Using ceramic demolition wastes for CO_2-reduced cement production

Anna Tokareva [a], Sinan Kaassamani [a], Danièle Waldmann [b],*

[a] *Department of Engineering, Faculty of Science, Technology and Medicine, University of Luxembourg, Luxembourg*
[b] *Institute for Solid Structures, Technical University of Darmstadt, Germany*

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ABSTRACT

This study focuses on assessing the pozzolanic potential of two types of ceramic demolition waste, namely terracotta roof tiles and sanitary porcelain, as substitutes for traditional calcined clays in blended and limestone calcined clay (LC3) cement production. Experimental methods employed include the modified Chapelle test and XRD for pozzolanic activity evaluation, flexural and compressive strength tests, capillary absorption measurements, and SEM for microstructure analysis. Mortars containing 10%, 20%, and 30% ceramic powders and 5%, 10%, or 15% limestone filler were tested. The findings showed that porcelain powders exhibited lower pozzolanic activity due to their lower surface area and higher firing temperature of the material. Up to 20% substitution of OPC with terracotta had minimal strength impact, with a 103% strength activity index (SAI) at 90 days. Ultrafine terracotta powder showed promise in LC3 production with up to 30% OPC substitution, achieving a 97% SAI after 90 days. The poor mechanical properties of porcelain-containing mortars were explained by surfactants present in sanitary porcelain. This research informs the cement and processing industries on the potential use of specific ceramic demolition waste materials in eco-cement production, offering insights into blended pozzolanic cements and LC3 formulations, with the goal of reducing the carbon footprint of cement manufacturing.

1. Introduction

The cement industry stands as a significant contributor to global carbon dioxide (CO_2) emissions, accounting for a substantial portion of the overall environmental footprint. These emissions arise mainly from the fuel combustion and decarbonation of limestone during clinker manufacturing, responsible for approximately 8% of global anthropogenic CO_2 emissions [1]. The urgency of this issue has driven extensive research and innovation in the search for sustainable alternatives to Ordinary Portland Cement (OPC). One promising approach to mitigate CO_2 emissions is the incorporation of supplementary cementitious materials (SCMs) into cement formulations. The utilization of SCMs not only reduces environmental impact but also enhances the performance and durability of concrete structures.

Special emphasis is placed on pozzolanic SCMs rich in amorphous silica and alumina, which can react with calcium hydroxide (CH) to produce additional calcium hydrosilicates and hydroaluminates. The use of pozzolans in concrete production has a long history. Historical records show that pozzolans of volcanic origin, such as volcanic ash, tuff, and pumice, were employed in the construction of concrete

structures in ancient Rome [2–5]. One notable characteristic of concretes incorporating pozzolans is their ability to withstand corrosion caused by salts present in seawater, attributed to their reduced CH content [5,6].

Calcined clays also find utility as pozzolanic SCMs. When clay containing kaolinite is calcined, metakaolin is formed, which is essentially an amorphous aluminosilicate ($Al_2Si_2O_7$) that can react with CH like a pozzolan to form C-(A)-S-H and aluminate hydrates [7]. These products fill space and promote mechanical and durability properties. The optimal temperature range for firing kaolinite clays is 600–900 °C, within which kaolin undergoes complete transformation into metakaolin [8–10]. However, metakaolin is also used in paper processing and ceramic and refractory industries, where there are strict requirements for colour and purity. This means that it typically costs for about 5 times the price of cement [11]. Thus, the use of conventional metakaolin is not practical for the production of general-purpose cement.

Scientists from EPFL in Switzerland and UCLV in Cuba proposed replacing part of the cement with a mixture of calcined clay and limestone filler. This formula is called LC3 (limestone calcined clay cements) [9]. After studying 46 substandard clays from around the world for use

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* Corresponding author.
E-mail address: waldmann@massivbau.tu-darmstadt.de (D. Waldmann).

in the production of LC3, they concluded that replacing 50% clinker with 30% fired clay, 15% limestone filler and 5% gypsum did not lead to a deterioration in the mechanical properties of the material.

Ceramics can also be considered as calcined clay, since it is clay with additives such as sand and feldspars, fired at a temperature of 800–1400 °C, and contains therefore amorphous matter [12,13]. Thus, there is obvious economic and environmental advantage of using ceramic waste in the cement industry compared to using calcined clay, because there is no need to develop quarries, extract natural resources and consume energy for calcination. In addition, ceramic waste is widespread throughout the world and requires recycling.

Many studies have already been conducted to demonstrate the acceptability of using various ceramic wastes as pozzolanic SCMs. Thus, Asensio et al. [14] after measuring the reactivity of ceramic demolition waste with lime and comparing the values with other standard pozzolans concluded that the ceramic waste has a pozzolanicity above fly ash and below silica fume, and after a year of reaction they bind almost all the lime. Wild et al. [15] proved the pozzolanic activity of 8 different brick powders collected in 4 countries using the Chappelle test, and Vejmelková et al. [16] obtained a Chapelle test result for ceramic powder of 840 mg(Ca(OH)$_2$)/g, which is high enough for metakaolin. Lavat et al. [17] examined the reactivity of glazed and unglazed red roof tiles using the Fratini method and concluded that both ceramic materials had good pozzolanic properties.

However, most researchers use indirect methods to study the reactivity of ceramic powders, such as XRD and TGA to measure the consumption of portlandite, or the development of compressive strength, instead of direct chemical methods. All researchers who have studied the development of portlandite during the hydration of cements with ceramic powders noted an increase in the content of portlandite in the early stages up to 28 days due to the hydration of cement phases, but its gradual decrease at later stages due to pozzolanic reactions with ceramic particles, which was not observed in reference samples [17–22]. This is also confirmed by the lower early strength of specimens made using ceramic powders compared to the strength of reference specimens due to cement dilution [23,24], but comparable or even improved late strength at substitution rates up to 20% [25–31]. Microstructure studies have shown pore refinement and matrix compaction in samples containing ceramic powders due to pozzolanic reactions [16,19,27,32,33], which leads to a decrease in water absorption [21,22,25,28,29,31], chloride ions [22,31,33] and sulfate ions permeability [31]. Thus, the improvement of the microstructure, mechanical and durability properties of cements due to the pozzolanic properties of ceramic additives has been repeatedly proven. Among the available studies on this topic, only in that of Abreu et al. [34] on the use of ground electrical porcelain insulator as SCM, the hypothesis of its pozzolanic reactivity was rejected, which may be due to the too high firing temperature of the original porcelain material.

The pozzolanic properties of ceramics are intricately influenced by the firing temperature to which they are subjected. Within the temperature range of 600–900 °C, clay minerals undergo a transformative process, rendering them reactive. This thermal treatment leads to the creation of disordered, amorphous states of silica and alumina, fundamentally altering their crystalline networks. These altered clay minerals possess the ability to react when combined with lime and water, forming insoluble CSH- and CAH-type compounds [10,17,28,35,36]. However, it is essential to note that when firing temperatures exceed 900 °C, silica and alumina can reorganize into stable compounds, such as mullite and tridymite, with diminished reactivity [10,36–38]. This makes firing temperature a critical factor in determining the pozzolanic potential of ceramic materials.

The firing temperature for porcelain is 1250–1400 °C, while terracotta ceramics and red bricks are fired at 950–1050 °C [12]. From the above, we can conclude that the pozzolanicity of red ceramics should exceed the reactivity of porcelain. However, for the moment there are not enough studies comparing the pozzolanic properties of different

types of ceramics in uniform experimental settings, and the results of the few that are available contradict each other. Thus, Pitarch et al. [39], after comparing SAI and the amount of lime fixed by porcelain tiles, sanitary porcelain and red bricks powders, indeed confirmed the slightly lower reactivity of porcelain compared to red ceramics. Pacheco-Torgal and Jalali [40] reached similar conclusions. On the other hand, Pereira-de-Oliveira et al. [38] found a more active development of strength by cement mortars with terracotta tile powders fired at a temperature of 1100–1200 °C than by mortars with red bricks powder fired at a temperature of 800–1000 °C. Also, Lasseuguette et al. [18] in their study concluded that white ceramics have better pozzolanic activity than red ceramics. However, it is worth noting that the manufacturing temperature of the ceramics used in their study was not known and judging by the greater amount of mullite in the red ceramic samples, it can be assumed that the firing temperature of the white ceramics was lower than the red ones.

Despite the proven pozollanic properties of ceramic waste, there has been no attempt to study different types of ceramic demolition waste as calcined clay in the LC3 formula. Mohit et al. [41], in their recent study, described the use of ceramic production waste to replace cement with a mixture of 15% limestone and 10, 20 and 30% ceramic. The results showed that samples containing 20% ceramic waste and 15% limestone filler had mechanical properties comparable to the reference specimen. The results of this study suggest that various ceramic demolition wastes can also be used to produce three-component limestone calcined clay cement.

2. Goals and significance of the research

The principal aim of this research is to investigate and compare the pozzolanic reactivity of two distinct types of ceramic demolition waste: specifically, terracotta roof tiles and a mixture of sanitary and tile porcelain collected after the renovation of sanitary rooms. The goal is to assess their potential suitability for use in LC3 as an alternative to traditional calcined clays. These materials have undergone minimal processing, avoiding washing and thermal treatment, aligning with a commitment to environmentally friendly and cost-effective procedures, tailored for industrial feasibility.

