Influence of particle size on the pozzolanic reactivity of waste clay brick accessed by R^3 test

Influência da granulometria sobre a reatividade pozolânica do resíduo de bloco cerâmico utilizando Teste R^3

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Abstract

The inclusion of wastes of clay brick (WCB) as a supplementary cementitious material (SCM) demands a processing process guided by the reactivity of the material. In this sense, the present study evaluated the influence of particle size on the pozzolanic reactivity of WCB. WCB of five distinct particle size distributions was compared to metakaolin, natural clay, and waste of clay tile (WCT). The assessment of pozzolanicity was based on the R^3 reactivity test, using isothermal calorimetry and quantification of bound water content, in addition to the compressive strength and analysis by XRD/Rietveld of composite cement pastes type CPII-Z and CPIV. Reducing the $D_{90\%}$ diameter of the WCB from 33 to 11 µm promoted an increase of 50% in pozzolanic reactivity. However, for $D_{90\%}$ diameters below 11 µm, the increase in reactivity (<2%) may not justify the costs associated with prolonging the grinding process. The results highlighted the feasibility of using the R^3 test and the importance of beneficiation guided by reactivity, aiming to optimize the recycling potential of new materials in the production of composite cements.

Keywords: Ceramic waste. Supplementary cementitious materials. Pozzolanic reactivity. Composite cements.

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Resumo

A inclusão do Resíduo de Bloco Cerâmico (RBC) como Material Cimentício Suplementar (MCS) demanda um processo de beneficiamento orientado pela reatividade do material. Nesse sentido, o presente estudo avaliou a influência do tamanho de partículas sobre reatividade pozolânica do RBC. O RBC, com cinco distribuições granulométricas distintas, foi comparado ao metacaulim, argila natural e resíduo de porcelanato (RP). A avaliação da pozolanicidade baseou-se no teste R^3, utilizando calorimetria isotérmica, quantificação do teor de água combinada, resistência à compressão e análise por DRX/Rietveld das pastas de cimentos tipo CPII-Z e CPIV. A redução do diâmetro $D_{90\%}$ do RBC de 33 para 11 µm aumentou 50% na reatividade pozolânica. No entanto, para $D_{90\%}$ inferiores a 11 µm, o incremento na reatividade (<2%) pode não justificar os custos associados ao prolongamento da moagem. Os resultados evidenciaram a viabilidade na utilização do teste R^3 e a importância do beneficiamento orientado pela reatividade, otimizando o potencial de reciclagem de novos materiais na produção de cimentos compostos.

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Palavras-chave: *Resíduo cerâmico; Materiais cimentícios suplementares; Reatividade pozolânica; Cimentos compostos.*

Introduction

Cement industries emit 8% of the total CO_2 emissions from anthropogenic activities (Zhang; Yang; Wang, 2023). Supplementary cementitious materials (SCMs) enhance cement sustainability by reducing clinker consumption. Clinker, a material resulting from the calcination of limestone and clay elements at high temperatures (~1450 °C), is the main contributor to CO_2 emissions from limestone decarbonation and fuel combustion in kilns (Black, 2016; Andrew, 2018).

Ceramic waste from brick manufacturing shows potential for use as SCMs (Navrátilová; Rovnaníková, 2016). It is estimated that ceramic brick production in Brazil is approximately 4.7 billion units per year, with around 30% of this total being discarded as demolition waste or in landfills (Awoyera *et al.*, 2018; Chang *et al.*, 2023; Cherene *et al.*, 2023; IBGE, 2021). Previous research has investigated finely ground ceramic brick waste (WCB) as SCMs. When combined with slag, WCB prolonged the induction period during cement paste hydration (Zhao *et al.*, 2022). This behavior was attributed to the reduced CaO concentration in WCB (1.9%) compared to slag (43.7%) and the difference in the content of amorphous phases, which stimulated the dissolution of Ca^{2+} ions in the alkaline environment. Incorporating WCB improved chloride attack resistance in mortars containing chemical admixtures and allowed for up to 40% clinker substitution while maintaining compressive strength at 28 days (Toledo Filho *et al.*, 2007). This effect was associated with pore refinement promoted by the pozzolanic reaction of SCMs. WCB was also used to produce ternary cements combined with marble waste as a source of carbonates (Costa; Gonçalves, 2021). Ternary cement pastes with 50% clinker replacement achieved at least 70% of the compressive strength of systems containing Brazilian high early strength Portland cement (type CPV-ARI) at 28 days.

