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1 **Developing mortar using limestone flash-calcined dredged sediment/ millstone-clay cement binder (LFC)**

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5 **Abstract**

6 This study aims to develop a sustainable cement binder that resembles limestone calcined clay cement by
7 examining the effects of flash calcination on the physical and mineralogical properties of materials, and their
8 potential as pozzolanic material. The study will evaluate whether flash-calcined material, made from marine
9 sediments or excavated clayey soils at 750°C, can be a substitute for metakaolin (MK), commonly used to
10 enhance cementitious formulations. The researchers aim to determine whether the flash-calcined material can
11 achieve similar properties to MK when developing mortar made of limestone flash-calcined sediment/millstone-
12 clay cement (LFC). The study shows that flash calcination improves pozzolanic reactivity by enhancing BET
13 surface area and density without altering chemical characteristics. The Chapelle test confirms pozzolanic
14 reactivity in the flash-calcined sediment (FCS) and flash-calcined millstone clay (FCMC), and physical,
15 mechanical, and microstructural tests are conducted to analyze their impact on various formulations of mortar and
16 paste samples. The results suggest that LFC with MK, FCS, and FCMC can achieve better strength than the
17 reference mix C0. Microstructural tests demonstrate that mortar designed with FCS and FCMC have similar
18 compositions to that with MK.

19 **Keywords:** Supplementary cementitious material, sediments, excavated clays, flash calcination.

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1 1. Introduction

2 The construction industry uses a lot of natural resources to make ordinary Portland cement (OPC), which can
3 harm the environment. Construction activities also release large quantities of greenhouse gases [1][2], and the
4 civil engineering sector contributes to this problem through the materials, techniques, and resources it uses. The
5 demand for cement has increased, leading to more CO₂ emissions from the process of burning raw materials to
6 make clinker [3][4]. However, using supplementary cementitious materials (SCMs) to replace some OPC can
7 reduce cement production and therefore decrease CO₂ emissions [5][6][7]. This substitution can also lead to
8 increased sustainability, strength, and long-term durability [8].

9 Another environmental concern is a large number of waste materials, such as dredged sediments and
10 construction waste. These sediments, which are fine, contaminated, and highly organic, are excavated from
11 marine ports and harbors [9]. In France alone, over 56 million m³ of sediment are dredged annually
12 [10][11][12][13], with even larger quantities in China and the USA [9][10]. However, these sediments can be used
13 in SCMs, particularly if they are thermally treated. A study by Aouad et al. [14] showed that sediments can replace
14 a portion of raw materials in OPC production. Flash calcination is an important method of treating sediments for
15 use as SCMs. Snellings et al. [15] found that the pozzolanic activity of dredged sediment, which is inferior to MK
16 but superior to siliceous fly ash, can be enhanced by flash calcination. Unlike cement, most SCMs do not exhibit
17 significant hydraulic reactions when mixed with water. However, SCMs with pozzolanic properties can form
18 hydration products similar to those in Portland cement systems [8]. The pozzolanic activity of SCMs is the
19 chemical reaction that consumes Ca(OH)₂ [8], affecting the mixture's kinetic reaction.

20 The waste produced from construction activities encompasses various materials such as concrete, ceramics,
21 asphalt, wood, paper, glass, and excavated soil [16]. The large quantities of excavated soil generated each year,
22 such as the 130 million tons produced in France [17], are also considered waste due to the disposal methods
23 required. A study has shown that calcined clay at 900°C can provide the same compressive strength as blast
24 furnace slag and pulverized fuel ash in concrete formulations [18]. Another study by Yanguatin et al. [19]
25 suggested that excavated clay with a kaolinite content of 20% to 30% can be used as SCMs when calcined at
26 either 550°C for 1 hour or 650°C for 3 hours. The authors further investigated the effect of thermal treatment on
27 pozzolanic activity using calcium hydroxide fixation, the Chappelle test, and compressive strength [19]. Their
28 findings suggested that calcined excavated clays can serve as SCMs, and the pozzolanic reaction significantly
29 contributes to the increase in compressive strength [19].

30 Calcination is a heat treatment process that is commonly used to remove organic matter, eliminate pollutants,
31 and completely evaporate the water present in granular materials, including free water [9][20]. It is also widely
32 used to transform clay minerals into SCMs by triggering pozzolanic activity through the transformation of kaolinite
33 into metakaolinite [21]. However, the temperature range for calcination can vary widely, from 500 °C to 1000 °C,
34 depending on the intended use. The temperature must be high enough to destroy the structure of clay minerals
35 but low enough to avoid recrystallization and the formation of inert phases [22]. Flash calcination is a more
36 efficient and innovative technique that consumes less energy, eliminates organic matter, and activates sediment
37 phases, including clay phases, resulting in the attainment of pozzolanic properties [23]. It is a heat treatment
38 process used for finely crushed materials under high temperatures, with a temperature range of 700 °C to 1000
39 °C. Flash calcination takes only a few seconds to manufacture metakaolin (MK), and it can be as effective or even
40 better than standard MK obtained by soak-calcination [24].

41 An example of this is the study conducted by Bunderen et al. [25], which focused on investigating the
42 pozzolanic reactivity of flash-calcined sediments and its impact on the durability of concrete when used as SCM.
43 The findings of the study concluded that flash calcination had a positive influence on pozzolanic reactivity, leading
44 to reduced shrinkage in cement blends and mitigating the risk of early-age cracking in concrete. In another study
45 by Bunderen et al. [26], the researchers examined the substitution of cement with 20%, 30%, and 40% of flash-
46 calcined sediments and its effects on the mechanical and durability properties of concrete. The results indicated
47 that the concrete formulations exhibited higher compressive strength after 28 days, along with reduced
48 autogenous and total shrinkage. Additionally, AMAR et al. [27] conducted a study that demonstrated an increase
49 in the compressive strength of mortar after 28 days when utilizing a 5% replacement of flash-calcined sediments
50 through flash calcination. Another study by Nicolas et al. [28] focused on the performance and durability of
51 concrete incorporating MK as a substitute for OPC. The findings of this study revealed lower permeability,
52 increased strength, and reduced chloride penetration in the concrete. Furthermore, San Nicolas et al. [28]
53 explored the durability of concrete containing flash-calcined MK as a replacement for OPC. The study indicated
54 that the higher mechanical strength observed in the concrete was primarily attributed to the pozzolanic reactivity
55 of flash-calcined MK.

56 Therefore, flash calcination with a temperature of 750 °C is used in this study as a low-energy heat treatment
57 technique applied to dredged sediments and excavated clay soils to produce SCMs that can replace MK.

1 Metakaolin is a highly reactive pozzolanic material that is produced by calcining kaolin clay at high
2 temperatures of 700-800 °C [29]. It is widely used as a SCMs blended with OPC and in geopolymers [30].
3 Metakaolin can significantly improve the strength and workability of concrete [31], while also reducing its carbon
4 footprint and environmental impact. This material has gained increasing attention in recent years as a sustainable
5 and cost-effective solution for improving the performance of concrete structures. Its use has been studied
6 extensively in various applications mentioned by Meraz et al. [32]. For example, Sfikas et al. [33] studied the
7 effect of MK on self-compacting concrete. Another study by Kadri et al. [34] has mentioned the positive effect of
8 MK on concrete strength due to its pozzolanic reaction, promotion of OPC hydration, and filler effect.

9 Similar to MK, LS also can be used to replace OPC by 15-20% [35][36]. When finely ground limestone is
10 added to cement, it reacts with water to form calcium silicate hydrates, which contribute to the strength [36][34],
11 [35], and durability of concrete [36][36], [37]. The blending of LS, calcined clay, and clinker will form LC3 known
12 as ternary cement which is widely available to reduce clinker content [41]. Additionally, this can enhance
13 workability [41][42] and can reach the same strength as OPC by replacing up to 40-50% of clinker [41][43].

14 The utilization of calcined clays and limestone in the production of a binding agent named limestone calcined
15 clay cement (LC3) is a sustainable approach used in different research studies [44]–[47]. LC3 is considered
16 environmentally friendly as it contains a high quantity of clays and limestone, making it a feasible choice [48][45].
17 Additionally, LC3 is effective in reducing CO₂ emissions as compared to clinker production, making it an ideal
18 candidate for developing low-carbon binders in the cement and concrete sector [45].

