties and shrinkage of concretes. After testing cement replacement for 20 wt%, 30 wt%, and 40 wt %, the results demonstrated that flash-calcined sediments are suitable for the production of sustainable cement and concrete. This technique has the advantage of lowering energy consumption and gas release due to the quickness of the process, and it is applied to sediments in this study. As a matter of fact, this process includes dehydroxylation that activates the material with thermal excitation between 700 and 850°C. Activation of the clay fractions present in sediments has recently been studied (Snellings et al., 2017; Snellings et al., 2016; Scrivener et al., 2004; Teklay et al., 2016; Van Bunderen et al., 2018). These types of products are also usable as SCM with trough cement replacement (Nguyen and Castel, 2020). Amar et al. (2017) have studied the reactivity of flash-calcined sediments. By using various pozzolanic tests, it was demonstrated that the calcination process has a positive impact on the pozzolanic activity of sediments (Amar et al., 2021). Snellings et al. (2017) and Snellings et al. (2016) also used the flash calcination technique on the same sediment studied by Van Bunderen et al. (2019). This last study investigated the influence of the temperature of treatment and the final reactivity of sediments. The authors tested three calcination temperatures, 820, 865, and 905°C, and they showed the effectiveness of the process.

The use of sulfoaluminate cement is of particular interest. This cement possesses advantageous properties such as low carbon emission, fast setting, low shrinkage, and resistance to corrosion (Allevi et al., 2015; Shang et al., 2022). To help lower cement industry impact and product costs, it seems interesting to use blends of CSA and SCMs (García-Maté et al., 2013). In this approach, the benefits will come in two folds: lower CO₂ emissions because of the clinker reduction, cost reduction, and alternative material reuse. Flash-calcined materials to cement may also modify blended product properties (Qian et al., 2008), such as decreased hydration heat, improved workability of mixes, and porosity (V. Ž.-C. and building Materials and undefined, 2000). A recent study conducted by Zhang



et al. (Ke et al., 2021) monitored pore characteristics in CSA–SCM-blended cement in comparison to OPC. It was demonstrated that with the action of fly ash, silica fume, and slags, the pore structure of the CSA cement paste was remarkably refined. However, the replacement ratio was limited to 15% for each SCM, and waste materials such as SC were not utilized. Gastaldi et al. (2021) adopted a similar approach and studied the hydration behavior of CSA–SCM blends without calcined sediments. Furthermore, in the particular case of sediments blended with CSA, SC was shown to be of particular interest for the construction sector (Zentar et al., 2021) whereas it was not assessed for cementitious matrix utilization. First, the use of flash-calcined sediments as a substitution for the CSA cement has not been largely investigated. Also, the physicochemical properties, reactions involved, and microstructure in CSA–SC blends are not well understood. From an ecological and economic point of view, the combination of the CSA cement and sediment as SCM is advantageous. It leads to a lower cement content by replacing it with SC. Hence, on one hand, as already discussed, the production of CSA cement requires less energy than is needed in OPC manufacture. Also, on the other hand, the use of SCM reduces the quantity of the clinker to be produced.