The pozzolanic properties of WCB stem from the activation of clay minerals during the thermal treatment in brick production, promoting characteristics similar to those of calcined clays (Gonçalves, 2007; He *et al.*, 2021). Although thermal activation enhances the reaction with calcium hydroxide in cement paste, the particle size distribution of the material also plays a crucial role in determining the pozzolanic reactivity of SCMs (Navrátilová; Rovnaníková, 2016).

Previous studies explored the optimization of grinding alternative raw materials, considering their physical properties (Benezet; Benhassaine, 2009; Costa; Gonçalves, 2022). Benezet and Benhassaine (2009) demonstrated the effect of SiO_2 powder particle size distribution during the pozzolanic reaction. The authors reported an enhancement in calcium silicate hydrate (C-S-H) formation. They classified the fractions as fast reactive nanopowder (between 0.1 µm and 1 µm), reactive micropowder between 28 and 90 days (from 1 µm to 10 µm), reactive mesopowder between 90 and 365 days (from 10 µm to 100 µm), and non-reactive granular powder (above 100 µm). Specific grinding energy and grindability index are important parameters for considering aspects such as energy consumption during milling and specific surface area variation (Costa; Gonçalves, 2022). However, beneficiation can be improved through complementary investigations using pozzolanic reactivity as decision criteria. Conventional methods for determining the pozzolanic reactivity of SCMs usually include the production of mortar or concrete specimens for compressive strength evaluation after 28 days. This approach implies a high consumption of raw materials and analysis time and requires greater infrastructure available for curing. In this sense, applying accelerated methods can optimize the definition of the optimal grinding condition, reducing resource consumption and expanding the potential for producing sustainable materials containing industrial byproducts.

The Rapid, Relevant, and Reliable Test (R^3 test) is an innovative method employed to assess the pozzolanic reactivity of SCMs in an accelerated system, providing results within 24 hours (Basto; Lima; Melo Neto, 2023; Snellings *et al.*, 2019). This technique enables the analysis of the pozzolanic reaction of new SCMs in a controlled environment, simulating the chemical interaction with Portland clinker and its hydrated products (Weise; Ukrainczyk; Koenders, 2023). The R^3 test has been successfully applied and validated in assessing the pozzolanicity of sugarcane bagasse ashes (Basto; Lima; Melo Neto, 2023), ground granulated blast furnace slags (Blotevogel *et al.*, 2020), calcined clays with different kaolinite contents (Avet *et al.*, 2016), and metakaolin (Weise; Ukrainczyk; Koenders, 2023). However, using the R^3 test to evaluate the pozzolanic reactivity of WCB according to its particle size distribution remains unexplored in the literature. Therefore, this study aims to evaluate the effects of particle size on the pozzolanic reactivity of ceramic brick waste used as SCMs. The investigation was divided into two steps. The first involved the application of the rapid testing method (R^3 Test), and the second step utilized the conventional method (compressive strength and XRD/Rietveld) applied to two types of composite cements.

Experimental methodology

Materials

The materials used in this study were wastes of ceramic brick (WCB) and clay tile (WCT), metakaolin (MK), natural clay, and Brazilian high early-strength Portland cement (CPV-ARI). The CPV-ARI was used as a source of Portland clinker, as it represents an alternative with the lower content of mineral additions available in the market. The material was purchased from suppliers in Salvador (Brazil). The WCB was obtained from a red ceramic brick factory in Alagoinhas (Brazil). The material comprises 22.35% quartz, 1.36% mullite, 0.98% hematite, and 75.31% non-crystalline phases. The WCT was obtained from a tile flooring distributor in Salvador (Brazil). The SCM is composed of 29.08% quartz, 7.45% mullite, 7.27% sillimanite, and 56.20% non-crystalline phases. The MK was acquired from regional suppliers, and the natural clay was obtained from grinding in a planetary ball mill PM 10 (Retsch) at a rotation speed of 2 rpm for 2 minutes, as detailed in previous investigations (Costa; Gonçalves, 2022; Oliveira *et al.*, 2022).