19 The objective of this study was to design mortar using an alternative cement binder using limestone flash-
20 calcined sediment/millstone-clay cement (LFC) binder based on flash-calcined sediments and clays. The
21 approach adopted in this paper is to develop mortar of high strength by using waste materials such as river-
22 dredged sediments and excavated clay soils to decrease both CO₂ emissions coming from OPC production and
23 reduce waste quantities from project excavations. Also, raw sediment (RS) and raw millstone clay (MC) are
24 thermally treated within a few seconds using flash calcination at 750 °C to eliminate their organic content and
25 activate their particle's pozzolanic phases. The effect of flash-calcination is noticeable when studying the
26 material's characteristics before and after this process. Also, to confirm the pozzolanic effect of flash calcination,
27 the Chapelle test is done to compare the fixation of Ca(OH)₂ on MK, FCS, and FCMC. Therefore, this study for
28 argues replacing MK with flash-calcined materials. The behavior of the designed mortar is evaluated by
29 conducting several experiments such as the mechanical strength, workability, porosity, X-ray diffraction (XRD),
30 thermogravimetric analysis (TGA), and Fourier transform infrared spectroscopy (FTIR) tests are done on LFC
31 mortar and paste samples to study the behavior of FCS and FCMC in comparison to MK.

32 2. Experimental investigation

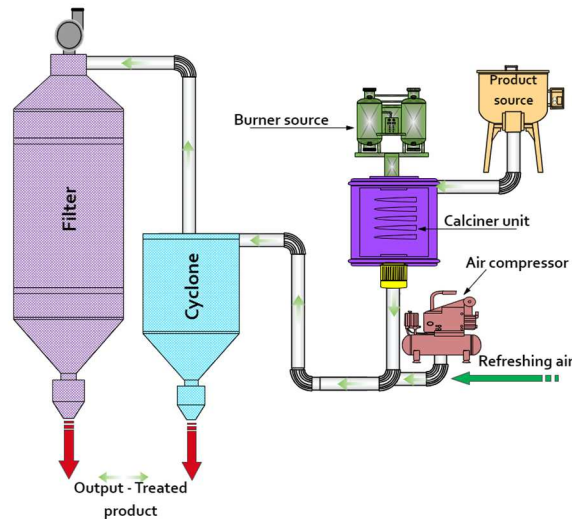
33 2.1. Materials

34 The OPC used is a CEM I 52.5 N according to the standard NF EN 196-1 [49], produced in France by the
35 company EQIOM, which is suitable for mortar and concrete formulations. The MK used is a pozzolanic material
36 from ARGICEM® and obtained from the clay mineral kaolinite calcined at 650-800°C [31]. The LS powder is
37 crushed and ground from natural limestone [50]. The RS were dredged from the Noyelles-sous-Lens river while
38 the MC was excavated from Paris region. Both materials were placed in the oven at 40 °C to be dried, then
39 crushed, and sieved at 120 µm. The flash calcination temperature is 750 °C and is applied to obtain FCS and
40 FCMC with improved physical, chemical, and mechanical properties and more active pozzolanic phases than
41 normal calcination [9][51]. The sand used is the normalized sand according to the European standard NF EN 196-
42 1:2016 [49] having 2 mm maximum particle size and a density of 2.64 kg/m³. The superplasticizer (SP) is
43 CHRYSO®Optima 372, to enhance the workability. Tap water was used in all mixes and for mortar curing.

44 2.2. Methodology

45 The process of flash calcination is composed of four steps. Materials are first dried and then ground to obtain
46 particles less than 40 microns in diameter. They are then fed to the dozer of the firing unit and transported by an
47 endless screw to the entrance of the combustion chamber where they are propelled inwards by compressed air.
48 The particles are then subjected to a heat flux to ensure firing at the target temperature in less than 1 second. The
49 process is presented in *Fig. 1*. These particles then fall into a calcining column where the material is de-structured
50 by the de-hydroxylation. All OH bonds are somehow cleaved and become potentially reactive sites. This material
51 becomes chemically active and pozzolanic. The final step consists of recovering the product in a cyclone unit by
52 95% of the processed material. A filtering system ensure no micro- and nanoparticles are left in the environment.
53 The final material obtained is considered a mineral addition (SCM) and is able to react with portlandite, a
54 compound produced by cement when it is hydrated.

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Fig. 1. Methodology of flash calcination.

4

5

Various methods were utilized to examine the physical, chemical, and mineral properties of raw and flash-calcined materials at a temperature of 750°C to determine the impact of flash-calcination and investigate the material properties. These methods included X-ray fluorescence analysis (XRF), organic matter content determination, X-ray diffraction (XRD), specific density determination, Brunauer-Emmett-Teller (BET) specific surface area measurement, particle size analysis (PSD), and Scanning electron microscopy (SEM). Also, the Chapelle test was performed on MK, FCS, and FCMC to compare their pozzolanic reactivity.

10

To create an eco-friendly limestone flash-calcined SCM cement binder (LFC), a mass fraction of clinker used is 64%, SCM's is 18-26%, and LS is 10-18%. Nine different formulations were prepared as presented in *Table 1*.

13

To compare the behavior of mortar with MK and that with FCS and FCMC, several tests were done, including a workability test, compressive strength test, porosity test. On day 28, mercury porosity test is done on the three mixes that includes highest percentages of MK/flash-calcined materials (C1,C4,C7). Also, on day 28, thermogravimetric analysis (TGA), XRD, and FTIR tests were done on paste samples (C1,C4,C7) to examine the difference in the influence of LFC with MK versus that with FCS and FCMC.

18

2.3. Materials characterization

19

Chemical composition was analyzed using X-ray fluorescence analysis (XRF) by Socachim XRF Phoenix 3000 R according to NF ISO 23345w, and mineral composition was studied using X-ray diffraction (XRD) with a Bruker D2 Advance device equipped with Cu K α radiation. Organic matter content was determined following to prEN 17685-1 [52]. Fourier transform infrared spectroscopy (FTIR) was conducted using thermo scientific Nicolet iS20. The specific density was measured using a helium pycnometer device according to NF EN 1097-7:2008 [53], while specific surface area was measured using the BET method calculated using a Micromeritics Autopore IV 9505 according to NF EN ISO 18757:2006 [54]. Particle size distribution (PSD) was measured using a laser particle analyzer, Beckman Coulter LS 13320 using the dry method. Finally, Scanning electron microscopy (SEM) analysis was performed using JCM-6000PLUS.

28

Overall, these methods were used to obtain a comprehensive understanding of the physical, chemical, and mineral properties of the material used in this study.

30

2.4. Mix design

31

This study follows the guidelines of standard NF EN 197-5 [55] to determine the mix proportions of CEM II/C-M cement. According to the standard, CEM II/C-M cement should have a cement ratio ranging from 50% to 64%, calcined material ranging from 36% to 50%, and limestone limited to 6% to 20% by mass. The mix formulations in this study are designed accordingly and are presented in Table 1. The study evaluates nine different formulations along with a reference mix called C0, which consists of 100% OPC by mass.

36

The first set of formulations, labeled as C1, C2, and, C3 adhere to a limit of 64% OPC, 18-24-26% MK, and 10-14-18% LS. The second set of formulations, referred to as C4, C5, and C6, also have a limit of 64% OPC, but with 18-24-26% FCS and 10-14-18% limestone LS. The third set of formulations, named C7, C8, and, C9 are limited to 64% OPC, 18-22-26% FCMC, and 10-14-18% limestone LS.

39

1 High-performance concrete is a specialized type of concrete that contains a reduced amount of water
 2 compared to the binder. This composition is necessary to achieve specific mechanical properties such as high
 3 compressive strength and excellent durability with minimal maintenance. In Europe, HPC is classified as concrete
 4 with a compressive strength exceeding 60 MPa after 28 days and a low water-to-binder ratio (<0.4) [56]. In order
 5 to achieve a mortar with high strength, the water-to-binder ratio (W/B) is set to 0.33 with the addition of the SP to
 6 compensate for the workability. The sand-to-binder ratio (S/B) is equal to 3 and is chosen constant with the
 7 percentage of SP added (2% of the total binder mass) for all formulations to compare the effect of each SCM on
 8 the mortar properties such as workability and mechanical strength under the same conditions.