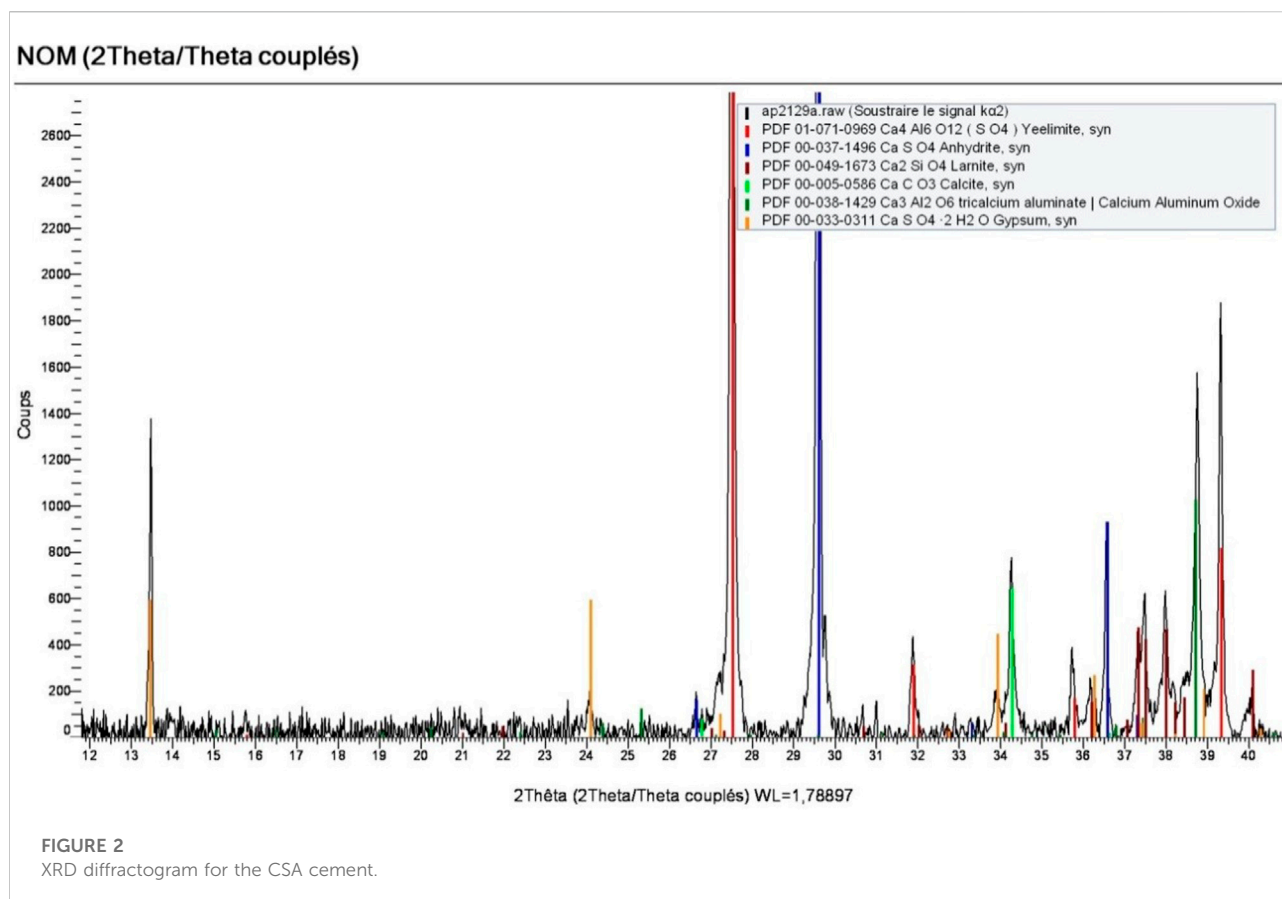
TABLE 1 Mortar composition of CSA, CSA5, CSA10, and CSA20.

Constituent/mortar	CSA	CSA5	CSA10	CSA20
Cement CSA (g)	450.0	427.5	405.0	360.0
Flash-calcined sediment (g)	0	19.6	39.2	78.8
Binder (CSA + SC) (g)	450.0	447.1	444.2	438.8
Normalized sand (g)	1,350.0	1,350.0	1,350.0	1,350.0
Water (g)	225.0	223.5	222.1	219.4
Binder/sand	0.33	0.33	0.33	0.33
Water/binder	0.50	0.50	0.50	0.50
Total volume (cm ³)	884.4	882.8	881.3	878.6

Hence, in this article, the flash-calcined sediment from Noyelles-Sous-Lens is blended with the commercial CSA cement in order to test its effect on the hydration process and the mechanical properties of mortars.

Materials and experimental program

The materials used in this study are sulfoaluminate cement (CSA)—Alpenat[®]—produced by VICAT[®] and the



fluvial sediments dredged from Noyelles-sous-Lens in the north of France. The latter was dried at 100°C, then crushed, and sieved. The sediment was treated through the flash calcination method at 750°C, as described by Amar (2017).

Calcination was carried out in a flash furnace at IMT Nord-Europe in France. The flash calcination process is illustrated in Figure 1 (Amar, 2017).

Four mortar mixes were designed and produced with and without flash-calcined sediments. Formulations were made under the NF EN 196-1 standard. Mixes were prepared following a water/binder ratio of 0.5 and fine siliceous sand (NS) in a proportion of 1:3. CSA stands for the control mortar without sediment, whereas CSA5, CSA10, and CSA20 refer to mortars containing, respectively, 5%, 10%, and 20% of flash-calcined sediments. The mortar compositions are given in Table 1.