In the landscape of current research, there exists a noticeable gap in the comparative analysis of different ceramic waste types under standardized conditions. This void is highlighted by the finding that existing studies yield divergent results concerning the pozzolanic properties of these materials. Therefore, the present research endeavours to fill this knowledge gap and bring clarity to the intricate realm of ceramic waste utilization in cementitious applications.

In addition, while the pozzolanic potential of ceramic waste in cementitious formulations has been well-established, there has been a conspicuous absence of investigations regarding the applicability of various ceramic demolition wastes as substitutes for calcined clay within the LC3 framework. In this context, the present research stands at the forefront of exploration, aiming to assess the suitability of terracotta and porcelain demolition wastes as alternatives to conventional calcined clay.

This research carries the potential to offer a cost-effective and environmentally conscious alternative to natural raw materials for LC3 production, thereby reducing the industry's ecological footprint. The findings are anticipated to pave the way for sustainable and efficient cement production, aligning with the demands of a rapidly evolving world with an increasing focus on eco-friendly practices and resource conservation.

3. Materials

Two different types of demolition waste ceramics shown in Fig. 1 were used in the study: the terracotta roof tiles (RT) and the mix of white ceramic tiles and sanitary ware (RP). The materials were collected on
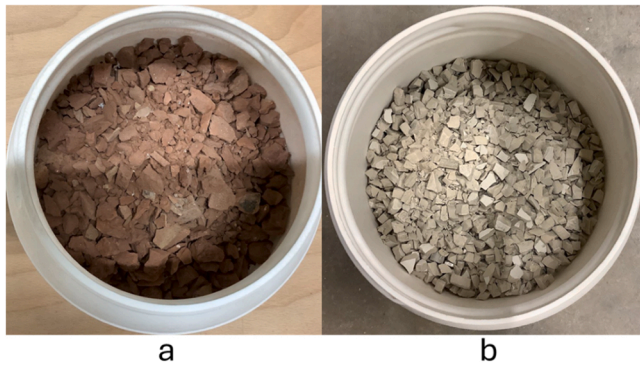
Fig. 1. Demolition waste ceramics used in the study: a – terracotta roof tiles; b – mix of sanitary ware and tiles.

different demolition sites in Luxembourg and North-East of France.

After being collected, the wastes were broken with hammer to pieces of about 3 cm in size and crushed with jaw crusher. Then they were dried in natural conditions to the constancy of mass and ground in the impact mill with a bottom sieve of 2 mm mesh size. After grinding, a half of wastes was sieved on a 125 μm mesh sieve to obtain fine ceramic powders (RT and RP) and the other half was sieved on a 63 μm mesh sieve to obtain ultrafine ceramic powders (RTU and RPU).

Besides the investigated ceramics in the research, a Portland cement (OPC) CEM I 52.5 R and limestone filler (LF) provided by the French manufacturer Vicat, CEN Standard Sand according to EN 196–1 [42], and tap water were used for the mortar specimens mixing. Limestone filler was used for mixing LC3 mortars and pastes with ceramic powders named LRT, LRP, LRTU and LRPU for the mixtures of cement, limestone filler and RT, RP, RTU and RPU powders, respectively. The characteristics of the investigated materials, as well as OPC and LF are given in Section 4.1.

4. Experimental methods

4.1. Materials characterization

The mineralogical composition of investigated ceramic powders was studied using D2 PHASER X-ray diffractometer with following parameters: Cu-Kα radiation, 10 mA, 30 kV, rotation between 5° and 70° with a 0.02° 2θ step and a step time of 0.6 s and measured by Rietveld method using Profex software. An approximate calculation of the amorphous phase was carried out by the amorphous halo method using a code written in the Python language.

The physical properties of ceramic, cement and limestone powders were also studied. The pycnometer method with petrol as liquid was used to determine the specific gravity of materials. Fineness of the powders was determined by Blaine method for air permeability specific surface area measurement as well as by particle size distribution analysis obtained with HELOS & RODOS laser granulometer. The chemical composition of the materials was investigated with Wave-Dispersive BRUKER S8-Tiger spectrometer.

4.2. Evaluation of pozzolanic activity

The modified Chapelle test [43] combined with the XRD were used to estimate the pozzolanic activity of the investigated materials.

For the Chapelle test, 1 g of ceramic powder, 2 g of calcium oxide and 250 ml of distilled water were placed in a 500 ml Erlenmeyer flask. The flask with magnetic stir bar and laboratory condenser was placed on a magnetic-stirrer hot plate heated to a temperature of 85 °C. The mixture was stirred for 16 hours. After cooling to a room temperature, the solution was mixed with 250 ml of saccharose syrup (0.7 M) and stirred for 30 minutes to extract unreacted lime. Then, the mixture was

filtered on a filter paper and 25 ml of the filtrate was titrated with 0.1 N solution of hydrochloric acid using phenolphthalein as an indicator. To calculate the amount of fixed lime a blank test without pozzolan was carried out. The pozzolanic activity was calculated according to the formula:

$$m_{g \ of \ Ca(OH)2fixed} = 2 \bullet \frac{V_1 - V_2}{V_1} \bullet \frac{M_{Ca(OH)2}}{M_{CaO}} \bullet 100 \tag{1}$$

where V_1 is the volume of HCl solution consumed in the blank test, V_2 is the volume of HCl solution consumed in the pozzolan test, $M_{Ca(OH)2}$ is the molar mass of calcium hydroxide (74 g/mol), M_{CaO} is the molar mass of calcium oxide (56 g/mol).

XRD was carried out on the paste samples with 20% of ultrafine ceramic powders (RTU_20, RPU_20, LRTU_20 and LRPU_20) and w/b ratio of 0.4 after 90 days curing and drying at 50 °C until weight constancy to check if there are differences in portlandite consumption by pozzolans particles and in formation of carboaluminates.

4.3. Evaluation of fresh pastes properties

To investigate the properties of fresh pastes, the water demand for standard consistency and setting time of binder pastes were measured using a Vicat apparatus in accordance with European standard EN 196–3 [44], without immersing the pastes in water.

The dry part of the paste mixtures consisted of 0.8 parts OPC and 0.2 parts ceramic powders, and 0.7 parts OPC, 0.2 parts ceramic powders, and 0.1 parts limestone filler. The water content in the pastes was determined through the standard consistency test following EN 196–3 [44].

4.4. Evaluation of mechanical properties

The mechanical properties of the reference mortar and mortars containing ceramic powders were evaluated using flexural and compressive strength tests on 40×40×160 mm mortar bars in accordance with the ISO 679 international standard [45].

The water-to-binder (w/b) and sand-to-binder (s/b) ratios of all mixtures were set at 0.5 and 3, respectively. The content of ceramic powders in the binders was set at 10%, 20% and 30%, and the content of LF was set at 0.5 of ceramic powder weight. The design of the mortars is presented in Fig. 2. The specimens were cured in a moisture chamber at 20°C for 7, 28, and 90 days before being tested on a hydraulic press.

Compressive strength test results of reference specimens and specimens containing SCMs were used to calculate Strength Activity Indices (SAI) using the following formula:

$$SAI = \frac{R}{R_{ref}} \bullet 100\%, \tag{2}$$

where R is the average strength of the mortar bars with SCMs, R_{ref} is the average strength of the reference mortar.

4.5. Microstructure of mortar specimens

After undergoing strength tests for 90 days of curing, the fragments remaining from the mortar specimens containing 20% ultrafine ceramic powders (RTU_20, RPU_20, LRTU_20 and LRPU_20), were coated with a 4 nm layer of gold and examined with a field emission scanning electron microscope JEOL JSM-6010LA to analyse their microstructure.

4.6. Capillary absorption measurement

After 90 days curing, two specimens of each mortar containing 20% of ceramic powders as well as of reference mortar without additives were dried at 105 °C for 24 hours and immersed in water to the depth of 5 mm by the face of 40×40 mm size. The change in their mass was
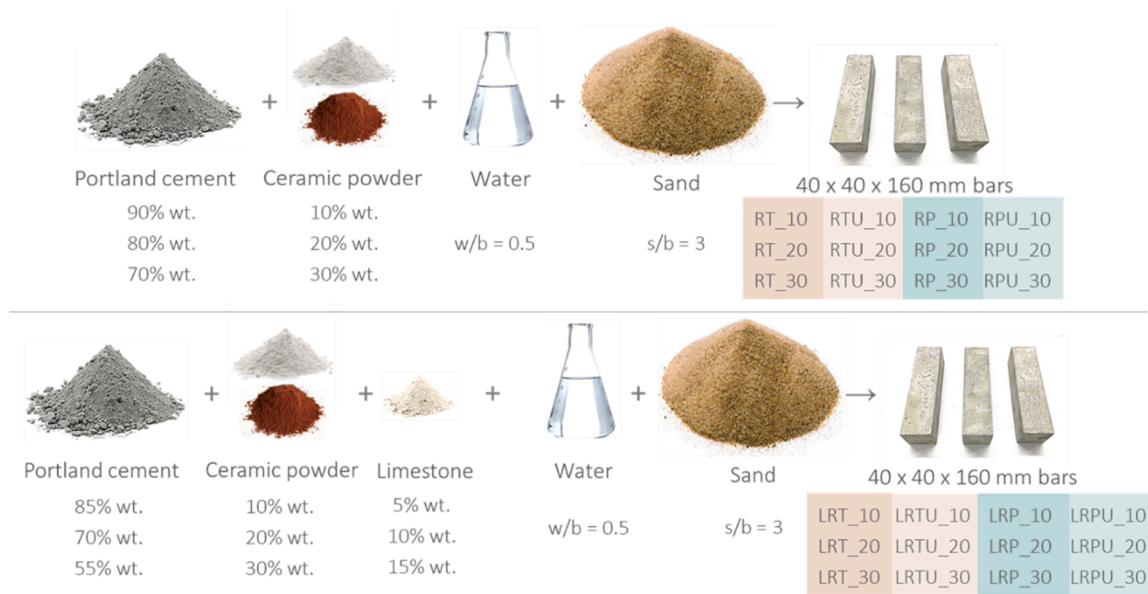
Fig. 2. Design of mortars containing ceramic powders.

measured after 0.25, 0.5, 1, 2, 4, 6, 24, 48 and 168 hours. The value of capillary absorption was calculated using the following formula:

$$C_A = \frac{m - m_0}{1600},$$ (3)

where m_0 is the mass in grams of the dry specimen, m is the mass in grams of the specimen after the required absorption time, and 1600 is the surface of the specimen's bottom in mm^2.