9 **Table 1**
 10 Mix proportions for mortar (kg/m³)

Mix kg/m ³	OPC	MK	FCS	FCMC	LS	Water	Sand
C0	560	-	-	-	-	185	1682
C1	354	144	-	-	55	183	1661
C2	354	122	-	-	78	183	1661
C3	355	100	-	-	100	183	1662
C4	355	-	144	-	55	183	1661
C5	355	-	122	-	78	183	1661
C6	355	-	100	-	100	183	1662
C7	355	-	-	144	55	183	1665
C8	355	-	-	122	78	183	1665
C9	355	-	-	100	100	183	1665

11

12 2.5. Mixing procedure

13 To ensure uniformity, the dry mixture is thoroughly blended for 60 seconds in a 5-liter mixer. Second, add
 14 water with the SP for the next 30 seconds. Afterward, sand is added and mixed using high speed for 90 seconds.
 15 Then, stop and scrape the material off the mixer walls before mixing continues for another 60 seconds. Finally,
 16 mix for an additional 60 seconds at high speed. All specimens are molded using steel molds and placed at room
 17 temperature at 20 °C for 24 hours. The samples are then removed from the molds and submerged in water for
 18 curing.

19 2.6. Lab tests

20 The Chapelle test is carried out to validate the pozzolanic reaction for the flash-calcined material used in
 21 HPM formulations. As indicated by the standard NF P18-153 [57], to study the pozzolanic activity of the powder
 22 materials, a reaction should take place with quicklime (CaO). Firstly, 250 ml of distilled water is boiled to remove
 23 the CO₂, then the decarbonized water is added to the mixture of (1g of powder and 2g of CaO) in an Erlenmeyer
 24 flask. The solution will be maintained at a temperature of 85°C for 16 hours under constant stirring using magnetic
 25 stirrers. After 16 hours, the system is cooled and 60 g of dissolved sucrose in 250 ml of water is added to the
 26 solution under continuous stirring for 15 mins. Then, the solution is filtered, and (25 ± 0.2) ml of clear solution is
 27 removed. Finally, titration is carried out with 0.1M HCl using phenolphthalein as a color indicator.

28 Workability was measured following the European standard NF EN 1015-3 [58] to measure the fluidity and
 29 flow diameter of fresh mortar using the flow table.

30 The compressive strength tests were carried out on 7 and 28 days following the European standard NF EN
 31 196-1 [49].

1 The porosity test was carried out on mortar samples after 28 days of curing following the mercury porosity
2 method according to standard ISO 15901-1:2016 [59].

3 The paste samples were crushed into a powder form before undergoing TGA, XRD, and FTIR tests.

4 The TGA test involved increasing the temperature from 40°C to 105°C at a rate of 2°C/min to remove excess
5 water from the samples. The temperature was held constant for 2 minutes at 105°C to stabilize the mass loss
6 before increasing the temperature from 105°C to 1000°C at a rate of 10°C/min to analyze the mass loss at
7 different temperatures. The same method is used by Chu et al. [60] on flash-calcined sediments to analyze the
8 bound water content and Ca(OH)₂ content.

9 The bound water content may be calculated from the TGA analysis according to the following equation:

$$10 \quad QB = M40 - M400 \quad \text{Eq. 1}$$

- 11 • QB: Bound water content (wt%).
- 12 • M40(40 °C): Mass of sample at 40 °C.
- 13 • M400 (400 °C): Mass of sample at 400 °C.

14
15 In addition, Ca(OH)₂ content may also be measured from the TGA analysis according to the following
16 equation:

$$17 \quad Q \text{ Ca(OH)}_2 = \frac{(\Delta m_{450^\circ\text{C}} - 550^\circ\text{C}(t) \cdot M_{\text{Ca(OH)}_2})}{MH_2O} \quad \text{Eq. 2}$$

- 18 • Q Ca(OH)₂: Ca(OH)₂ content in lime mixture (wt%).
- 19 • M_{Ca(OH)₂}: Molar mass of Ca(OH)₂.
- 20 • MH₂O: Molar mass of water.

21
22 Ca(OH)₂ content consumed by the pozzolanic reaction was estimated using the following equations:

$$23 \quad \text{Ca(OH)}_2 \text{ reacted} = \text{Ca(OH)}_2 \text{ total} - \text{Ca(OH)}_2 \text{ non reacted} \quad \text{Eq. 3}$$

$$24 \quad \text{Ca(OH)}_2 \text{ non reacted} = \Delta m_{450^\circ\text{C}} - 550^\circ\text{C}(t) \cdot M_{\text{Ca(OH)}_2} / MH_2O + \text{Ca(OH)}_2 \text{ carbonated} \quad \text{Eq. 4}$$

25
26
27 Similar to the method of characterization of the materials, XRD and FTIR are done using the same process
28 however on the powdered paste samples.

29 3. Experimental results

30 3.1. Effect of flash calcination on material characteristics

31 This study didn't conduct trials to reach an optimum flash calcination temperature for the tested materials,
32 however a study done by Chu et al. [60] shows that flash calcination at 750 °C is optimum for sediments for
33 Noyelles-sous-lens. The author has studied the properties of sediments flash-calcined at three different
34 temperatures, 650 °C, 750 °C, and 800 °C. Comparing the amount of hydrates formed and the fixation of Ca
35 (OH)₂, flash calcination at 750 °C showed the best results. Also, it's important to note that even though flash
36 calcination at 800 °C can remove organic content more than that done at 750 °C by 5 %, however at 800 °C, the
37 density has decreased due to recrystallization of mineral phases [60][61], and the specific surface area has
38 decreased from lowering the melting temperature of the material [60][62]. Similar to the flash calcination of
39 sediments at 750 °C, the same temperature is used for millstone clay. The transformation of clay into MK is a
40 common method that could be done by either calcination or flash-calcination. Nicolas et al. [30] mentioned that
41 the transformation of clay into MK at 700 °C can be done with flash calcination. Also, according to the
42 mineralogical composition XRD, MK, and FCMC have very similar mineralogy as shown in Fig. 4, therefore flash
43 calcination at 750 °C is effective for MC treatment.

44 Flash calcination at 750 °C with a duration of one tenth of a second was able to eliminate the organic content
45 in both raw sediment (RS) and the raw millstone clay (MC) by 99% and 94%, respectively. For both materials,
46 their density was higher after flash calcination. That of RS has increased from 2.33 g/m³ to 2.60 g/m³, while that of
47 MC increased from 2.49 g/m³ to 2.71 g/m³. Faure et al. [38] have mentioned in a study about using calcined
48 sediments as SCMs, that raw dredged sediments have an organic fraction that decreases their density.
49 Therefore, the high elimination of the organic content of RS and MC by flash calcination at 750 °C led to an
50 increase in the material's density.