The mineralogical composition of the CSA cement and the calcined sediment was determined by X-ray diffraction (XRD) using a Bruker D2 instrument with Cu K α radiation. The X-ray patterns were acquired in 2θ (10–80°) with a step of 0.02 and 1 s per step.

The chemical composition of the CSA cement and the flash-calcined sediment SC was determined by X-ray fluorescence (XRF) using a Bruker S4 Pioneer spectrometer.

The particle size distributions of the materials (cement, sediment, and sand) were determined using a Coulter laser granulometry type LS 13 320.

The heat flow was measured using an isothermal calorimeter at 20°C to study the reactivity of the cement paste with a water/cement ratio of 0.5. The calorimeter used is a homemade calorimeter equipped with flowmeters, allowing the calorimeter to equilibrate in less than 5 min.

The initial setting time of the cement pastes was determined according to the standard NF EN 196-3 and using the VICATRONIC I06 091 device.

The compressive strength of mortars was determined according to the standard AFNOR NF EN 196-1 on prismatic samples 40*40*160 mm. The mechanical properties were measured at 1, 7, and 28 days using the INSTRON 5500 R apparatus.

The porosity was also investigated on specimens at 28 days with the mercury porosimeter MicroActive AutoPore V9600 2.00.

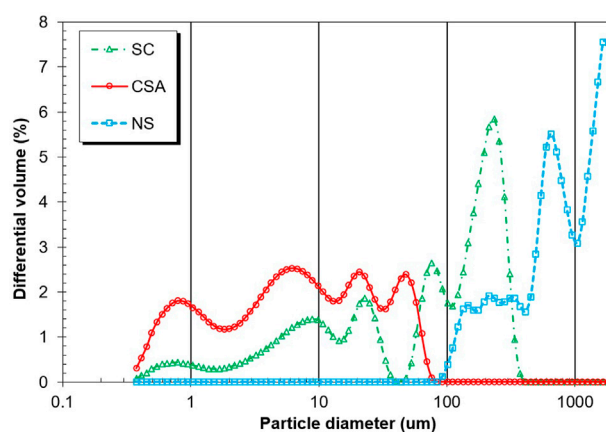


FIGURE 3
XRD diffractograms of raw and flash-calcined sediments.

TABLE 2 Concentrations of the major oxide elements for the CSA cement Alpenat from VICAT and the flash-calcined sediment.

Oxide (%)	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	SO ₃	K ₂ O	CaO	TiO ₂	MgO	Na ₂ O	P ₂ O ₅	ZnO	LOI
CSA	19	8.7	7.9	14.4	0.3	44.2	1	1.3	-	-	-	3.2
SC	12	49.7	5.3	0.2	2.2	11.2	-	1.1	0.7	2.3	0.3	13.7

LOI, loss of ignition.

Results and discussion

Chemical and mineralogical characterization

The chemical and mineralogical analyses consist in determining the presence and the proportion of each chemical element and the mineralogical phases by XRF and XRD, respectively. The results show that the CSA cement contains ye'elinite, belite, anhydrite, and perovskite as major phases (Figure 2). Figure 3 shows the mineral composition of the raw sediment RS (before treatment) and SC sediment (after flash calcination). The main mineral phases identified are quartz, calcite, albite, and mica for RS and SC. The main difference is in the amorphous phases generated, as demonstrated earlier by Amar et al. (2017).

Physical properties

Table 2 presents the chemical composition of the CSA cement and SC. The main oxides in both materials are CaO, Al₂O₃, SiO₂, and Fe₂O₃ in addition to SO₃ in the CSA cement.

The physical properties of materials are shown in Table 3. The results show that the SC is a fine material, and this could impact the activity and water demand of the material.

The particle size distribution determined by laser granulometry shows that the CSA cement and SC sediment have approximately the same D50 (7.23 and 7.22 μm for CSA and SC, respectively). Therefore, partial substitution of cement by the flash-calcined sediment is possible. The particle size distribution is given in Figure 4.

Calorimetry

The reactivity of cement pastes, with and without the sediment, was followed by isothermal calorimetry at 20°C. The hydration of the cement paste is an exothermic process; therefore, the heat released can be correlated with the kinetics of cement hydration.