Methods

Particle size analysis by laser diffraction

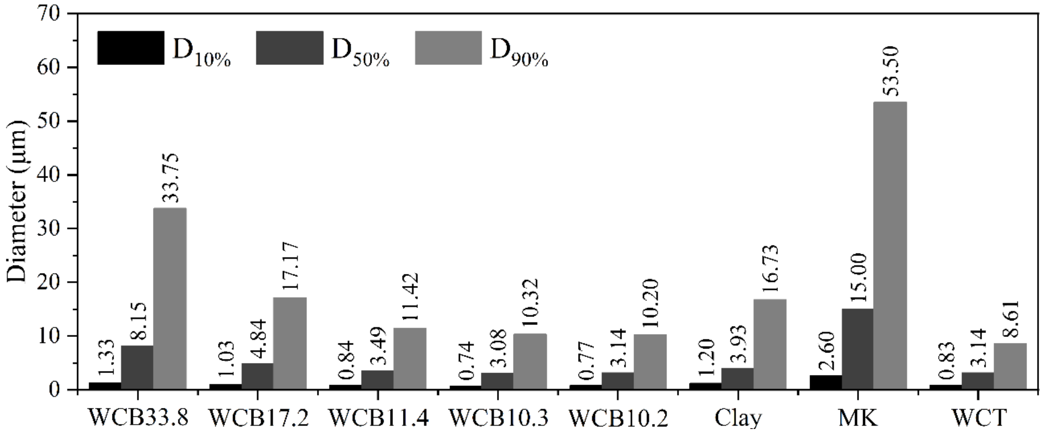
Laser diffraction was used to verify the particle size distribution of the materials to assess their influence on pozzolanic reactivity. The samples were analyzed using the particle size analyzer Mastersizer 3000 (Malvern Instruments). Figure 1 shows the $D_{10\%}$, $D_{50\%}$, and $D_{90\%}$ diameters of the materials. Samples containing WCB were named according to the quantified $D_{90\%}$ diameter.

R³ test by isothermal calorimetry and determination of bound water content

The R³ test is used to assess the pozzolanic reactivity of SCM by quantifying the bound water content in the mixture, heat release during hydration, and consumption of $Ca(OH)_2$ (Avet *et al.*, 2016). The method combines analytical purity reagents and the SCM into a paste, simulating the chemical conditions during cement hydration. The mixture for the R³ test consisted of 10 g of SCM, 30 g of $Ca(OH)_2$, 5 g of $CaCO_3$, and 54 g of alkaline solution. The alkaline solution was prepared by dissolving 4 g of KOH and 20 g of K_2SO_4 in 1 L of deionized water. The mixtures were conditioned and produced according to the procedure specified in a previous study (Avet *et al.*, 2016). The pastes were evaluated by isothermal calorimetry using the TAM Air equipment (TA Instruments) for 24 hours at 40 °C, following C 1897 standard (ASTM, 2020).

The mixtures for the R³ test were also analyzed for the mass variation associated with the loss of free and bound water during heating up to 200 °C. For this purpose, after 24 hours of production, about 5 g of the pastes were conditioned in alumina crucibles and heated in an oven for 2 hours at 40 °C to remove free water. Then, the samples were conditioned at 200 °C for 2 hours to determine the bound water content. The mass loss between 40 and 200 °C is associated with the presence of hydrated products, including calcium silicate hydrate (C-S-H), calcium trisulfoaluminate hydrate (Aft or ettringite), calcium aluminosilicate hydrate (C-A-S-H), monocarboaluminate (Mc), and hemicarboaluminate (Hc), which reflect on the pozzolanic reactivity of SCM.

Figure 1 – **Diameters $D_{10\%}$, $D_{50\%}$, and $D_{90\%}$ of the SCM particles, designated based on the quantified $D_{90\%}$ for waste of clay brick (WCB) - MK represents metakaolin, and WCT refers to waste of clay tiles**



Production of composite cements

To assess the potential development of commercial composite cements containing pozzolans, Portland cements types CPII-Z and CPIV were produced according to the Brazilian standard NBR 16697 - Portland Cement - Requirements (ABNT, 2018). CPII-Z was produced by combining 86% CPV-ARI and 14% SCM. For CPIV, 50% CPV-ARI and 50% SCM were used (Table 1). In the substitution of clinker by CPV-ARI, the carbonate content in the material (~11%) was considered. The substitution rates adopted fully comply with regulatory requirements, as the percentage of carbonate material is up to 15% for CPII-Z and up to 10% for CPIV (ABNT, 2018). The composite cements were homogenized for 5 minutes in a planetary ball mill model PM 100 (Retsch) at 200 rpm. The jar was filled with 100 g of raw materials and 15 stainless steel balls (5 mm). The pastes were produced by mixing 60 g of composite cement and 33 g of deionized water using a Hamilton Beach mixer (GM20). The samples were molded into small cylindrical specimens (2 x 4 cm) and subjected to curing in water saturated with $Ca(OH)_2$ starting from 24 hours.

Compressive strength

The evaluation of compressive strength was used to verify the influence of the pozzolanic reactivity of SCM on the mechanical properties of composite cement pastes. The specimens were analyzed at 28 days using an HD-20T hydraulic press (Contenco), applying a loading rate of 1 mm/min.