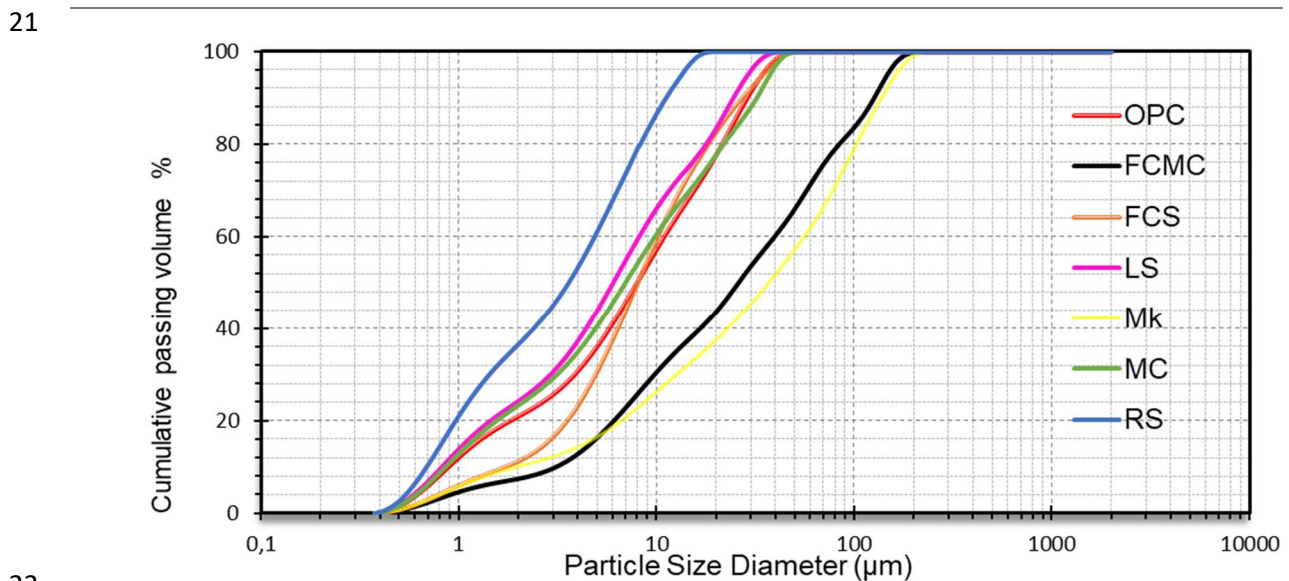
51 The BET surface area for RS showed a sharp increase from 7.48 m²/g to 27.45 m²/g after flash calcination.
52 However, the BET surface area for MC decreased slightly from 34.36 m²/g to 33.01 m²/g. This behavior difference
53 between RS and MC is because pre-treated sediments had a smaller surface area and a higher organic content.
54 Also, their organic content was equal to 13.5%. Ramarosan et al. [63] has stated that the high temperature of

1 normal calcination could increase the surface area with the increase of temperature up to a maximum of 400 °C.
 2 The last has mentioned that this increase is explained by the loss of organic content which generate pores into
 3 the sediment particles, thus increasing the surface area [63]. Although flash-calcination has a temperature of 750
 4 °C, however during this process, the organic material is rapidly burned off, leaving behind a porous structure with
 5 a higher surface area, thus increasing their pozzolanic reactivity. MC had a small percentage of organic content
 6 equal to 8.6%. After eliminating it, the BET was only slightly affected. This suggests that the BET surface area
 7 may remain constant for some materials after flash calcination if the material being calcined is already highly
 8 porous and has a high surface area before the calcination process. Volet et al. [64] has explained the porous
 9 structure of clayey material such as kaolin and metakaolin. The author has deduced that both clayey materials
 10 has a disordered porous matrix. Therefore, the porous structure of MC is the explanation why the BET surface
 11 area wasn't affected.

12 The chemical analysis demonstrates that the percentage of major oxides remained almost the same after
 13 flash calcination, as shown in Table 2. According to the ASTM C618, the class N pozzolan should have the sum
 14 of (SiO₂, Al₂O₃, Fe₂O₃) is > 70%, and the LOI is <10% by mass. FCS has a summation of SiO₂, Al₂O₃, and Fe₂O₃
 15 approximately equal to 67 %, but that of FCMC is equal to 87%. Therefore, FCMC satisfies the requirements of
 16 ASTM C618 standards for class N pozzolan. FCS is not very far from meeting this requirement, but different
 17 studies have mentioned the pozzolanic activity of flash-calcined sediments [9][15][23][65].

18 **Table 2**
 19 Chemical and physical composition for materials used

Chemical composition (%)	RS	FCS	MC	FCMC	MK	OPC	LS
Silicon dioxide, SiO ₂	41.06	44.49	60.45	61.64	58.6	20	0.47
Aluminum oxide, Al ₂ O ₃	12.08	12.66	18.27	18.58	31.17	5.1	0.21
Ferric oxide, Fe ₂ O ₃	6.72	9.5	6.63	6.87	3	3.4	0.08
Calcium oxide, CaO	20.15	21.89	2.02	2.85	1.26	63.5	65.39
Magnesium oxide, MgO	0.99	2	0.75	0.77	5.98	0.8	0.54
Sulphur trioxide, SO ₃	0.2	0.2	-	-	-	-	-
Sodium oxide, Na ₂ O	2	2	-	-	-	-	-
Potassium Oxide, K ₂ O	1.9	1.9	-	-	-	-	-
LOI	13.5	0.2	8.6	0.57	-	-	29
Physical characteristics							
Density g/m ³	2.33	2.60	2.49	2.71	2.6	3.15	2.73
BET m ² /g	7.48	27.45	34.36	33.01	13.59	1.26	1.26

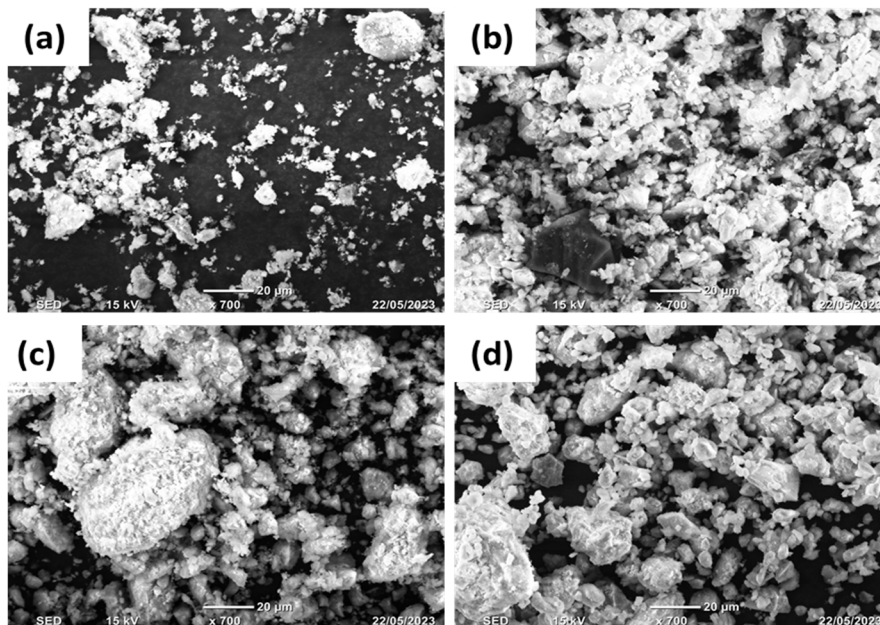


23 Fig. 2. Particle size diameter for raw and flash-calcined materials.

24 As shown in Fig. 2, the PSD was higher after flash calcination. For example, the D50 for both sediment and
 25 millstone clay has increased from 3.58 µm and 6.95 µm to 8.01 µm and 25.92 µm, respectively. This increase is