Figure 5 presents the heat flow released and cumulative heat released for the four cement pastes: CSA, CSA5, CSA10, and CSA20. The results show that the calorimetric curves for cement pastes with and without flash-calcined sediments have the same shape. Three peaks are identified. The first peak is formed after the cement's contact with water and the dissolution of the

TABLE 3 Physical properties of the flash-calcined sediment SC and CSA.

Material	Density (g/cm ³)	Blaine-specific surface (cm ² /g)	Bet-specific surface (cm ² /g)
CSA	3.0	4,500	14,357
SC	2.65	5,260	15,590

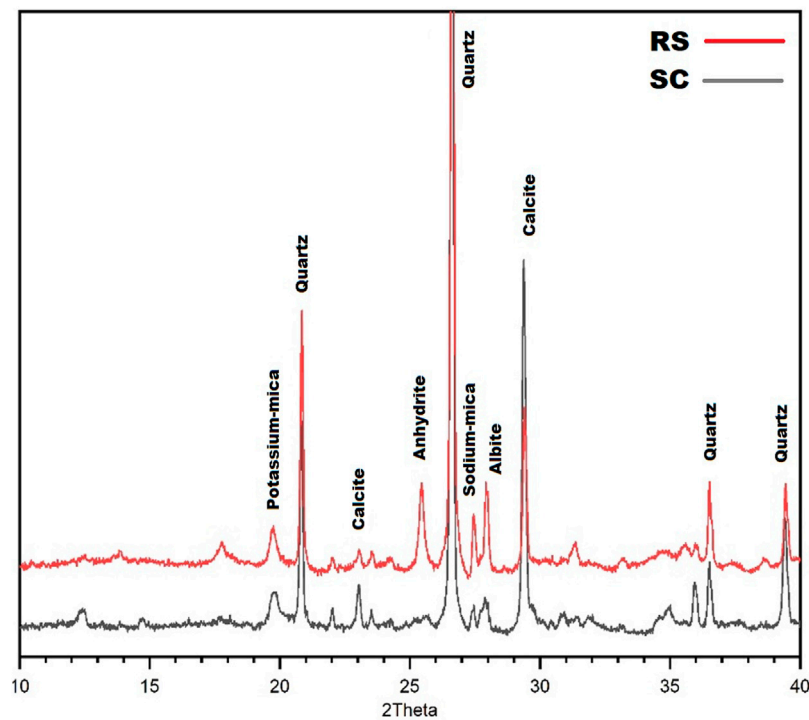


FIGURE 4
Particle size distribution for the CSA cement, SC, and normalized sand NS.