5. Results and discussion

5.1. Materials characterization

The chemical composition of the materials is given in the Fig. 3a. The main oxides of both ceramic samples are SiO_2 and Al_2O_3, followed by CaO, Fe_2O_3 and K_2O. The sum of contents of silica, alumina, and iron oxide, which are considered as pozzolanic oxides in RT and RP is 76.72% and 85.74%, respectively, which is higher than the minimum threshold
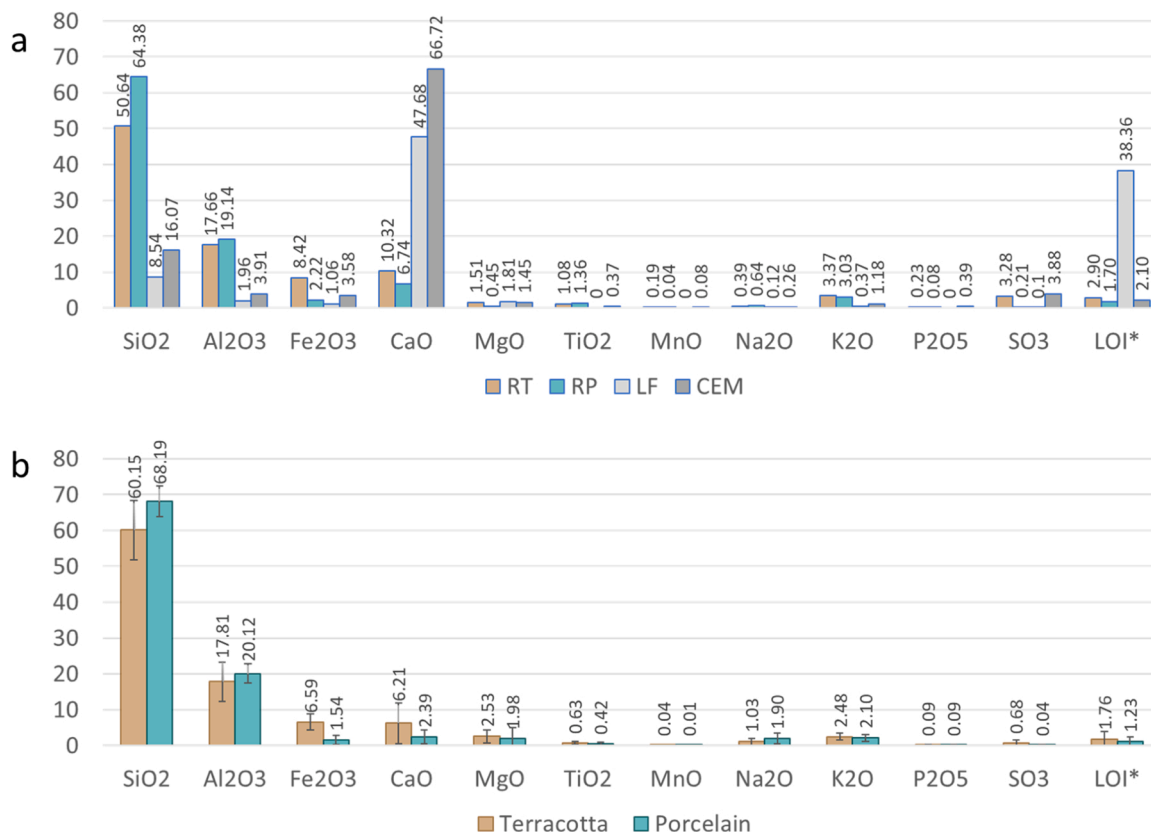

Fig. 3. Oxide composition of the investigated materials (a) and the average composition of ceramic materials according to the literature review (b), mass %.

of 70% established by ASTM C618 [46] standard for pozzolanic materials. In general, the materials investigated in this study have a composition that aligns with typical red and white ceramics, as indicated by the data in Fig. 3b, based on an analysis of 121 samples gathered from various sources in the literature. The higher content of calcium in the studied porcelain can be explained by the presence of adhering cement on the tiles.

While the chemical composition of investigated ceramics corresponds to calcined clays, it can be highlighted that the pozzolanic oxides have to be present in an active amorphous state. Additionally, the materials need to be sufficiently fine, with a minimum of 66% of particles smaller than 45 µm [46], in order to ensure high reactivity.

The X-ray diffraction patterns and calculated mineralogical composition of the investigated ceramics, given in Fig. 4 and Table 1, respectively, indicate the presence of high levels of crystalline quartz in both samples. The approximate calculation of the amorphous phase by the amorphous halo method showed that the amount of glassy phase in RP was higher than in RT and was 40% and 28%, respectively. The presence of a high content of mullite, which is formed through the recrystallization of clay minerals when they are heated above 1000 °C [49–51], in the RP and its absence in the RT indicates the difference in firing temperatures between the two types of ceramics. In general, the mineralogical composition of the studied materials corresponds to the typical composition of red and white ceramics [15,18,23,32,47,48,52–55].

The specific gravities of the LF, RT (and RTU), RP (and RPU) and cement shown in Fig. 5 were determined to be 2.70, 2.42, 2.59, and 2.95 g/cm³, respectively. After undergoing the same grinding and sieving procedure, it was observed that the terracotta powders were finer compared to the porcelain powders (Fig. 5). Thus, the specific surface area of RT and RTU was measured as 5239 and 6249 cm²/g, respectively, while the corresponding values for RP and RPU were 3608 and 5543 cm²/g, respectively. This difference can be attributed to the greater hardness of porcelain, which makes it more challenging to grind. The specific surface area of cement and limestone filler was 4887 and 10465 cm²/g, respectively.

Laser granulometry also confirmed these findings (Fig. 6). The particle size distribution curves revealed that the D10, D50, and D90 values for RT were determined as 1.55, 19.82, and 85.23 µm, respectively, whereas for RP, they were determined as 3.33, 31.97, and 95.71 µm, respectively. As for the ultrafine ceramic powders, the corresponding values were 1.19, 11.09, and 38.39 µm for RTU and 2.09, 17.87, and 49.91 µm for RPU. The distribution density curves clearly show that the limestone filler has a granulometry different from the other materials and contains notably higher amount of ultrafine particles with size <10 µm, which means that it can contribute to the packing density improvement.

5.2. Evaluation of pozzolanic activity

The results of the modified Chapelle test presented in Fig. 7a indicate that RP powder does not exhibit pozzolanic activity. The amount of Ca (OH)₂ fixed by 1 g of the material is lower than the threshold of 436 mg established by Raverdy et al. [56] for materials exhibiting pozzolanic activity. All other materials exceed this threshold, although they do not reach the 700 mg/g mark set for metakaolin. Overall, the results of the Chapelle test are consistent with the data for ceramic materials collected from other studies (Fig. 7b). Unfortunately, there is currently a lack of sufficient studies in the literature regarding the pozzolanicity of ceramic powders using the Chapelle test, which prevents a reliable statistical analysis. Therefore, only four values, notably 595 mg Ca(OH)₂ given by Medeiros et al. [57] and 234, 409 and 512 mg Ca(OH)₂ given by Pereira et al. [58] could be used to characterize porcelain at this point.

It is intriguing that porcelain powders, despite having a higher amorphous phase and pozzolanic oxide content, exhibit weaker pozzolanic behaviour compared to terracotta powders. This can be attributed to the lower specific surface area of porcelain powders, which may limit the availability of active sites for pozzolanic reactions. Additionally, the high firing temperature of porcelain may contribute to reduced reactivity of the amorphous phase. When clay is heated at temperatures higher than 1000°C, kaolinite undergoes recrystallization into mullite, and the change in the composition of the amorphous phase makes it less active for pozzolanic reactions [39,49–51].

X-ray diffraction analysis of pastes containing 20% RTU and RPU after 90 days of curing, shown in Fig. 8 and Table 2, did not reveal a significant difference between samples made using RTU and samples made using RPU. As expected, the pastes with limestone filler contained more carboaluminates than the pastes without it, indicating reactions between the limestone and the pozzolanic ceramic powders. Both pastes with terracotta and porcelain contained the same amount of carboaluminates and calcite, which indicates the same degree of reaction of both ceramics with calcite. The RTU_20 paste contains 1.2% less portlandite than RPU_20. Moreover, RTU_20 includes 2.7% more amorphous phase, primarily composed, as is known, of hydration products. These two observations confirm that RTU exhibits slightly better pozzolanic activity compared to RPU.