X-ray diffractometry (XRD) and quantitative analysis by the Rietveld method

XRD was used to identify the mineralogical composition of the cement pastes at 28 days. For this purpose, the pastes were analyzed using a D8 Advance diffractometer (Bruker AXS) with Cu Kα radiation, a radius of 280 mm, and a wavelength of 0.154 nm at 21 °C. The X-ray tube was operated at 40 kV and 40 mA. The cement pastes were sealed in plastic packages immediately after mixing. After 28 days of curing (21 °C), the samples were manually ground in a porcelain mortar until they passed entirely through a 75 µm opening standard sieve. The pastes were then directly analyzed without interrupting hydration. The XRD/Rietveld analysis was performed on two selected samples of composite cements based on the pozzolanic reactivity test using the R³ method. The ratio between the pozzolanic reactivity of WCB and the milling time was used as a decision criterion. The diffraction patterns were obtained from 5 to 45° (2θ) with a step of 0.02 °/s and sample rotation at 15 rpm. Phase quantification was determined by the Rietveld method using GSAS II software version 3913 (Toby; Von Dreele, 2013) and the ICSD (Inorganic Crystal Structure Database).

Results and discussions

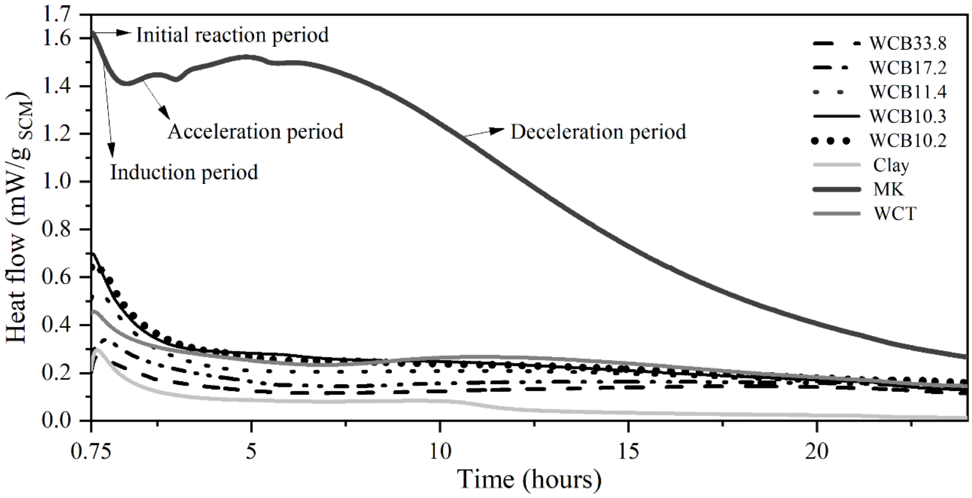
Pozzolanic reactivity by R³ test

The results of the mixtures characterization by the R³ test are presented in Figures 2, 3, and 4. Samples containing WCB were named according to the $D_{90\%}$ diameter quantified by laser diffraction. Figure 2 illustrates the evolution of the heat flow curves of the pastes, normalized by the mass of SCM and obtained by isothermal calorimetry. The heat rate as a function of time during the hydration of SCM pastes can be divided into four main steps: initial reaction, induction, acceleration, and deceleration period (Hou *et al*., 2013; Zhao *et al*., 2020). In this study, these steps are more evident in the system containing MK due to its high reactivity (Figure 2). For all samples, the peak corresponding to the onset of the reaction occurred around the first two hours of hydration (Zunino; Scrivener, 2021). In these systems, reactions are anticipated, unlike conventional systems using Portland cement, since pozzolanic reactions occur late, allowing their evaluation only at 28 days (Hewlett; Liska, 2017). For pastes with WCB, the heat flow was higher for samples with reduced particle size (WCB11.4, WCB10.2, and WCB10.3). Previous studies reported that reducing the particle size of ceramic wastes can accelerate hydration in these systems (Zhao *et al*., 2020). Additionally, the dehydroxylation resulting from clay minerals makes them more reactive in alkaline environments (Yoon *et al*., 2022). Between 2 and 5 hours, the curve of the system containing MK reached a second peak (~1.5 mW/g_{SCM}). Similar behavior was observed in calcined clay with ~50% amorphous material (Yoon *et al*., 2022). However, the heat flow rate obtained was approximately three times higher (~4.8 mW/g_{solids}) than the value reached for the MK investigated in this study. Such an effect can be attributed to the formation of hydrated products such as the C-A-S-H phase (Avet *et al*., 2022). The WCB, WCT, and natural clay did not show an identifiable second peak but a broad and smooth curve after the first peak. The decrease in heat flow after 10 h indicates the onset of the deceleration period in the systems. In this case, hydration continues but at a slower rate, tending to much lower values. Over the 24-hour reaction, natural clay showed a lower heat flow rate, considered the least reactive SCM among all materials analyzed in this research.