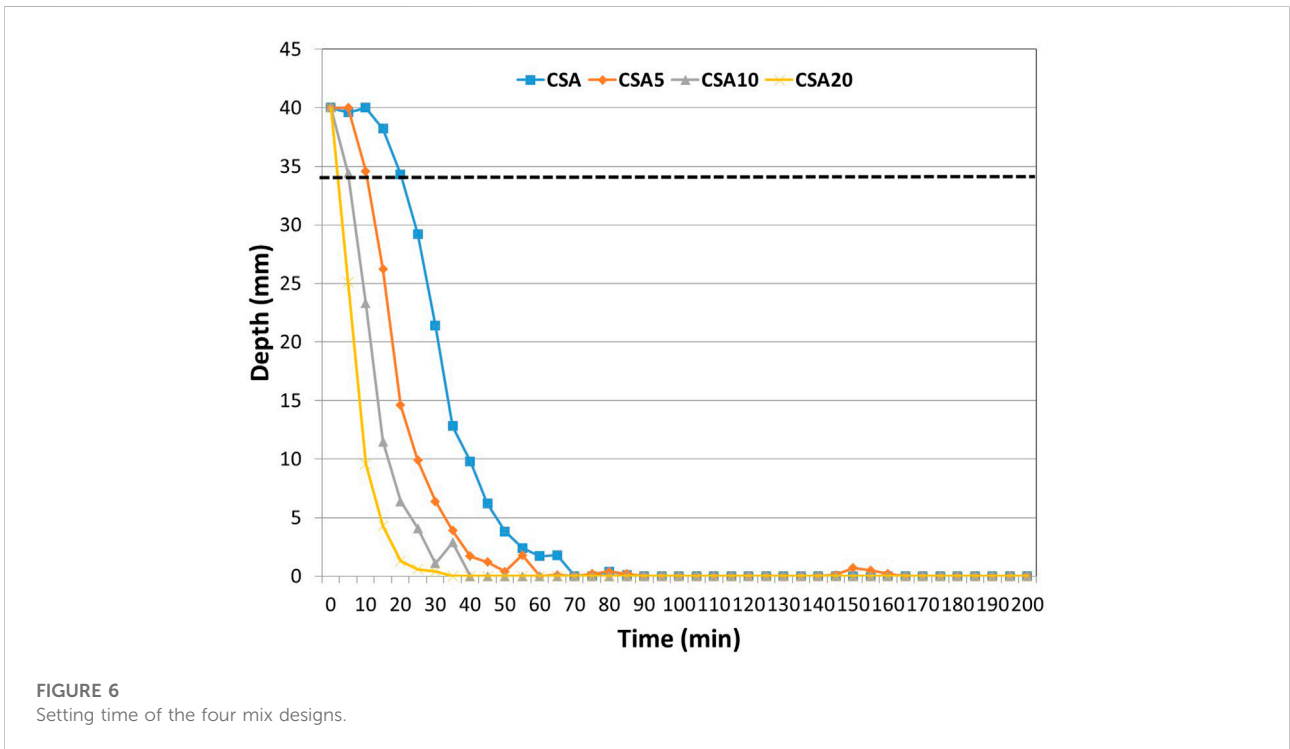
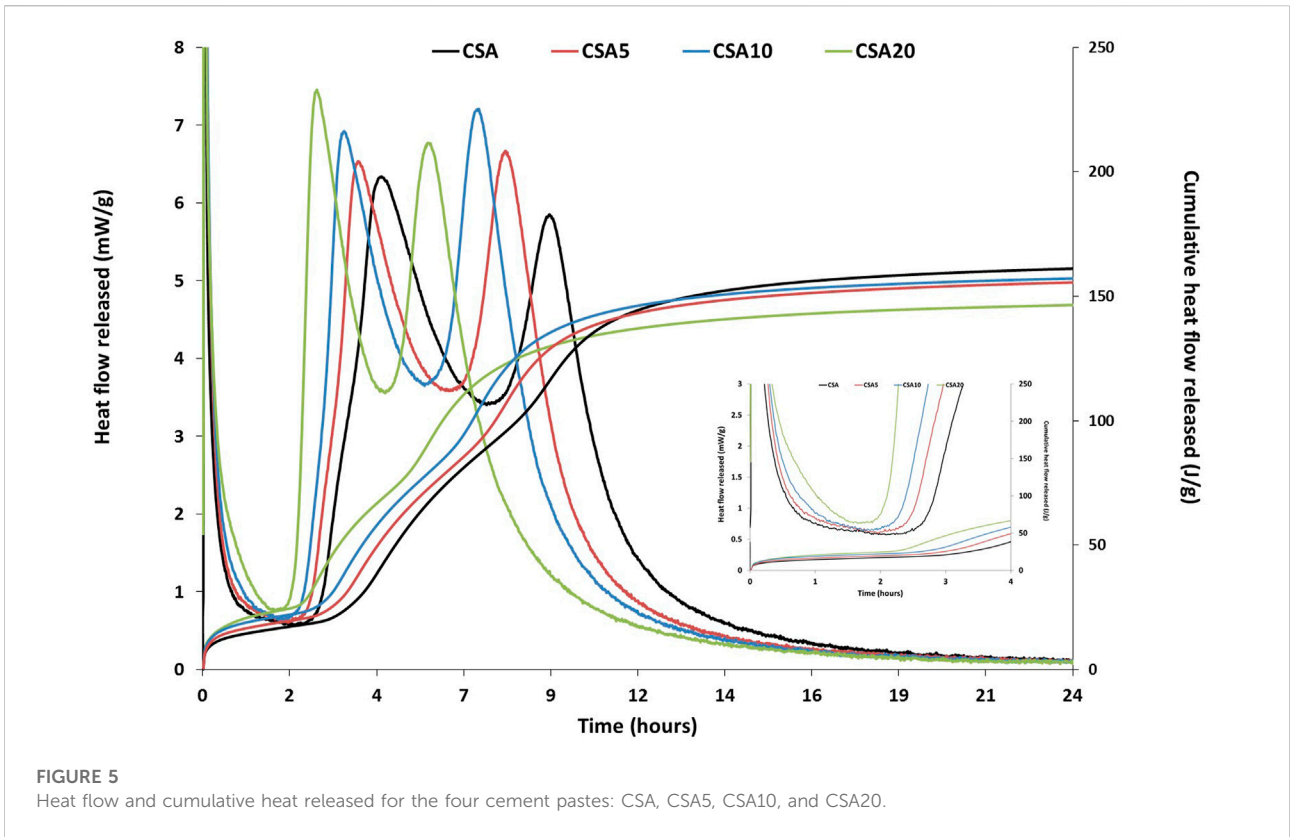
anhydrous grains within the first few minutes of hydration. After the first maximum, an induction period with low thermal activity is observed. Then, a second peak is formed for all of the mixes and corresponds to the reaction between ye'elinite and the sulfates coming from the anhydrite/gypsum. This reaction gives ettringite $C_3A \cdot 3C\check{S} \cdot H_{32}$ as the main hydration product. Then, after depletion of the sulfate sources, a third peak is formed and the decomposition of ettringite will occur, leading to the production of the monosulfoaluminate hydrate. The same hydration mechanisms and isothermal curves were obtained in the literature, indicating the formation of three peaks during CSA cement paste hydration (Winnefeld and Barlag, 2009; Chen and Juenger, 2011; Wang et al., 2022; Žibret et al., 2022).