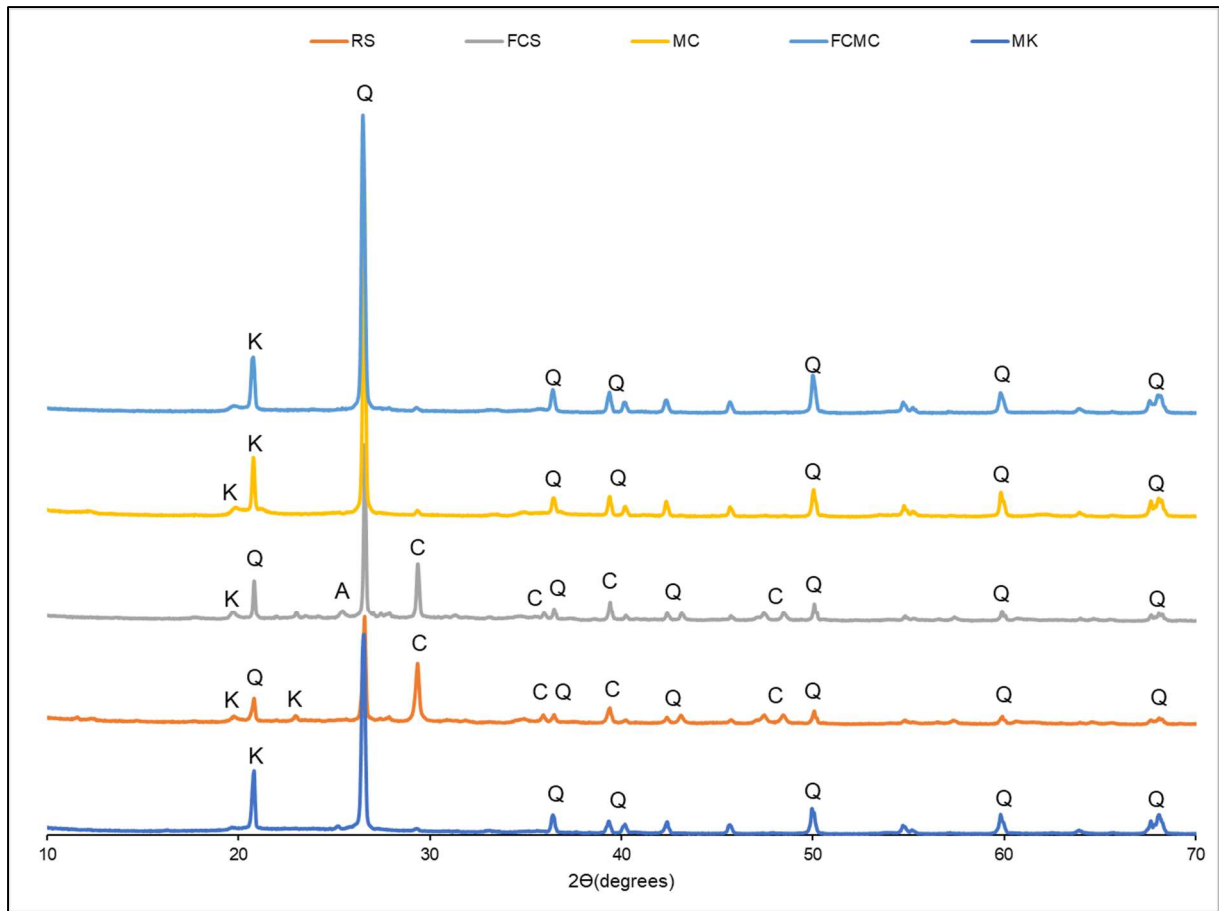
1 related to the increase in the BET surface area of sediment and millstone clay resulting in very-fine particles that
2 agglomerate together and increase the PSD. Chu et al. [60] conducted a study that yielded similar results for
3 sediments, which they attributed to the agglomeration of clay minerals rather than sintering during the calcination
4 process [66]. For clays, a study by Skibsted et al. [39] has mentioned the agglomeration phenomenon. The author
5 has deduced calcination of clays at high temperatures will achieve ultra-fine particles that will join together and
6 increase their PSD [39].

7 The utilization of scanning electron microscopy (SEM) involves the examination of the inner structure of
8 materials at an exceptionally high level of detail. This technique enables a powerful level of magnification,
9 reaching up to 500,000 times, allowing for a comprehensive investigation and analysis of the microscopic
10 components of mortar [27]. It is evident that there are notable differences in the SEM analysis of RS and RC as
11 shown in Fig. 3. Specifically, following flash-calcination, the particles have clearly undergone a transformation,
12 adopting a more uniform and spherical shape. Additionally, there is a greater degree of particle cohesion
13 observed after flash-calcination, with increased joining between the particles. Various studies have conducted
14 analyses using SEM for both raw and flash-calcined materials. One such study, conducted by AMAR et al. [27],
15 demonstrates that the irregular shapes of particles present in the raw sediments are identified as Pyrite, indicating
16 the necessity for thermal treatment. The author of the study also notes that after flash-calcination, the particle
17 shape becomes more spherical. These particles have been identified as flash-calcined kaolin clay [27][67],
18 originating from the agglomeration process of submicron particles [27][68]. Consequently, the findings of this
19 study are consistent with other research, as evidenced by the observed increase in BET surface area and particle
20 size distribution (PSD) resulting from the agglomeration phenomenon, which were analyzed through SEM
21 examinations of both raw and flash-calcined materials.
22



23
24 Fig. 3. Electron microscopy for raw materials and flash-calcined materials: (a): RS, (b): FCS, (c): RC, (d): FCMC.

25
26 The XRD analysis results are presented in Fig. 4. This test can detect mineralogical changes after flash
27 calcination and provide insights into the transformation behavior and thermal stability of minerals in RS and MC.
28 Calcite can decompose into CaO and CO₂, resulting in a decrease in the intensity of its diffraction peaks in XRD
29 analysis, while quartz is stable and less affected by flash calcination. RS and MC consist mainly of quartz and
30 kaolinite, with calcite present only in RS. Flash calcination caused a decrease in the intensity of the calcite peak in
31 FCS, but the quartz remained the same in both FCS and FCMC. Anhydrite formation was observed in FCS due to
32 the reaction of CaO from the decomposition of CaCO₃ and sulfate to form CaSO₄ [60][69]. Flash calcination also
33 transformed kaolin to metakaolin, leading to a decrease in the intensity of the kaolin peak in XRD patterns for both
34 RS and MC.
35



1
2 Fig. 4. XRD for raw and flash-calcined materials.

3 3.2. Chapelle test

4 Both flash calcined materials have registered positive values of Ca(OH)_2 with MK demonstrating the highest
 5 of all, as shown in Table 3. FCMC has registered 257.27 mg of Ca(OH)_2 which is slightly higher than FCS, with
 6 233.88 mg of Ca(OH)_2 . Therefore, FCMC tends to be more pozzolanic than FCS which is compatible with the
 7 chemical analysis results. The negative Ca(OH)_2 values for LS is due to their high CaO percentages, as shown by
 8 the XRF results in Table 2. The high percentages of CaO may have caused the dispersion of Ca ions into the
 9 solution which prevented the fixation of Ca(OH)_2 .

10 **Table 3**
 11 Chapelle test.

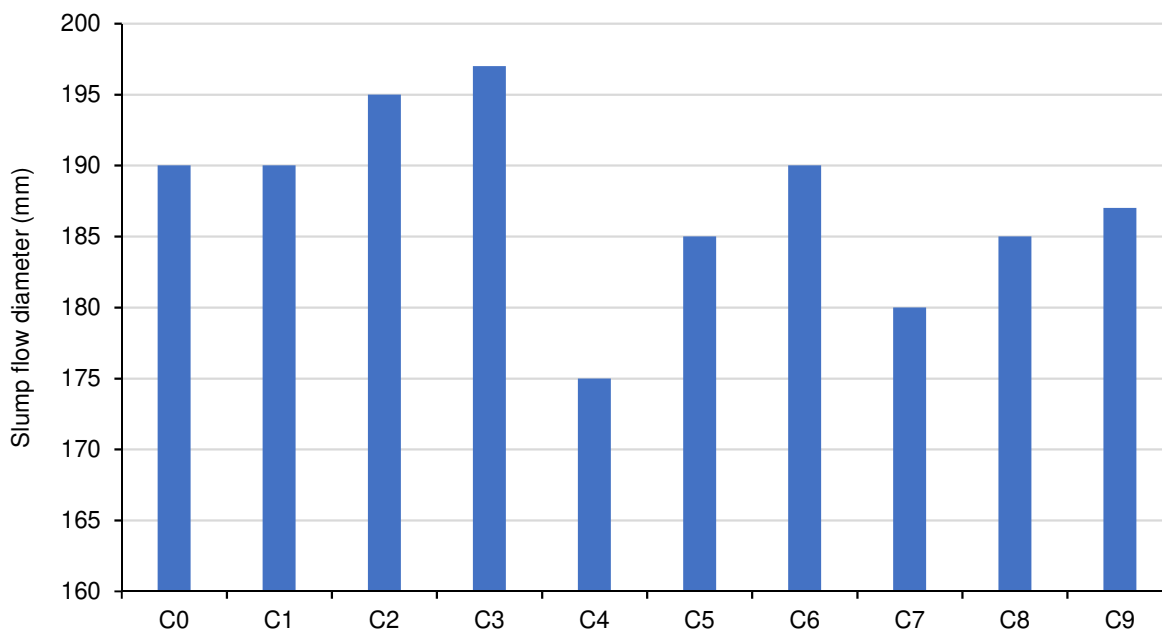
Materials	MK	FCS	FCMC	LS
Mass (mg) of Ca(OH)_2 fixed	1286.35	233.88	257.27	-561.3

12 Then, according to Chapelle's test results, both FCS and FCMC showed pozzolanic reactivity but are not superior
 13 to MK.