Table 1 – **Composition of composite cements. Supplementary cementitious materials (SCM): natural clay, metakaolin (MK), and wastes of clay tile (WCT) and clay brick (WCB)**

Compounds (%)	CPV-ARI	CPII-Z	CPIV
Clinker and sulfates	88.88	76.44	44.44
Carbonates	11.12	9.56	5.56
SCM	--	14.00	50.00

Figure 2 – **Heat flow over the 24 hours of hydration of SCM pastes (40 °C)**



The heat flow curves can indicate the presence of pozzolanic reactivity since the precipitations of hydrated phases are mainly exothermic reactions (Brial *et al.*, 2021). The evolution of the total accumulated heat highlights the high reactivity of MK from the first minutes, reaching ~80 J/g_{SCM} in 24 hours of reaction (Figure 3). This reactivity of MK originates from its manufacturing process, which occurs through the dehydroxylation and rearrangement of kaolinitic clay minerals during calcination, forming an amorphous and pozzolanically reactive material (Sabir; Wild; Bai, 2001). Previous investigations analyzed the total heat of samples containing between 40 and 50% of metakaolin, obtaining values comparable to those reached in this study for MK (70 to 125 J/g_{SCM}) (Avet *et al.*, 2016; Li; Lei; Plank, 2022). On the other hand, natural clay showed reduced pozzolanic reactivity, indicating a lower rate of heat released over 24 hours and reaching 5.18 J/g_{SCM} at the end of the experiment. The pozzolanic properties of natural clays, in general, are promoted by thermal activation (calcination) or mechanical methods (mechanochemistry) (Ilić *et al.*, 2016; Tole *et al.*, 2022).

The reduction in particle size of WCB improved the pozzolanic reactivity of samples with $D_{90\%}$ above 11 µm (CBW33.8, CBW17.2, and CBW11.4), and for $D_{90\%}$ less than 11 µm (CBW10.2 and CBW10.3), the total heat released after 24 hours stabilized with a variation of less than 2%. In this case, the costs associated with prolonging the grinding aiming to increase the pozzolanic reactivity of the material may not be worthwhile, as the energy consumption in the mill is inherently associated with the grinding time. With the reduction in particle size, there is an increase in the specific surface area, improving the WCB reactivity, due to the enhancement of interaction between calcium hydroxide ($Ca(OH)_2$) and clay minerals, optimizing the pozzolanic reaction (Ramanathan; Tuen; Suraneni, 2022). The samples WCB10.2 (20.46 J/g_{SCM}) and WCB10.3 (20.12 J/g_{SCM}), with $D_{90\%}$ less than or equal to 11 µm, reached reactivity similar to that of calcined clays with up to 17% kaolinite (<28 J/g_{SCM}) and sugarcane bagasse ash (~23 J/g_{SCM}) reported in previous investigations (Avet *et al.*, 2016; Basto; Lima; Melo Neto, 2023).

WCT showed intermediate reactivity, being compatible with sample WCB10.3. This indicates that, although the materials are manufactured at different temperatures, the R^3 test suggests that the pozzolanic reactivity among these samples is similar. However, it should be considered that the R^3 test carried out for 24 hours at 40 °C has limitations for low-grade clays since the pozzolanic reaction in these systems occurs gradually, requiring an extension of the test time to ideally seven days (ASTM, 2020; Avet *et al.*, 2016).

The quantification of the bound water content (Figure 4) of the mixtures for the R^3 test showed a similar trend to the total heat released during 24 hours of reaction (Figure 3). This correlation occurs because the quantified bound water is attributed to the dehydration of phases such as C-S-H, ettringite, C-A-S-H, and carboaluminates, which are formed by exothermic reactions between SCM and $Ca(OH)_2$, resulting in the typical heat flow curve obtained by isothermal calorimetry for cement pastes (Costa *et al.*, 2021; Ramanathan; Tuen; Suraneni, 2022). Investigations conducted on a range of SCMs (fly ashes, slags, calcined clays, natural pozzolans, and silica fume) identified an excellent correlation (0.9) between cumulative heat (7 days) and the quantification of bound water, indicating that both techniques can be used interchangeably for predicting the pozzolanic reactivity of alternative raw materials (Zuluaga *et al.*, 2022).