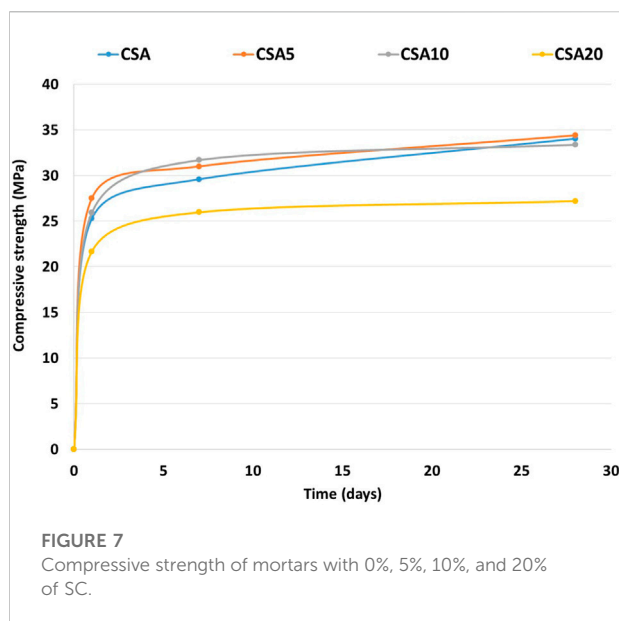
When comparing the heat released from the CSA cement paste (without the SC sediment) to cement pastes with sediments (CSA5, CSA10, and CSA10), it can be seen that the peaks are

shifted to the left when adding the flash-calcined sediment in the mortar as supplementary cementitious materials, indicating that the reactions are accelerated. This acceleration increases with the increase in the sediment amount in the cement paste. The initial setting time for CSA begins after 3 h of hydration, whereas for CSA5, CSA10, and CSA20, it begins after 2 h 45 min, 2 h 30 min, and 2 h 15 min of hydration, respectively.

Concerning the cumulative heat released from the cement pastes, the results show that at the beginning of the hydration process and until approximately 8 h of hydration, the cumulative heat released increases with the amount of the flash-calcined sediment in the mortar. However, after 24 h of hydration, the cumulative heat released from the CSA cement paste is the highest, and the CSA20 has the lowest cumulative heat released.

Different parameters could influence the heat release of the CSA cement according to the literature. Burriss and Kurtis (2022)





showed that the water/cement ratio (W/C) and the cement composition (anhydrite/ye'elinite content in the cement) have an influence on the hydration mechanisms (retarder or accelerator effect depending on the ye'elinite content in the clinker).