14 3.3. Workability

15 The slump flow diameter of C0 was measured to be 190 mm, while C1 had the same slump flow diameter as
 16 shown in Fig. 5. However, C4 and C7 had lower values of 175 mm and 180 mm, respectively. The decrease in
 17 workability upon substituting with flash-calcined materials such as FCS and FCMC is attributed to their high BET
 18 surface area, which is greater than that of MK. This increase in the specific surface area leads to an increase in
 19 water requirements to initiate the flow [70], negatively impacting workability. The fine sediments also have a high-

1 water absorption capacity [71], which deteriorates the fresh state of the mix. Studies have shown that at a fixed
 2 content of SP [72], the workability decreases when substituted with calcined clay. Therefore, the flash-calcined
 3 materials have a similar trend to MK and have not improved workability due to their large surface area and high-
 4 water absorption capacity. When increasing the substitution of LS and decreasing the percentage of MK, FCS,
 5 and FCMC, the workability increased for all formulations. For example, C2 and C3 showed higher workability than
 6 C0 and C1, equal to 195 and 197 mm, respectively. Also, C5 and C6 showed an increase in workability higher
 7 than C4, equal to 185 and 190 mm. Finally, C8 and C9 showed an increase in workability as well equal to 185
 8 and 187 mm. The increase in workability for all formulations is attributed to an increase in LS percentages and a
 9 decrease in MK and flash-calcined materials. Different researchers have mentioned the positive effect of LS on
 10 the workability of concrete [73][74][75]. This is attributed to the fineness and spherical nature of the LS particles
 11 [74].



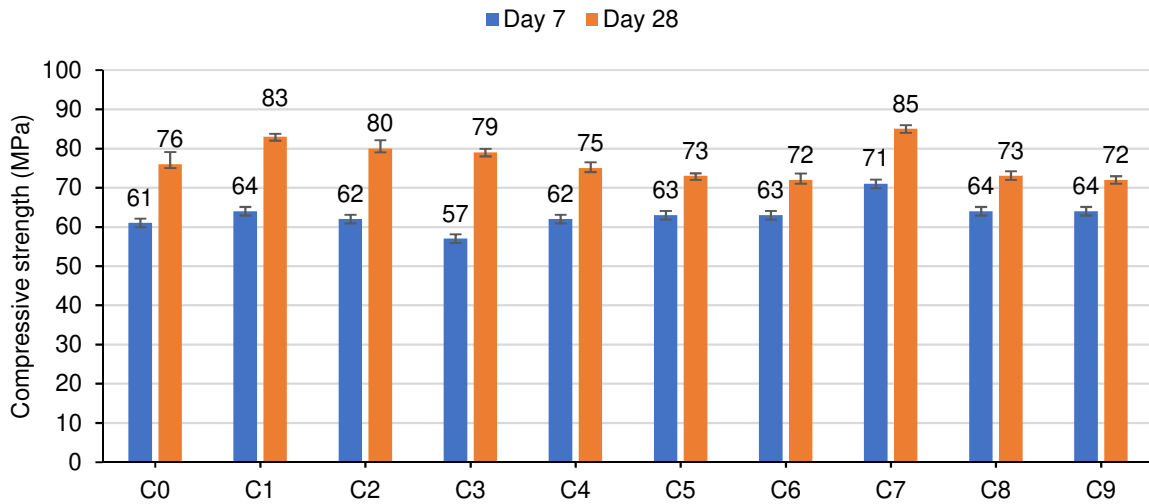
12
 13 Fig. 5. Workability test.

14 3.4. Compressive Strength

15 The results of the compressive strength tests on various LFC formulations were compared to a reference mix
 16 called C0, which recorded strengths of 61 MPa at 7 days and 76 MPa at 28 days. The results are recorded in Fig.
 17 6. C1 had compressive strengths of 64 MPa at 7 days and 83 MPa at 28 days, and the addition of pozzolanic
 18 material MK and filler material LS in this formulation resulted in improvements in both early and late-age
 19 compressive strength. Other formulations, such as C4 and C7, also had higher compressive strengths than C0.
 20 On day 7, C4 showed improved compressive strength compared to C0 and recorded a strength of 75 MPa at day
 21 28. Meanwhile, C7 had higher strengths than C0, recording 71 MPa at day 7 and 85 MPa at day 28. C4 had less
 22 strength than C1 by 3% and 10% at day 7 and 28, respectively. However, C7 had higher strength than C1 by 11%
 23 and 2% at day 7 and 28, respectively.

24 This is attributed to the pozzolanic reaction of the flash calcined materials, which contributed to the
 25 development of strength. These results are similar with research studies. After day 7, Du et al. [65] has concluded
 26 that replacement of OPC with limestone calcined clay cement can increase the compressive strength in
 27 comparison with the reference mix. The author has explained that this increase is due to the reaction of
 28 portlandite, calcined clay, and LS which cause densification to the microstructure and the pore connectivity could
 29 decrease [65][47]. When the formulations were modified by increasing the percentage of LS and decreasing the
 30 percentage of MK, FCS, and FCMC, a gradual decrease in compressive strength was observed. For instance, on
 31 day 28, C2 and C3 exhibited a reduction of 4% and 5% in compressive strength, respectively. Similarly, C5 and
 32 C6 showed a decrease of 3% and 4% in compressive strength. The compressive strength of C8 and C9
 33 formulations followed a similar trend, with a decrease of 14% and 15% on day 28. Therefore, as the percentage of
 34 LS was increased from 10% to 14% and 18%, while simultaneously decreasing the percentage of MK and flash-
 35 calcined materials, the compressive strength decreased. Literature studies have demonstrated that the
 36 combination of limestone and calcined clays can enhance the mechanical and physical properties of materials

1 and allow for higher substitution of OPC [76]. However, the highest compressive strength is achieved when using
 2 a 1:2 ratio of calcined clay to limestone [76]. In this study, three different ratios of MK or flash-calcined materials
 3 to limestone, namely 2.6, 1.6, and 1, were examined. The results indicate that the ratio of 2.6 yields the highest
 4 compressive strength, while the ratio of 1 exhibits the lowest compressive strength. Another study conducted by
 5 Parsompech et al. [77] on LC3 mixes reveals that as the percentage of calcined clay exceeds that of LS, the
 6 compressive strength increases linearly. This increase can be attributed to the pozzolanic reactivity of the
 7 calcined clay, where the amorphous $Al_2O_3 \cdot 2SiO_2$ in the calcined clay reacts with calcium hydroxide from OPC,
 8 forming a calcium aluminum silicate hydrated gel that refines the pore structure and improves compressive
 9 strength [77].



10
 11 Fig. 6. Compressive strength test (MPa).