Pozzolanic reactivity in composite cements

Figure 5 presents the compressive strength of composite cement pastes at 28 days of curing. The samples show a similar trend to that observed in the R³ test, with MK and clay, respectively, associated with the upper and lower limits of pozzolanic reactivity. The compressive strength of composite cements containing WCB increased as the $D_{90\%}$ diameter decreased. However, the values tended to stabilize when $D_{90\%}$ was less than 11 µm (WCB10.2 and WCB10.3). Although the results of the rapid and conventional methods show similar trends, the proportionality between the samples differs for each technique. WCB11.4 showed 22% of the total heat of the MK system. Although, this ratio was 86% (CPII-Z) and 85% (CPIV) for compressive strength. However, the similar behavior between CPII-Z and CPIV indicates that other factors may influence the reactivity of SCM depending on the applied technique. Previous investigations evaluated the influence of ceramic brick powder on composite cement pastes. The authors demonstrated that systems with smaller particle sizes consume more calcium hydroxide to react, which could make the microstructure denser and achieve more significant improvement in compressive strength at later ages (Zhao *et al.*, 2020).

Figure 3 – **Cumulative heat over the 24 hours of reaction of SCM pastes (40 °C)**

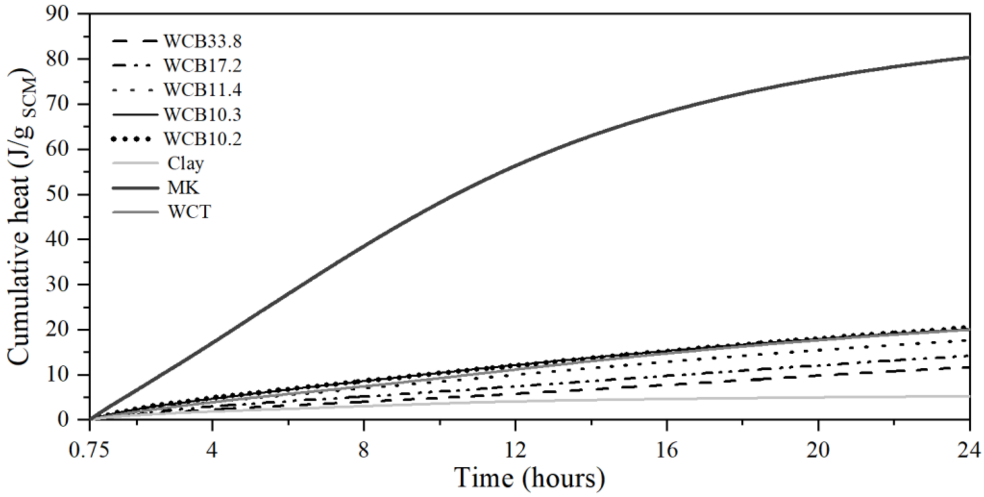
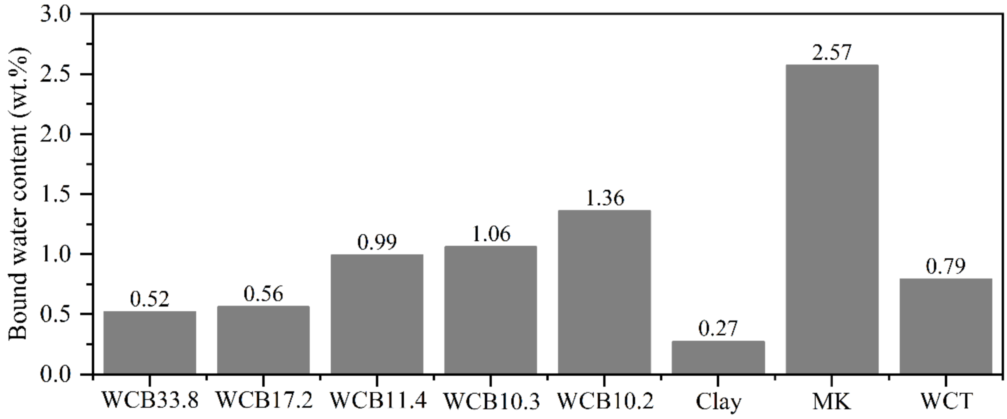


Figure 4 – **Mass loss associated with bound water of the mixtures for the R³ test after 24 hours, between 40 and 200 °C**