In our case, the acceleration effect and the reduction in the induction period when adding the flash-calcined sediment to the cement paste could be related to the additional quantity of anhydrite brought by the flash-calcined sediment. In fact, as it can be seen from the mineralogical analysis (Figure 2) of the sediment before and after treatment, flash calcination leads to the formation of anhydrite in the flash-calcined sediment compared to the raw sediment (RS) before treatment. Similar results were established in a previous study conducted by Chu et al. (2022). Therefore, more sulfates are available to react with ye'elinite when adding more SC to the cement paste, and the hydration mechanisms are accelerated. Concerning the cumulative heat release, in the first 8 h, the cement paste containing the sediment released more heat than the CSA cement paste due to the acceleration of ye'elinite hydration. However, after 24 h, the total heat released is lower for the sediment-based cement paste, especially for CSA20, due to the dilution effect. Therefore, there is less ye'elinite in the mixture to react.

Setting time

The setting time results are presented in Figure 6. The initial setting time is determined when the needle penetrates the mixture to a depth of 34 ± 1 mm.

The results show that the initial setting time is reached faster when the mortar contains the flash-calcined sediment. This time

decreases with the increase in sediment substitution in the cement paste. The same behavior is observed for the final setting time. The latter is shorter when adding the sediment to the cement paste.

As for the calorimetric study, many factors could influence the setting time of the cement paste, such as the temperature, W/C ratio, and cement composition (Burris and Kurtis, 2022).

Therefore, the faster setting time with sediment cement pastes could be due to the additional anhydrite brought by the flash-calcined sediment as CSA reactions rely on anhydrite availability.

Mechanical performances and porosity

The compressive strength results at 1, 7, and 28 days are presented in Figure 7 for the different mix designs.

The results show that up to 10% substitution with SC, the compressive strength of mortars is slightly higher than the reference mortar CSA without SC, especially at 1 and 7 days. At 28 days, the three mortars (CSA, CSA5, and CSA10) present almost the same compressive strength of around 34 MPa, whereas for the mix with 20% of SC substitution, the compressive strength is the lowest compared to the three other mortars and at all testing times. In addition, in 1 day, almost 80% of the compressive strength for all of the mix designs is reached.

The decrease in the compressive strength for CSA20 could be related, on one hand, to the dilution effect, which is dependent on the high cement substitution by the sediment. Therefore, the ye'elinite amount decreases considerably in the mix, and consequently, the ettringite formation decreases. These low properties are also related to the high water demand of the sediment, which increases porosity during hardening. The mechanical strength results confirm the results obtained by isothermal calorimetry where CSA20 exhibits the lowest cumulative heat released (see Section 3.2) and the lowest compressive strength. The total porosity of the mortars was measured after 28 days of hydration (Table 4). The results show that CSA20 has the highest porosity among the four designed mortars. As porosity is correlated with resistance, mortars with lower porosity present better compressive strength (CSA, CSA5, and CSA10, respectively).

Hence, it could be considered that the presence of the calcined sediment has no significant negative effect on matrix properties for a substitution rate below 10%.

Activity monitoring

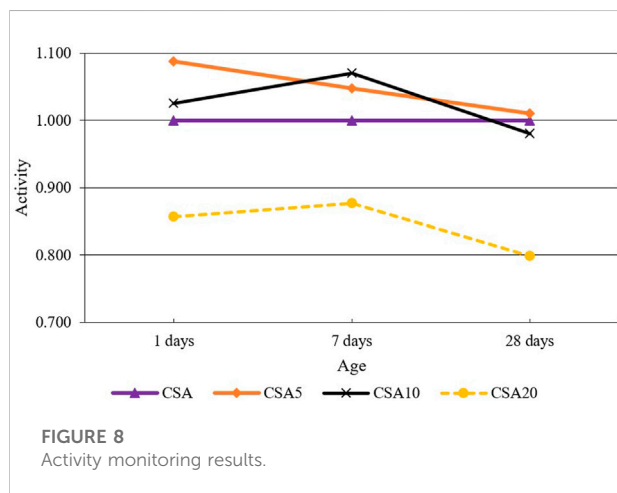
The pozzolanic activity of the formulated mortars was studied using the mechanical activity index i and activity coefficient χ_A . The compressive strength results at 28 days are

TABLE 4 Mercury porosity, average pore diameter, and total pore area on the mortar tested at 28 days.

Characteristic	Age (days)	CSA	CSA5	CSA10	CSA20
Porosity (%)	28	13.922	13.35	13.13	14.80
Average pore diameter (4V/A) (μm)		0.0277	0.0248	0.0258	0.0268
Total pore area (m^2/g)		7.462	7.235	8.537	9.971

TABLE 5 Activity index and activity coefficient calculated at 28 days.