12 3.5. Porosity

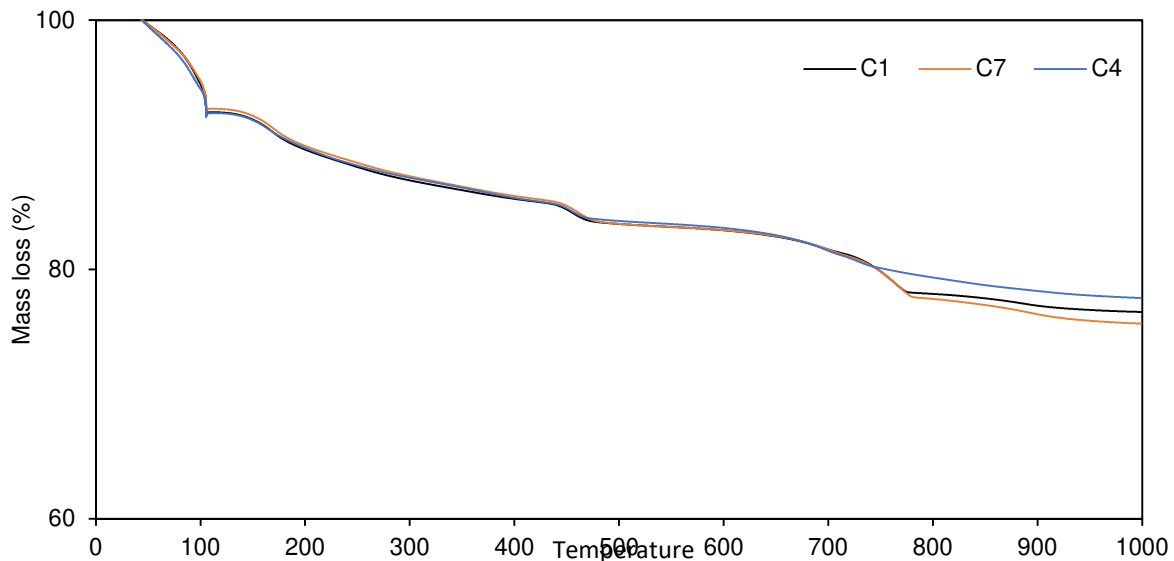
13 The study requires the porosity test to confirm the high compressive strength results of the LFC formulations.
 14 The presence of low porosity is crucial in improving the strength of cement-based materials, as noted by various
 15 studies [77][78][79][80]. The porosity results are shown in Table 4. At day 7, all formulations show critical pore
 16 diameters below 100 nm, which research suggests have minimal effect on cement binder properties [81][82][36].
 17 Thus, the formulations on Day 7 have negligible critical pore sizes that can affect mortar properties. At day 28, the
 18 critical pore size distribution for all formulations decreased significantly, with values less than 100 nm.
 19 Furthermore, table data shows that the porosity percentage for all formulations decreased at day 28. To check the
 20 validity of porosity measurements with compressive strength results, the study analyzed the porosity % as a
 21 function of compressive strength for the mortar formulations on day 28. The compressive strength results for C7,
 22 C1, and C4 were 85 MPa, 83 MPa, and 75 MPa, respectively. The porosity % for these formulations was 4.55%,
 23 5.65%, and 7.52%, respectively. The study concludes that the porosity measurements validate the compressive
 24 strength for LFC since higher porosity values correspond to lower compressive strength, as observed in various
 25 studies [80][83][84]. It's also noticeable that on day 28, the porosity for C4 formulation has a higher strength and
 26 porosity than C1 formulation on day 7. Since C4 is composed of FCS instead of MK, then the hydration process is
 27 different which is validated when conducting the TGA analysis in section 3.6. On day 28, C4 exhibits a greater
 28 decrease in mass compared to C1 within the temperature range of 230 °C to 420 °C, which aligns with the
 29 decomposition of calcium carbonate ($CaCO_3$). This indicates that the hydration process differs between the two
 30 materials, and it's worth considering that the compressive strength duration might not fully reflect the long-term
 31 characteristics of the mortar samples.

32 **Table 4**
 33 Porosity test

Mortar Mix	Porosity %		Average pore diameter (nm)		Critical pore size diameter (nm)	
	Day 7	Day 28	Day 7	Day 28	Day 7	Day 28
C1	5.66	5.65	29	17	50	12
C4	10.42	7.52	60	23	70	50
C7	7.98	4.55	40	21	70	17

1 3.6. TGA

2 TGA is a thermal analysis technique used to determine the changes in the mass of a material as it is heated,
3 and it can provide information about the thermal stability and decomposition behavior of the material. LFC
4 formulations have demonstrated from the TGA curves 4 major transformation phases. The first phase occurs
5 between 40 °C to 230 °C, which corresponds to the evaporation of water at 105 °C and then the decomposition of
6 ettringite and the dehydration of hydration products [85]. The hydration products identified in the research for LC3
7 powder are C-S-H, ettringite, monocarboaluminate, and hemicarboaluminate, and portlandite [44][86]. The
8 second phase of mass loss is between 230 °C and 420 °C which is C-S-H dehydration and the beginning of
9 portlandite decomposition [87][88]. The portlandite decomposition is between 420 °C and 500 °C. Finally, the de-
10 carbonation reaction takes place between 500 °C and 1000 °C.
11 On day 28, the TGA curve demonstrated in Fig. 7 a similar trend of mass loss (%) and temperature (°C) for the
12 LFC formulations with MK and flash-calcined materials, indicating that these formulations may have undergone a
13 comparable hydration reaction during curing.
14 The three mortar formulations experienced mass loss at different temperature ranges. C1, C4, and C7 all lost
15 water between 40°C and 105°C, with a loss of 1.26%, 1.18%, and 1.22%, respectively. Between 105°C and
16 230°C, the samples lost mass due to the decomposition of calcium hydroxide (Ca(OH)₂). The amount of mass
17 loss for C1, C4, and C7 was 4.79%, 4.68%, and 5.14%, respectively.
18 The temperature range of 230°C to 420°C led to the decomposition of calcium carbonate (CaCO₃) in the samples,
19 causing them to lose 2.77%, 2.84%, and 2.88% of their mass, respectively. Any remaining organic compounds in
20 the samples began to decompose between 420°C and 500°C, causing them to lose 1.83%, 1.59%, and 2% of
21 their mass.
22 Between 500°C and 1000°C, the samples experienced a significant loss of mass, with C1, C4, and C7 losing
23 7.05%, 6.18%, and 7.99% of their mass, respectively. The total loss for the mortar samples C1, C4, and C7 was
24 25.74%, 22.28%, and 24.33%, respectively.
25 Notably, the mass loss between the three formulations was very similar at all stages, especially for C1 and C7,
26 which had almost identical trends. C4 experienced a lower mass loss at a temperature of 500°C. These findings
27 provide further evidence that the three mortars had comparable hydration products at day 28.



28 ...
29 Fig. 7. TGA on paste samples.

30 3.7. XRD

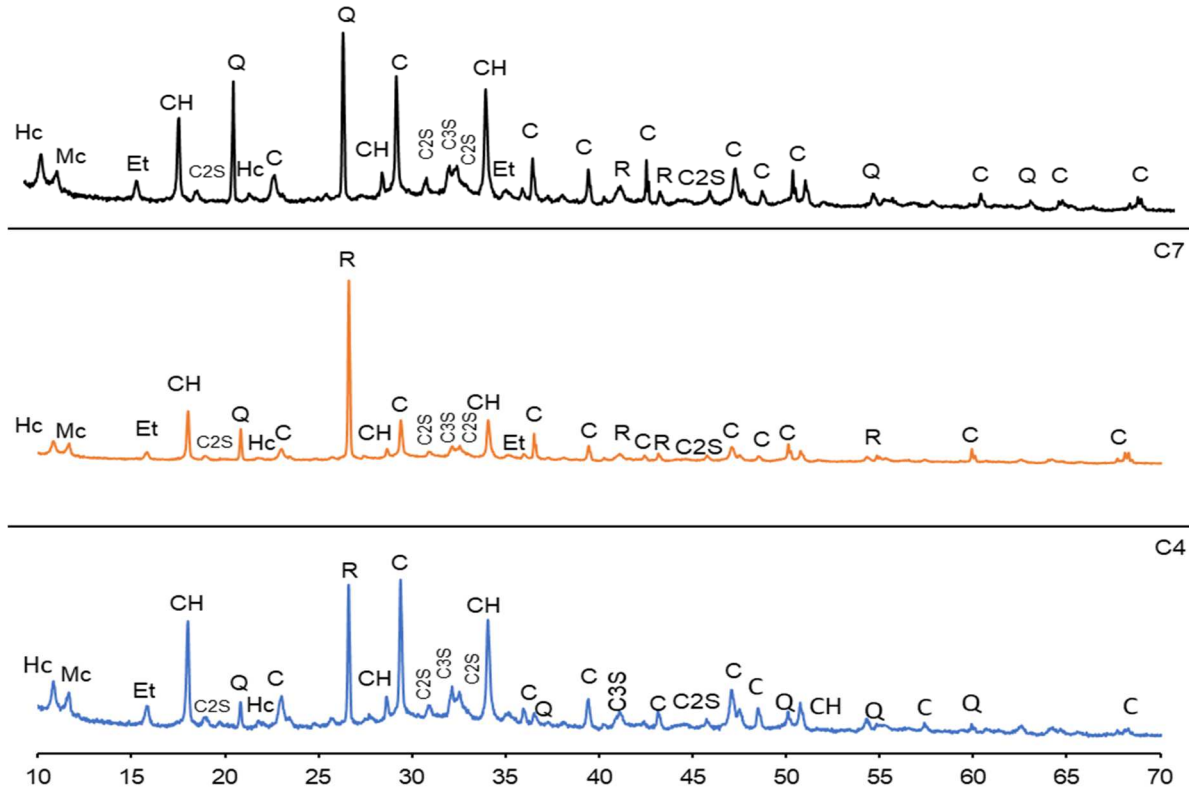
31 Fig. 8 displays the XRD results for the three pastes, which have ettringite (ET), calcium hydroxide (CH),
32 calcite (CaCO₃), calcium monocarboaluminate (Mc), hemicarboaluminate (Hc), and calcium silicate hydrate (CSH)
33 as their main components. Notably, a small variation in peak intensity is the only difference observed among the
34 XRD results for the three paste samples.
35 Drissi et al. [89] conducted a study that examined the hydration process of a binder that consisted of OPC+
36 MK+ LS. The last examined various samples using XRD at different time periods. On the 28th day, the XRD
37 outcomes were comparable to the current ones. Specifically, they detected Et at around 16° and 35°, and CH at