The CPII-Z type composite cements showed higher compressive strength values compared to CPIV, as the SCM content in these systems (14%) is lower than in CPIV (50%). This difference in replacement degree resulted in a decrease of about 20% in compressive strength, except in the cement containing natural clay. In this paste, the variation reached 47% and may be related to the deficiency in optimizing the proportion of non-crystalline Al_2O_3 and $CaCO_3$, responsible for the formation of carboaluminates and improvement of compressive strength (Zunino; Scrivener, 2021). The results for the CPIV pastes were satisfactory. In these systems, the high content of quartz from SCM may have favored nucleation to optimize hydration reactions by acting on grain packing (Liu *et al.*, 2023). The difference in compressive strength performance of composite cements may also be associated with variations in reaction and formation of phases from raw materials and the precipitation of hydrates (Zhang; Yang; Wang, 2023).

The mineralogical composition of composite cement pastes containing WCB11.4 is detailed in Figure 6 and Table 2. These samples show remaining anhydrous phases and elements from the raw materials used. The results corroborate the previous steps, indicating the presence of C-S-H and the formation of hemicarboaluminate as hydration products. C-S-H does not have a fixed composition and can vary between highly crystalline minerals such as tobermorite ($Ca_5(Si_6O_{18}H_2)\cdot 8H_2O$) and jennite ($Ca_9(Si_6O_{18}H_2)(OH)_8\cdot 6H_2O$), as well as intermediate minerals classified as C-S-H gel (Hewlett; Liska, 2017). C-S-H gel is the main form observed in cement pastes. It exhibits low crystallinity, which is not identifiable by XRD analysis but quantified by complementary techniques integrated with the overall amorphous content of the sample. The contents identified in this study were 69.95% for CPII-Z and 76.47% for CPIV (Table 2). Carboaluminates are phases resulting from the interaction between calcium hydroxide, carbonates, and phases containing alumina (non-crystalline clay minerals and tricalcium aluminate from clinker) (Krishnan *et al.*, 2018; Scrivener *et al.*, 2018). These compounds are often reported as components of ternary cement pastes and are responsible for refining the pore structure of the cement matrix, enhancing compressive strength beyond seven days (Dhandapani; Santhanam, 2017; Krishnan; Arun; Bishnoi, 2019). Carboaluminates indicate the pozzolanic reactivity of SCM containing clay minerals and are reported as poorly crystalline phases, with only the highly crystalline fraction identifiable by XRD. Thus, the complementary fraction is included in the total amorphous content (ACn) of the sample. Monocarboaluminate is a product formed from the progression of hemicarboaluminate hydration, with its precipitation favored in systems with higher reactivity (Ipavec *et al.*, 2010).

The predominance of quartz in CPIV cement (12.08%), compared to CPII-Z (3.47%) (Table 2), is associated with the higher level of SCM content. However, the substantial presence of $Ca(OH)_2$ indicates a more pronounced pozzolanic reactivity in the CPII-Z system. It is noteworthy that, despite the differences in clinker contents, both cements show a similar formation of Hc. This behavior can be attributed to the balance of precursor phases in the system. While CPIV has a higher alumina content derived from WCB, CPII-Z has a higher clinker content, increasing the availability of C_3A to compensate for the formation of carboaluminates.

Figure 5 – **Compressive strength of composite cement pastes at 28 days**

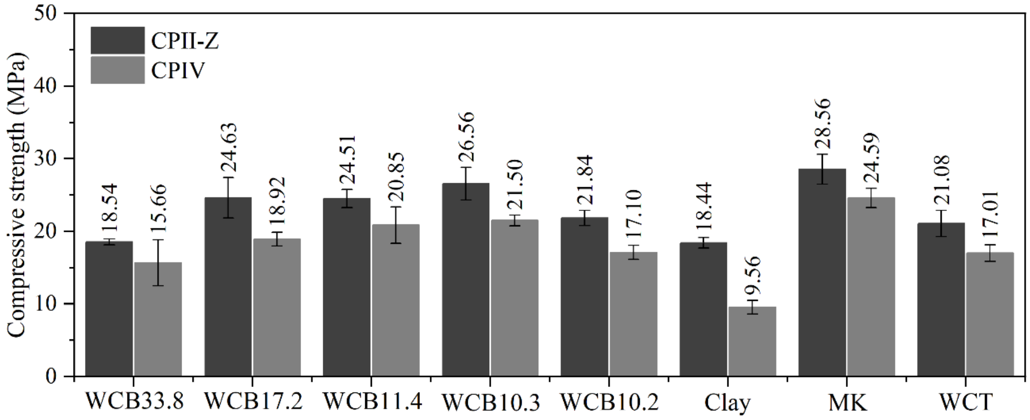


Figure 6 – **X-ray diffractometry (XRD) of composite cement pastes containing WCB11.4 at 28 days**