5.3. Evaluation of fresh pastes properties

Fig. 9 demonstrates that water demand increases after the addition of terracotta powders, which aligns with findings of Pitarch et al. [39].
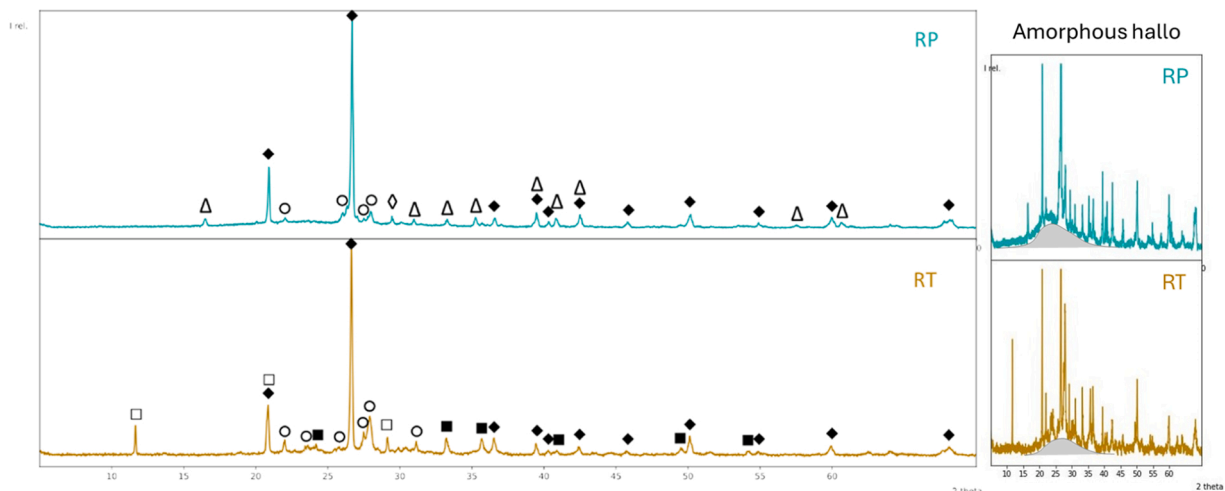

Fig. 4. XRD patterns of the investigated ceramics: ◆ - quartz, △ – mullite, ◇ - calcite, □ - gypsum, ○ - feldspars, ■ - hematite.

Table 1
Mineralogical composition of investigated ceramics, mass-%.

	Quartz	Feldspars	Mullite	Calcite	Hematite	Gypsum	Amorphous phase
RT	32.4	29.9	-	-	5.2	4.5	28.0
RP	34.9	9.3	14.1	1.7	-	-	40.0
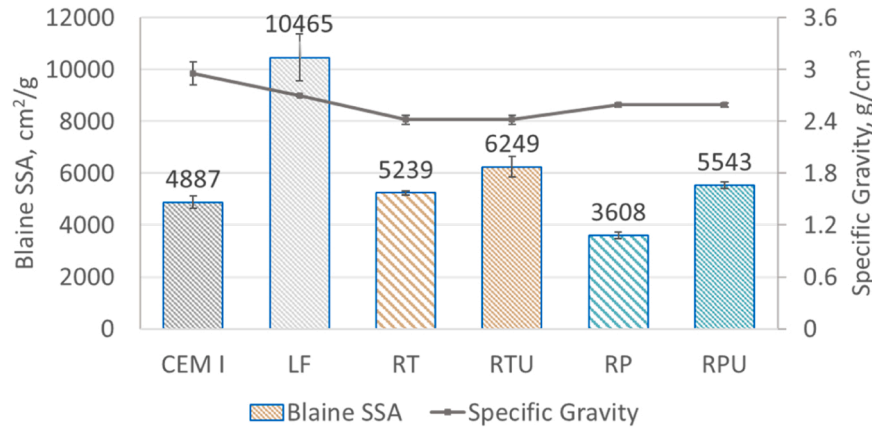
Fig. 5. Physical properties of powder materials.
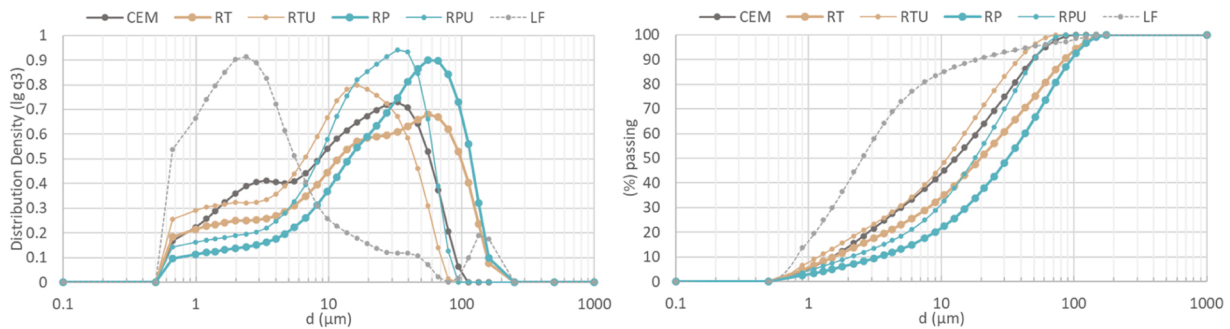
Fig. 6. Particle size distribution of powder materials.
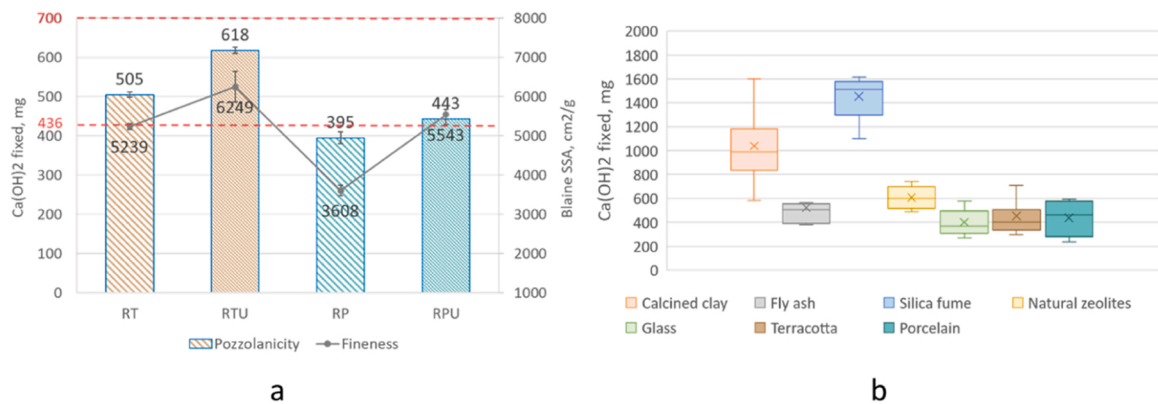
Fig. 7. Chapelle test results for pozzolanic activity: a – investigated ceramic powders; b – data collected from literature.

Pereira-de-Oliveira et al. [38] attribute this to the higher specific surface area of the ceramic powder. Conversely, RPU has almost no effect, and the addition of RP leads to a decrease in water demand due to its low specific surface area, as powders with larger particles tend to have lower water demand [59]. Additionally, the decrease in water demand when adding limestone filler to the samples containing ceramic SCM can be attributed to an increase in the packing density of the dry mixture [60, 61]. The particle distribution curves shown in Fig. 6 clearly demonstrate that the particle size distribution of the limestone filler differs significantly from that of other materials. Consequently, the fine limestone particles fill the spaces between the cement and ceramic particles, reducing the available space for water.

Regarding the setting time of cement pastes, it can be seen from Fig. 9 that the inclusion of terracotta powders resulted in an increase in
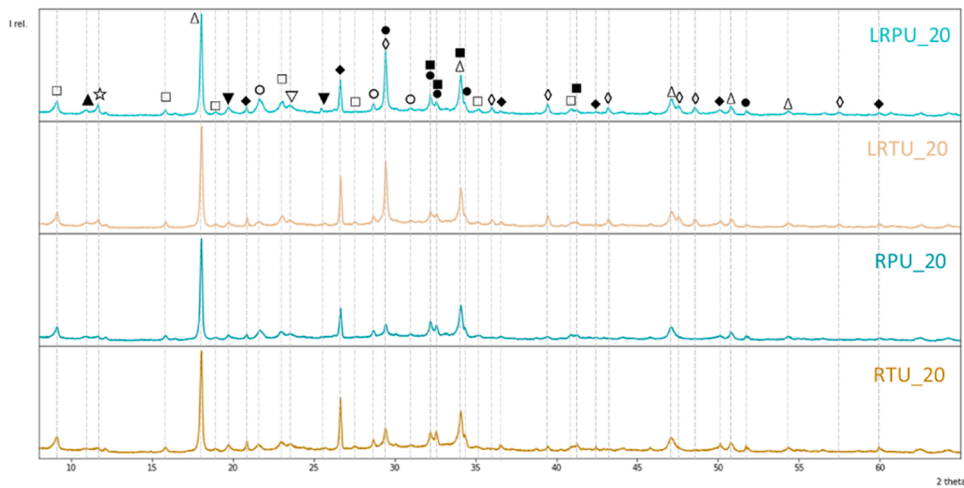
Fig. 8. XRD patterns of the pastes containing 20% of ceramic powders RTU and RPU after 90 days curing: Δ – portlandite ($Ca(OH)_2$), $\lozenge$ - calcite ($CaCO_3$), $\square$ - ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}\cdot26\ H_2O$), $\circ$ - C-S-H ($CaSiO_3\cdot H_2O$), $\bullet$ - alite (Ca_3SiO_5), $\blacksquare$ - belite (Ca_2SiO_4), $\blacklozenge$ - quartz (SiO_2), ∇ - ye'elimite ($Ca_4Al_6O_{12}SO_4$), $\blacktriangledown$ - grossite ($CaAl_4O_7$), $\star$ - monocarboaluminate ($Ca_4Al_2CO_9\cdot11\ H_2O$), $\blacktriangle$- hemicarboaluminate ($Ca_8Al_4CO_{16}\cdot22\ H_2O$).