1 18°, 29°, and 34°. Moreover, the author identified the peaks at 12° as Hc and Mc, which exist in LS-binary and
 2 ternary binders. Another study done by Zadeh et al. [46] shows that LC3 binder has CH at 18° and 34°, CaCO₃ at
 3 30°, and Mc at 12°.

4 Cao et al. [44] indicated that in LC3 blends replacing 50% cement, calcined clays have strong pozzolanic
 5 reactivity and work together with limestone to use up the portlandite generated during cement hydration. As a
 6 result, lower levels of C2S and C3S are observed when compared to OPC mixtures. The XRD findings from this
 7 study indicate that the quantities of C2S and C3S are the same for all three formulations, implying that the
 8 reaction between MK and LS is comparable to that of FCS and LS, as well as FCMC and LS.

9 Therefore, the XRD findings for C1, C4, and C7 are similar to those in their prior study. Thus, both FCS and
 10 FCMC can be used to replace MK to form LFC binder.

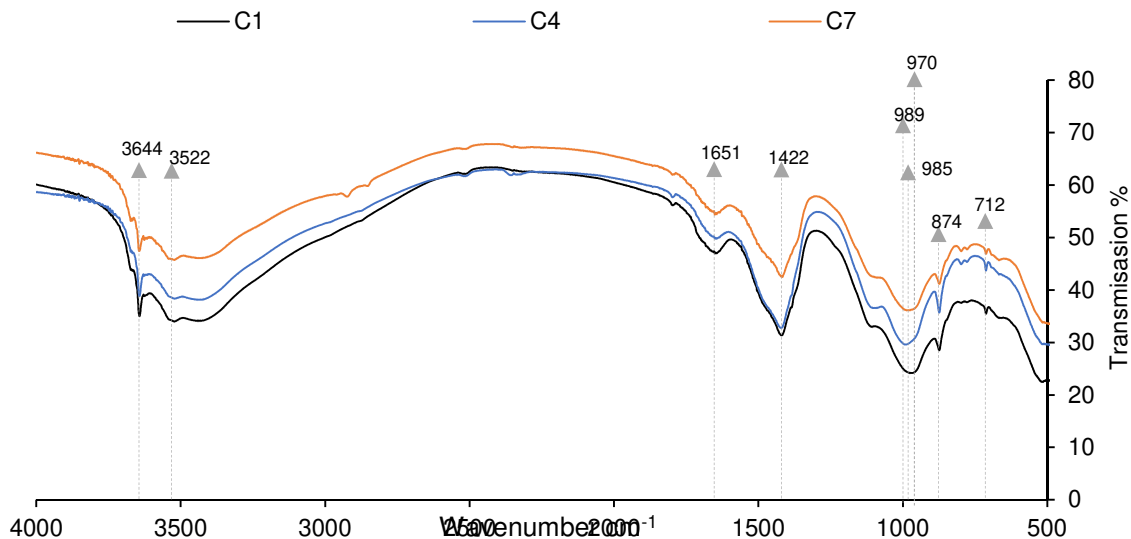
C1



11
 12 Fig. 8. XRD on paste samples

13 3.8. FTIR

14 The FTIR Spectra are utilized to identify the structural bonds present in the designed mortars, as shown in
 15 Fig. 9. The results indicate that the three formulations exhibit a similar structure, as evidenced by the presence of
 16 identical peaks at 3644 cm⁻¹, 3522 cm⁻¹, 1651 cm⁻¹, 1422 cm⁻¹, 874 cm⁻¹, and 712 cm⁻¹. Furthermore, C1, C4, and
 17 C7 display peaks at 970 cm⁻¹, 989 cm⁻¹, and 985 cm⁻¹, respectively. The peak observed at 3644 cm⁻¹ is attributed
 18 to portlandite and corresponds to the symmetric stretching vibration of the -OH bond [44][90]. The broad-centered
 19 peaks at 3522 cm⁻¹ and 1651 cm⁻¹ are caused by symmetric and asymmetric stretching vibrations and bending
 20 vibrations of -OH in free or chemically bound water [44][91]. Additionally, the peaks centered at 1422 cm⁻¹, 874
 21 cm⁻¹, and 712 cm⁻¹ correspond to the asymmetric stretching vibration and plane vibration of calcium carbonate
 22 [44][92]. The peaks at 970 cm⁻¹, 989 cm⁻¹, and 985 cm⁻¹ represent the Si-O-T asymmetric vibration which
 23 indicates the C-S-H structure [44]. Given that all three formulations possess the same structural bonds on day 28
 24 of the test, FCS and FCMC can both be used as substitutes for MK in producing LFC.



1

2 Fig. 9. FTIR on paste samples.

3 **4. Conclusion**

4 The aim of this study is to investigate how flash calcination at a temperature of 750°C affects marine dredged
 5 sediments and excavated millstone clays as a replacement for MK in the development of limestone calcined clay
 6 cement mortar. Nine different mortar formulations are created, all consisting of 64% OPC, 10-18% LS, and 22-
 7 26% allocated to either MK, FCS, or FCMC for each mortar formulation. Furthermore, a reference mix C0 is
 8 prepared for comparison with the LFC mortar mixes developed in terms of workability and compressive strength.
 9 In order to examine the impact of flash calcination, physical and chemical tests are conducted both before and
 10 after the process at 750°C. Additionally, microstructural tests are carried out on LFC paste samples to study the
 11 behavior of the LFC designed.

12 According to the results obtained from the XRF and XRD tests, utilizing flash calcination at 750 °C as a thermal
 13 treatment method is quite efficient in removing organic matter, and it has a favorable impact on the density and
 14 BET surface area. However, the technique does not significantly alter the chemical composition and mineralogical
 15 structure. The findings of this study are summarized below.

- 16
- 17 • The Chapelle test indicates that FCS and FCMC exhibit favorable pozzolanic reactivity. However, they
 18 are not more reactive than MK.
 - 19 • The high BET surface area of FCS and FCMC has a negative impact on the workability of the LFC
 20 mortars.
 - 21 • Using FCS and FCMC in LFC mortar formulations exhibited a positive impact on compressive strength.
 - 22 • The porosity test results are compatible with the compressive strength results.
 - 23 • According to the microstructural analysis conducted, it can be concluded that FCS and FCMC can be
 24 utilized in the production of LFC mortars.
 - 25 • The microstructural analysis conducted on the paste samples indicates that LFC mortars made with FCS
 26 and FCMC exhibit similar characteristics to those made with MK. As a result, it can be concluded that
 flash-calcined materials can be utilized in the production of LFC mortars.

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31

32

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