Mortar	CSA	CSA5	CSA10	CSA20	
Age	28 days				
Strength (MPa)	34.04	34.40	33.37	27.19	
p (%)	0	5	10	20	
E (g)	225	223.5	222.1	219.3	
Binder	450	447.1	444.2	438.8	
A (gap in %)	0.0%	1.1%	-2.0%	-20.1%	
$i(t)$	1.000	1.011	0.980	0.799	
χ_A	Amar et al. (2017)	100.00%	100.14%	99.87%	99.30%
	Rozière et al. (2015)	100.00%	100.16%	99.85%	99.24%
	De Larrard (2000)	106.76%	109.78%	101.24%	53.40%



used to calculate i and χ_A . The results are summarized in Table 5 where p is the substitution rate; E is the water content, and α is the relative strength gap between the substituted mortar and the control mortar CSA. The formulas used for activity coefficient determination were previously established by Amar et al. (2017), Rozière et al. (2015), and De Larrard (2000).

The mineralogical and chemical composition of the sediment (siliceous, clayey, and limestone) can be the reason why it can be used as a suitable supplementary cementitious admixture. In multiple cases, an activation process similar to that applied to

clay (Gastaldini et al., 2015) must be used to enhance the sediment's properties. Sediments have also been shown to be effective as a potential mineral addition in concretes and mortars (Chen et al., 2010; ANGER et al., 2014; Bouamrane et al., 2014). The results presented in Table 5 and Figure 8 provide evidence that processes are different and SC has a low pozzolanic activity at an early age. However, CSA5 at 28 days has a better activity ($i = 1.01$ and $\chi_A = 1.09$). These results can also be explained by the fact that the pozzolanic activity generally needs portlandite (Ca(OH)_2) to be operated, which is not (or is slightly) produced in a CSA mix. To improve general properties, it can be used with alkalis that conduct greater ye'elime consumption and ettringite formation (Tambara et al., 2020).

In addition, multiple parameters such as fineness, heat treatment temperature, and calcination time may also impact directly and control the final product efficiency and pozzolanic activity (Gastaldini et al., 2015; Walker and Pavia, 2011; Zhang, 2013; Lynn et al., 2015). Therefore, a better investigation of these could be interesting and helpful in understanding the involved physicochemical processes.

Investigations have shown the benefits of the use of the flash-calcined sediment in CSA blends. The presence of SC generates an acceleration of reactions involved during hydration. This effect is attributed to the anhydrite identified in the XRD analysis. The same reason also impacts the setting. The compressive strength test demonstrated that below 10% replacement of CSA by SC, no negative effect has been

identified as the mercury porosity test has also shown a similar pore structure between CSA and CSA10.

Conclusion

This work aims to study the effect of the substitution of the calcium sulfoaluminate cement by the flash-calcined sediment from Noyelles-sous-Lens in France. The main conclusions that can be drawn throughout this work are the following.

- 1) Isothermal calorimetry shows that adding the flash-calcined sediment has an acceleration effect on cement paste hydration. The presence of anhydrite in the sediment after treatment could be the reason for this acceleration. This result is consistent with the setting time test.
- 2) The cumulative heat release after 24 h of hydration is the same for cement pastes containing up to 10% of flash-calcined sediments in the mixture. Adding 20% sediment to the cement paste decreases the total heat released at 24 h, and this could be due to the dilution effect and the decrease in the ye'elinite content and consequently in ettringite formation.
- 3) The compressive strength results show, as for the calorimetry, that adding up to 10% of the sediment in the mortar does not influence the mechanical performances at 28 days.

This study shows that the flash-calcined sediment could be recycled, up to 10%, as supplementary cementitious materials in a CSA cement matrix without affecting the performance of the cementitious material. It would be interesting to continue this study in order to evaluate the effect of this substitution on the durability of the cement matrix when incorporating sediments.

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Data availability statement

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

Author contributions

JK, MA, MB, and N-EA contributed to the methodology and reviewing and editing of the study. JK and MA contributed to the investigation of the study and to the experimental program. JK and MA wrote the first draft of the manuscript. All authors contributed to the manuscript revision and read and approved the submitted version.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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