Table 2
Mineralogical composition of the pastes containing 20% of ceramic powders after 90 days curing, mass. %.

	RTU_20	RPU_20	LRTU_20	LRPU_20
Portlandite	15.2	16.4	13.9	13.5
Calcite	1.9	1.7	6.4	6.5
Ettringite	6	6.9	6	6.4
C-S-H	5.8	6.8	6.4	6.9
Alite	6.2	6.3	4.9	4.2
Belite	6.1	6.8	5.3	5.4
Quartz	5.4	4.1	4.8	3.7
Ye'elimite	2.3	2.4	2.1	2.2
Grossite	1.2	0.9	0.9	0.9
Hemicarboaluminate	0.9	0.9	1.5	1.6
Monocarboaluminate	0.8	1.3	4.1	4.5
Amorphous phase	48.2	45.5	43.7	44.2

both the initial and final setting time, while the addition of porcelain powders had negligible effects on the setting time. The delay of the setting can be explained by the dilution effect of cement phases, which are responsible for the early hydration. Moreover, a significant correlation between setting time and water demand was evident. Similar delays in setting have been reported by other researchers when incorporating ceramic powders [24,41].

The addition of limestone filler had different effects on the setting time depending on the type of ceramic powder used. For pastes with porcelain powders, the inclusion of limestone filler reduced the initial and final setting time, with values of 155 and 200 minutes, respectively, observed for LRP. However, the paste LRTU exhibited the longest initial and final setting times, with values of 260 and 310 minutes, respectively. The impact of LF on cement setting time is multifaceted and depends on factors such as the amount of additive, particle size, and cement properties. On one hand, LF can accelerate setting by creating nucleation centres, thus promoting cement hydration. On the other hand, it can also retard setting due to the dilution effect of the cement.

The graphs demonstrate that LF increased the setting time of pastes containing RTU, which possesses a higher specific surface area, and had no significant effect on pastes with RT. At the same time, LF reduced the setting time of pastes with both types of porcelain powders. This can be attributed to LF acting as nucleation centres and as inert filler, facilitating cement hydration in some instances, while also potentially retarding setting due to the dilution effect of the cement.

5.4. Evaluation of mechanical properties

Fig. 10 illustrates the flexural strength of cement mortar samples incorporating ceramic powders and limestone filler after 7, 28, and 90 days of curing. The replacement of 10% of cement with terracotta powders (RT_10 and RTU_10) or 15% with a limestone-terracotta mix (LRT_10 and LRTU_10), regardless of the fineness of the ceramic powder, did not significantly impact the strength compared to the reference
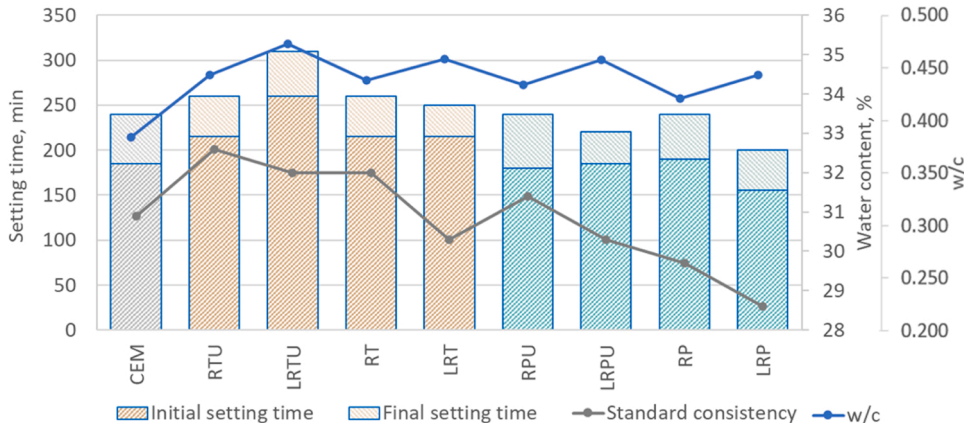

Fig. 9. Standard consistency, initial and final setting times of pastes with 20% of ceramic powders and 10% of limestone filler.
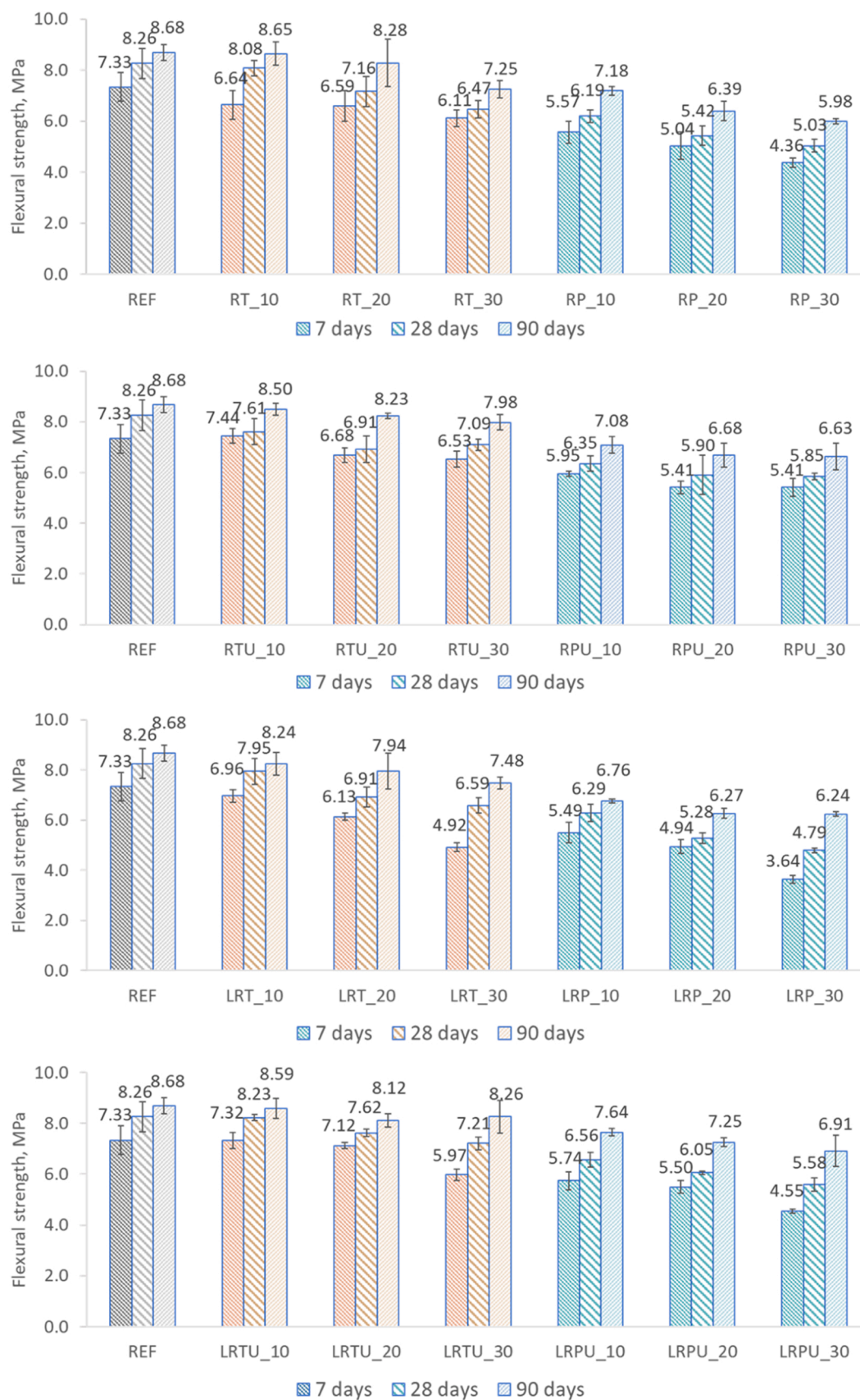
Fig. 10. Flexural strength of mortar specimens.

mortar. However, a more noticeable reduction in strength was observed with higher substitution rates. Nevertheless, after 90 days of curing, all samples containing terracotta powders exhibited good flexural strength, regardless of their fineness. The addition of limestone filler did not significantly affect the late-stage curing flexural strength, but it did decrease the early strength in high-substitution rate specimens.

In contrast, the replacement of cement with porcelain powder substantially reduced the flexural strength of cement mortars. Similar to terracotta powders, the fineness of the porcelain powders did not impact the flexural strength, and the inclusion of limestone filler only reduced strength during the early stages of curing.

The compressive strength of the samples provides insights into cement hydration and pozzolanic reactions. Generally, a higher compressive strength is expected with greater hydration extent, which typically increases with longer curing time. Fig. 11 displays the compressive strengths of both the reference and blended cement mortars after 7, 28, and 90 days of curing. The SAI values, shown in Fig. 12, provide a clear comparison between the strength of the OPC control mortar and the blended mortars. Over time, the SAI values of all specimens containing ceramic powders increase, indicating longer compressive strength development compared to the reference specimen. This can be attributed to the occurrence of pozzolanic reactions, which primarily take place during the later stages of hydration.