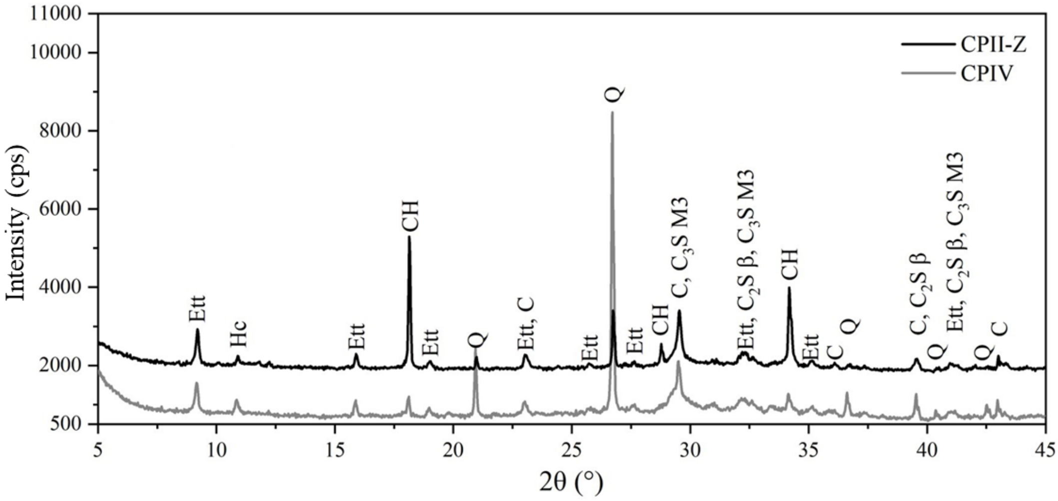


Table 2 – **Composition of composite cement pastes containing WCB11.4 at age 28 days**

Phase (wt%)	Composition	ICSD code	CPII-Z	CPIV
Quartz (Q)	SiO_2	62404	3.47	12.08
Calcium hydroxide (CH)	$Ca(OH)_2$	15471	7.83	1.11
Calcite (C)	$CaCO_3$	28827	6.69	3.24
Dicalcium silicate (C_2S β)	$2CaO.SiO_2$	81096	2.55	1.89
Tricalcium silicate (C_3S M3)	$3CaO.SiO_2$	94742	2.53	1.28
Ettringite (Ett)	$Ca_6Al_2(OH)_{12}(SO_4)_3·26H_2O$	155395	6.40	3.42
Hemicarboaluminate (Hc)	$Ca_4Al_2(OH)_{12}.OH.0.5CO_3.4H_2O$	263124	0.56	0.50
Amorphous (ACn)	-	-	69.95	76.47
Rwp (%)	-	-	8.01	7.06

Conclusions

This study evaluated the influence of particle size on the pozzolanic reactivity of ceramic brick waste with different granulometries. The WCB pozzolanicity was evaluated using the rapid test (R^3 test), correlating it with the compressive strength (at 28 days) of composite cement pastes. Based on the results, the main conclusions obtained were:

(a) the reactivity of WCB was enhanced with the reduction of particle diameter up to a specific limit, stabilizing when $D_{90\%}$ was greater than or equal to 11 µm;

(b) regardless of the granulometric distribution of WCB, MK was the most reactive, and natural clay was the least pozzolanic reactive among all investigated SCM;

(c) beneficiation of by-products, guided by reactivity, contributes to reducing energy consumption, as it allows identifying the time limit at which the prolongation of grinding minimally interferes with the increase in pozzolanic reactivity of SCM, and the associated costs may not be worthwhile;

(d) regarding compressive strength, the CPII-Z cement showed the best performance, justified by the higher amount of clinker in the system. A reduction of approximately 20% in compressive strength was observed in CPIV pastes compared to CPII-Z, except for the cement containing natural clay (47%). Despite this reduction, the results were consistent since this system was composed of 50% SCM;

(e) the reactivity of materials by the R^3 test corroborated the results identified by the conventional method (compressive strength and XRD/Rietveld). However, further investigations are pertinent since there is a difference in the scale of reactivity degree identified by each technique; and

(f) the use of the R^3 test to guide the pozzolanic reactivity of innovative alternative materials proves to be a viable measure for optimizing waste recycling in the production of composite cements.

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