The strength development is primarily attributed to the formation of hydration products, such as calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H) [18], and in the case of limestone-ceramic specimens, the carboaluminate hydrates. At 7 days, the reference specimen achieves the highest compressive strength among the 25 mixes of cement mortars, reaching 50.87 MPa. This is expected since the control mortar contains the highest concentration of reactive calcium silicates that react extensively with water during primary hydration. After 28 days, the strength of mortars containing 10% terracotta powders without limestone filler reaches the strength of the reference specimen of 60.22 MPa and is 60.62 and 62.74 for RT_10 and RTU_10, respectively. After 90 days of curing, the strength of RT_10, RTU_10, and RTU_20 specimens surpass that of the control sample (65.94 MPa), with values of 71.07 MPa, 73.72 MPa and 68.24 MPa, respectively. Moreover, the strength of RT_20 and RTU_30 samples nearly match that of the reference, measuring 64.20 MPa and 62.81 MPa, respectively. These results indicate that a substitution rate of 20% OPC by terracotta powder is close to an optimum choice to produce high-strength mortar. Similar substitution rates for ceramic powder have been reported in other studies [17,28,29,32,38,39], to achieve comparable mechanical properties to those of OPC.

Regarding the limestone filler, the addition of LF reduced the strength of samples compared to those containing only ceramic powders. This indicates a low pozzolanic activity of ceramics compared to traditional calcined clay, making them unsuitable for use in the LC3-50 design. However, samples LRT_10 and LRTU_10, with an OPC substitution rate of 15%, as well as sample LRTU_20, with an OPC substitution rate of 30%, exhibit fairly good strength, reaching 64.74 MPa, 68.49 MPa and 63.91 MPa, respectively, after 90 days of curing. This makes it possible to use terracotta powders in the manufacturing of LC3 with lower substitution rates. Nevertheless, all samples with porcelain powders showed unsatisfactory strength.

Based on the results obtained, it can be concluded that the following samples comply with the requirements of the EN 197–1 standard [62] for cement strength:

— Strength class 52.5: RT_10, RTU_10, RTU_20, LRT_10, LRTU_10.
— Strength class 42.5: RTU_30, LRT_20, LRTU_20.
— Strength class 32.5: RT_30, RP_10, RPU_10, LRTU_30, LRP_10, LRPU_10.

In general, the results of the compressive strength of samples containing porcelain powders differ significantly from the findings of other researchers who reported satisfactory strength values for mortars containing up to approximately 20% porcelain powder [18,19,25,33,39].

The SAI calculations shown in Fig. 12 demonstrate that the strength of samples RP_10 and RPU_10 is only 53% and 60% of the reference mortar strength after 7 days of curing, and 62% and 74% after 90 days of curing, respectively. For samples with 30% RP and RPU, these values are 37% and 43% after 7 days and 46% and 56% after 90 days. Such results indicate that the investigated porcelain powders are not suitable for use as SCM in cement production. According to the ASTM C311 standard [63], a material can be considered a pozzolanic SCM if a mortar with a cement substitution rate of 20% exhibits an SAI of at least 75% after 7 and 28 days of curing. The examined terracotta powders, regardless of their fineness, meet this requirement, while the samples containing porcelain powders do not reach the 75% threshold even at a substitution rate of 10% after 90 days of curing.

The analysis of the mineralogical composition of pastes containing terracotta and porcelain presented in Fig. 8 and Table 2, revealed no significant difference between samples containing these different types of ceramics. Therefore, such striking differences in the strength of the samples cannot be explained by the composition of the cement matrix and the difference in the reactivity of ceramic powders. To identify the reasons for the low strength of samples with porcelain, it is necessary to compare the microstructure of samples with terracotta and porcelain powders.

5.5. Microstructure of mortar specimens

Upon examining the SEM micrographs at a magnification of 200x, as shown in Fig. 13, it becomes evident that the mortar sample containing 20% RPU exhibits a considerably more porous structure compared to the sample containing RTU. At a magnification of 1000x, it becomes also apparent that the microstructure of the sample with porcelain powder is looser and contains a higher number of micropores. As previously mentioned, the X-ray diffraction analysis of paste samples containing 20% RTU and RPU (Fig. 8, Table 2) did not reveal significant differences in their mineralogy, indicating that the cement matrix of both samples is quite similar. Thus, it can be concluded that the substantial decrease in strength observed in mortars with porcelain powders, as opposed to mortars with terracotta powders, is primarily attributed not to differences in their hydration, but rather to variations in their porosity caused by other reason, which will be discussed in the Section 5.7.

The microstructure analysis of samples LRTU_20 and LRPU_20 at a magnification of 200x did not exhibit noticeable differences. However, at a magnification of 1000x, it can be observed that the pores in both samples are filled with ettringite crystals, with larger pores apparent in the LRPU_20 sample. Matschei et al. [64] explain the formation of secondary ettringite in carbonate-rich systems through the displacement of sulphate from Afm by carbonate, leading to the formation of monocarboaluminate and hemicarboaluminate. The sulphate released in this process contributes to the formation of additional ettringite, which likely fills the pores. The presence of mono- and hemicarboaluminate peaks in the X-ray diffraction pattern given in Fig. 8 supports this explanation.

Considering these observations, it is evident that further comprehensive investigations are necessary to study the long-term strength and microstructure development of LC3 samples beyond 90 days of curing, as well as the durability aspects of concrete structures using the new binder. The filling of pores with ettringite crystals can potentially enhance the properties of hydrated cement; however, the extensive delayed crystal growth can create internal stresses and lead to the formation of microcracks. Therefore, a more detailed examination is warranted to gain insights into these phenomena.

5.6. Capillary absorption measurement

The results of the capillary absorption test on specimens containing 20% ceramic powders after 90 days of curing align with the observations from the SEM micrographs. Fig. 14 illustrates that the addition of
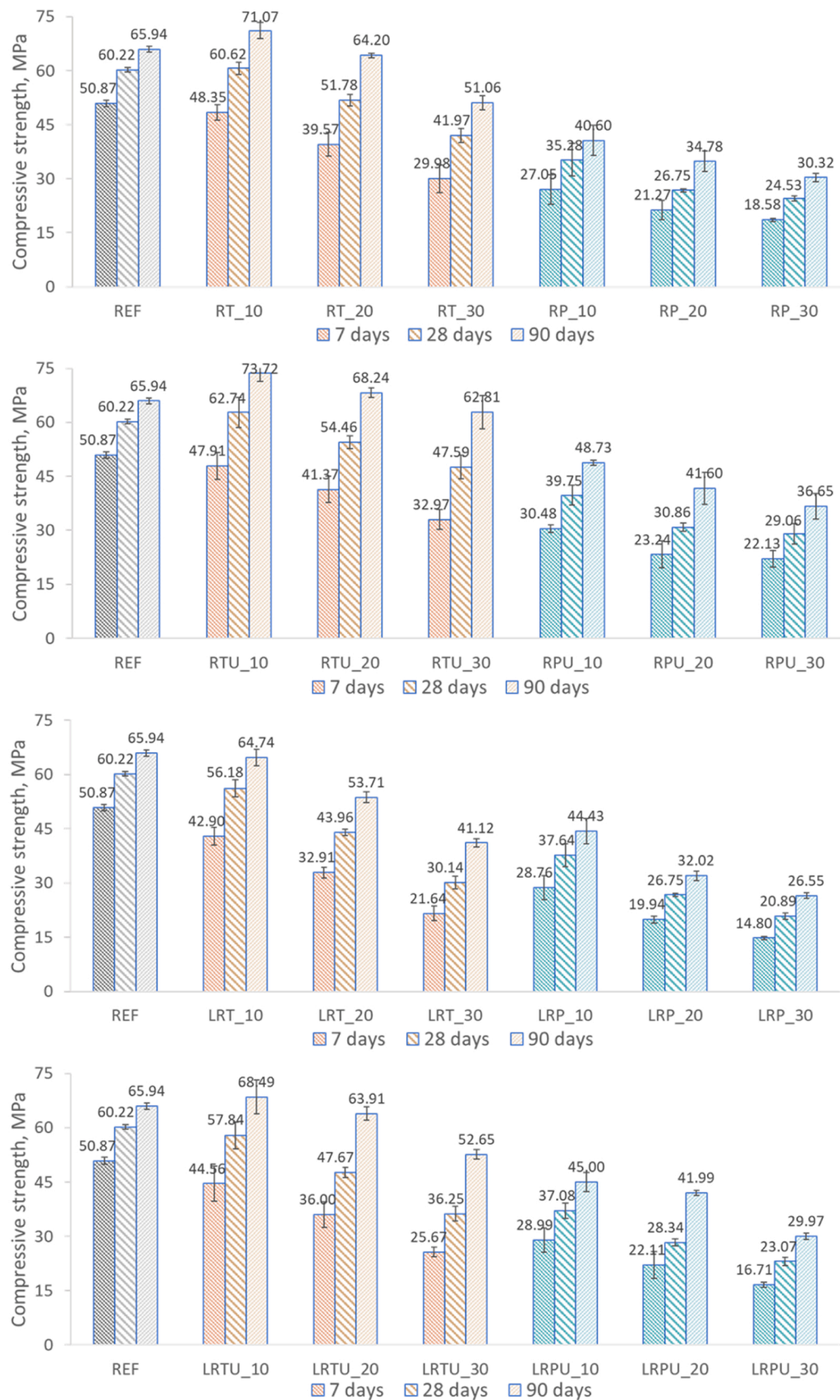
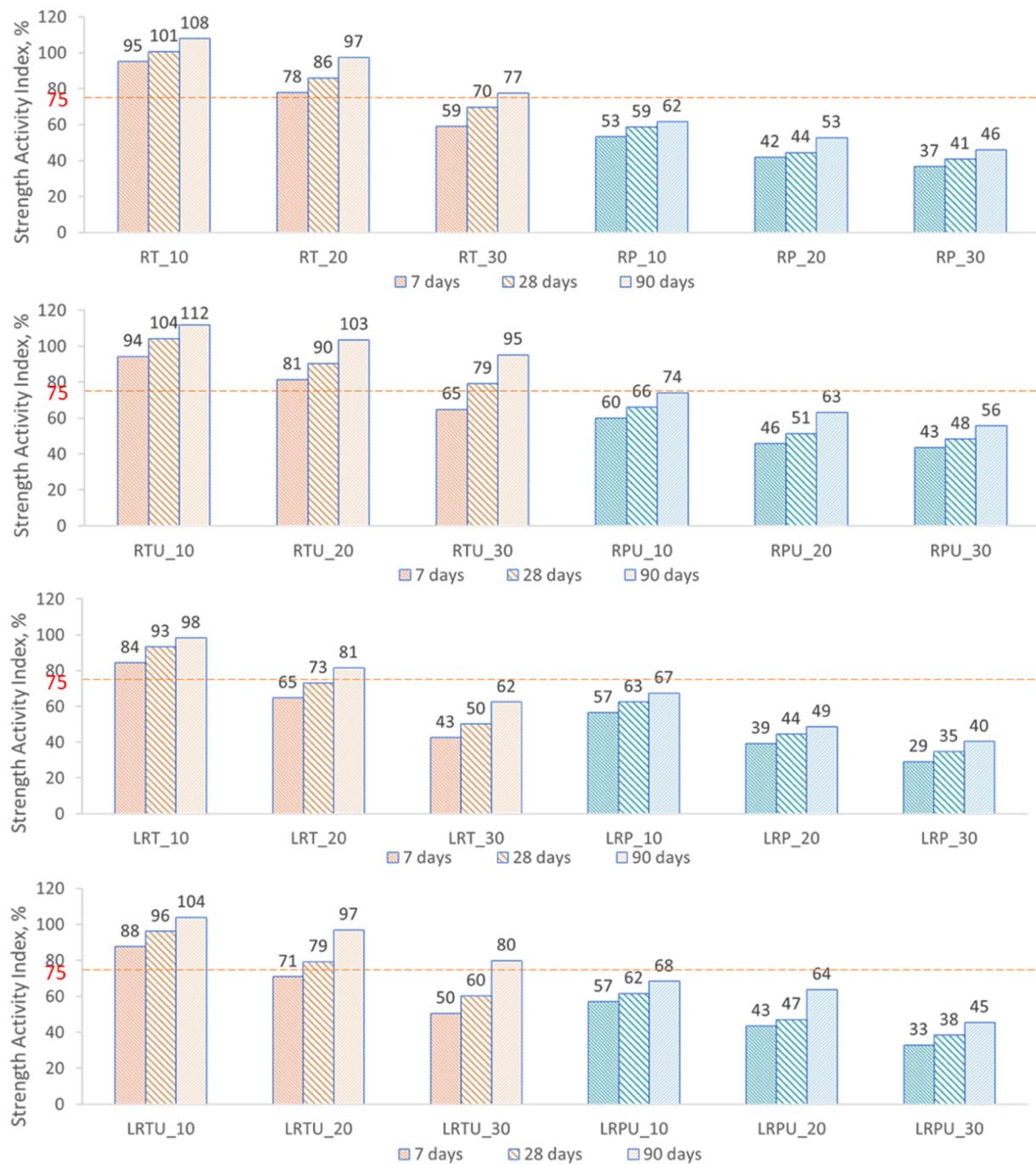**Fig. 11.** Compressive strength of mortar specimens.

Fig. 12. Strength Activity Indices of mortar specimens.

limestone filler decreases the absorption of the samples, indicating a reduction in open porosity. This reduction is attributed to the filling of pores with secondary ettringite crystals, as revealed by the SEM results. Furthermore, it is evident that the fineness of the ceramic powders influences the capillary absorption of the mortars, with finer powders leading to lower absorption. This suggests that finer powders have a more favourable impact on the microstructure of the cement mortar, primarily due to the filler effect and the increased availability of pozzolanic particles for reactions during cement hydration.

Samples containing porcelain powders exhibit significantly higher absorbance compared to samples with terracotta powders, confirming the SEM findings of higher porosity in mortars with porcelain. Additionally, it should be noted that the samples containing terracotta powders show comparable to the reference sample capillary absorption.

Other authors report a decrease of capillary absorption of samples containing ceramic powders due to pozzolanic reactions and the filler effect, which leads to pore refinement and compaction of the microstructure [22,28,29,31].

5.7. Discussion of the reasons for the high porosity of mortars containing porcelain powders

Based on observations, it was found that cement mortar samples containing porcelain powders had significantly higher porosity compared to samples containing terracotta powders. Since the mineralogical composition of pastes containing terracotta and porcelain did not show significant differences, it can be assumed that the porcelain used in the study contained air-entraining agents. Such agents are
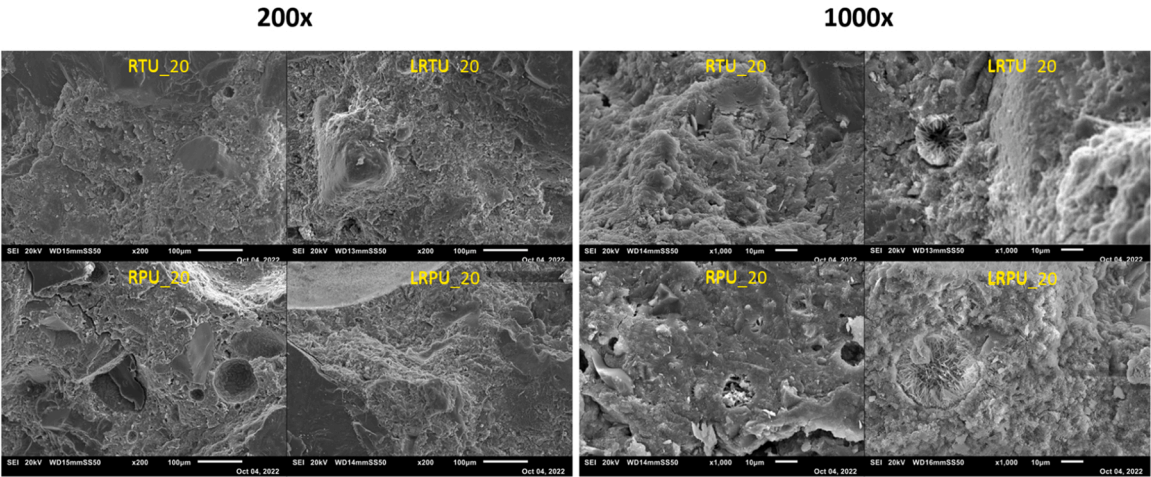
Fig. 13. SEM pictures of mortars with 20% of ceramic powders after 90 days of curing at magnification of 200x (left) and 1000x (right).
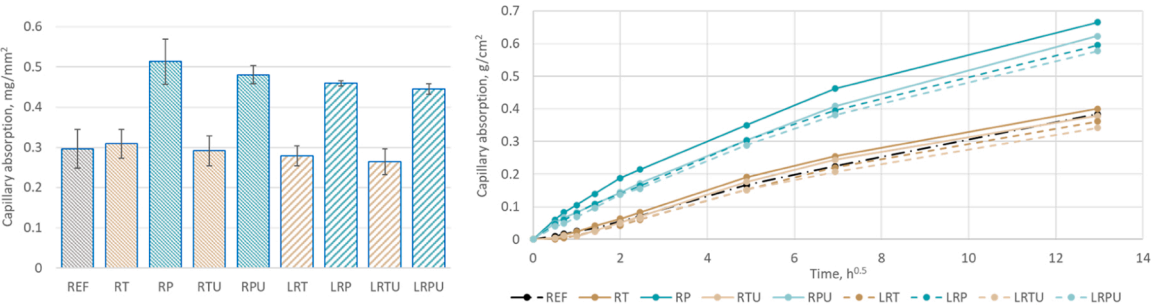

Fig. 14. Capillary absorption of reference OPC mortar and specimens containing 20% ceramic powders after 90 days of curing and 7 days of immersion in water.

substances like resins, fats, fatty acids, and surfactants that create stable foam when mixed with water [65].

The material under study was sanitary porcelain and faience, collected directly during demolition and repair work after prolonged use, without being washed before mixing with cement. It can be assumed that during lifespan the porcelain was contaminated with various surfactants, including soaps, shampoos, and cleaning products, which gradually penetrated the cracks in glazes and pores in ceramics, as well as remained on the surface of tiles and sanitary ware. Indeed, during the mixing of cement mortars containing porcelain powders, excessive bubble formation was observed, which was not observed when mixing mortars with terracotta powders (Fig. 15a and Fig. 15b).

To confirm the presence of surfactants in porcelain powders, a simple foaming test was performed [66]. In this test, 6 g of RPU and RTU
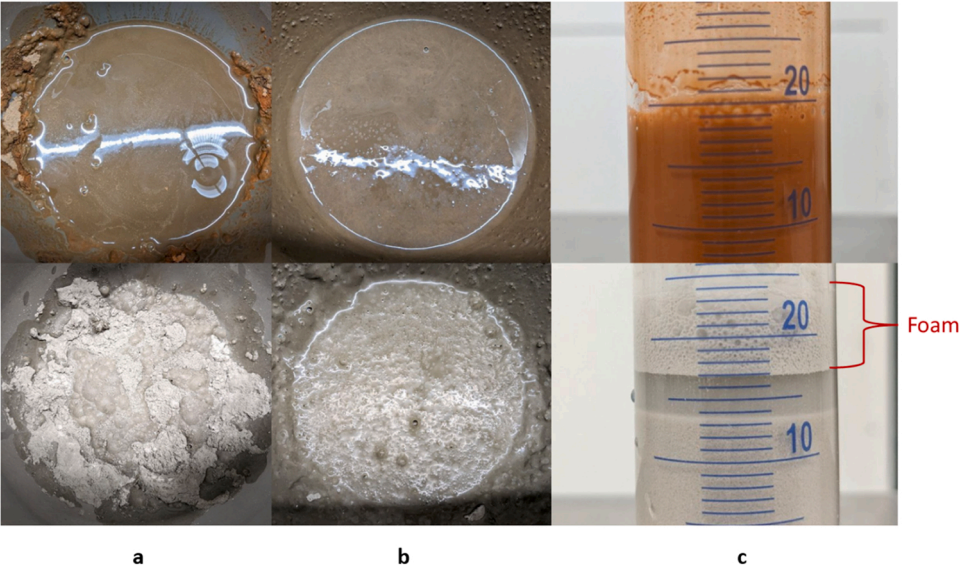

Fig. 15. Foam formation: a – after contact of cement-ceramic mix with water, b – after mixing of cement-ceramic mix with water for 30 s, c – after shaking of ceramic powders with water for 30 s.

powders were mixed with 15 ml of water and shaken for 30 seconds. After the manipulation, a small number of large bubbles, which disappeared after a few seconds, formed on the surface of the mixture with RTU. On the other hand, a stable foam, which disappeared only after a day, formed on the surface of the mixture with RPU (Fig. 15c). This confirms the presence of air-entraining agents in the used porcelain, which significantly deteriorated the mechanical properties of cement mortars, which was not observed by other researchers.

To further confirm this theory and assess the effectiveness of porcelain powder purification in improving the mechanical properties of cement mortar, thermal purification of ultrafine porcelain powder (RPU) was conducted in a muffle furnace at 500 °C for 2 hours. Subsequently, cement mortar samples were prepared following the above-described scheme with a substitution rate of 20%. The results of strength tests after 7 and 28 days of curing, as shown in Fig. 16, demonstrated a noticeable improvement in the mechanical properties of the mortar specimens. Thus, on the 7th day, the flexural strength increased from 5.41 MPa for the mortar with untreated porcelain powder (RPU) to 6.61 MPa for the sample with calcined porcelain powder (RPU500). By the 28th day, this value had increased from 5.90 MPa to 7.47 MPa. The compressive strength after 7 days of curing increased from 23.24 MPa to 42.67 MPa, corresponding to SAI values of 46% and 84%, respectively. After 28 days of curing, the compressive strength increased from 30.86 MPa to 53.92 MPa, corresponding to SAI values of 51% and 90%.

These results can infer that the calcination of porcelain powder at 500 °C for 2 hours eliminates organic surfactants, rendering the powder suitable for use as a pozzolanic SCM since specimens containing 20% calcined ultrafine porcelain powder meet the SAI requirement of 75%. It means that the contamination of sanitary ware and tiles with surfactants during their use necessitates pre-washing and drying or calcination of the material before it can be used in the production of blended cement for normal use. However, these procedures require a substantial amount of water and energy, which contradicts the goals of eco-cement production. Nonetheless, since air-entraining agents are introduced into the cement mass during the production of cellular concrete, it can be assumed that sanitary porcelain without pre-treatment can be used as a SCM for the manufacture of this type of building material. However, comprehensive studies on the properties of the new cellular concrete are necessary to validate this assumption.

6. Conclusions

This study presents an assessment of the feasibility of using various types of ceramic demolition waste, including terracotta roof tiles and sanitary porcelain, as alternatives to calcined clay in eco-cement production. The chemical and mineralogical composition of the investigated materials aligns with typical terracotta and porcelain, making them representative of these ceramic types. The analysis has highlighted the nuanced factors influencing the comparative assessment, including the presence of impurities and the powder fineness. The following conclusions can be drawn from the obtained results:

(1) Both ceramic waste materials display a chemical composition abundant in pozzolanic oxides, namely SiO_2, Al_2O_3, and Fe_2O_3, accounting for 76.72% in terracotta and 85.74% in porcelain. Their mineralogical characteristics align with typical red and white ceramics. Terracotta powders had finer granulometry than porcelain powders due to differences in material hardness.

(2) Despite having a higher amorphous phase and pozzolanic oxide content, porcelain powders exhibited weaker pozzolanic behaviour than terracotta powders. According to the Chapelle test results, terracotta powders bound 505 and 618 mg $Ca(OH)_2$ for RT and RTU, respectively, while porcelain powders bound 395 and 443 mg $Ca(OH)_2$ for RP and RPU, respectively. This lower reactivity can be attributed to the lower specific surface area of porcelain powders and the high firing temperature.

(3) The substitution of up to 20% of OPC with terracotta powders had a minimal impact on the strength of the mortars. The SAI at 90 days of curing was 97% for RT_20 and 103% for RTU_20, indicating their potential as SCMs. However, the inclusion of porcelain powder significantly reduced the strength of the specimens, with the maximum SAI at 90 days of curing being 62% for RP_10 and 74% for RPU_10, rendering them unsuitable as SCMs.

(4) The incorporation of limestone filler led to a reduction in strength compared to samples containing only ceramic powders. Nevertheless, LC3 samples containing terracotta powders with up to a 30% substitution rate of OPC demonstrated relatively strong performance, achieving a SAI of 81% for LRT_20 and 97% for LRTU_20 after 90 days of curing. This suggests the potential for utilizing terracotta powders in the production of LC3 with lower substitution rates. However, all specimens containing porcelain powders exhibited inadequate mechanical properties, with the maximum SAI at 90 days of curing being 67% for LRP_10 and 68% for LRPU_10.

(5) The porous microstructure and poor mechanical properties of mortars containing porcelain powders are attributed to the presence of surfactants in sanitary porcelain, which was proved by foam-formation test.

(6) SEM microstructure analysis of LC3 specimens after 90 days of curing showed the presence of secondary ettringite crystals in the pores of all samples.

(7) The calcination of sanitary porcelain powder contaminated with surfactants at 500 °C leads to its purification and improves the compression strength of mortars with 20% waste powder from 23.24 MPa to 42.67 MPa after 7 days of curing and from 30.86 MPa to 53.92 MPa after 28 days of curing.

These findings offer valuable insights for the cement and processing industries regarding the treatment and application of different ceramic demolition waste materials in eco-cement production, including blended pozzolanic cements and LC3 formulations. Moreover, this research has unveiled promising avenues for further study, including the analysis of cement mixtures containing different ceramic powders in terms of their durability, long-term strength development, and
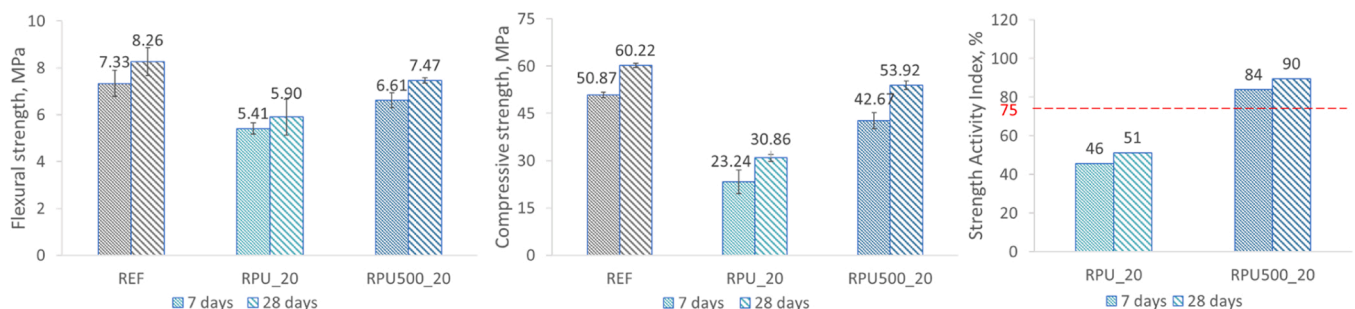

Fig. 16. Flexural and compressive strength and Strength Activity Indices of mortar specimens containing 20% untreated and calcined ultrafine porcelain powders.

microstructure characteristics at advanced stages of curing. Additionally, it is important to conduct economic and environmental assessments related to the purification of porcelain waste for its integration into cement production processes. Since the comparative assessment presented in this study is influenced by various factors, including the source and type of ceramic waste, pre- and post-grinding preparation, powder fineness and resulting surface area, future statistical research should delve into these factors to enhance the understanding of their impact on properties of final binder, contributing to the ongoing evolution of eco-cement production.

CRediT authorship contribution statement

Anna Tokareva: Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Sinan Kaassamani:** Writing – review & editing, Validation, Resources, Methodology, Conceptualization. **Danièle Waldmann:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

The data that has been used is confidential.

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