Journal of Building Pathology and Rehabilitation (2023) 8:63
https://doi.org/10.1007/s41024-023-00308-z

RESEARCH ARTICLE

Physical and mechanical characterization of calcined clays for use as supplementary cementitious material

Gustavo Gutierrez de Oliveira Rodrigues[1] · Natália Salamoni[1] · Abrahão Bernardo Rohden[1] · Guilherme Carrara[1]

Received: 12 April 2023 / Revised: 16 May 2023 / Accepted: 17 May 2023 / Published online: 3 June 2023

Abstract
Cement is the second most used material in the world. However, its production causes serious environmental impacts. The carbon dioxide emission of this type of industry has a significant contribution to global warming. The sustainability that civil construction promotes must not only be analysed after the construction of buildings, but in the entire construction stage, from the material to be used to the losses in the process. In this context, it is important to note that several alternatives have already been studied, such as the use of supplementary cementitious materials in concrete, such as rice husk ash, sugarcane bagasse and calcined clays. Although calcined clays are already used as a partial replacement for clinker in composite cements, several authors suggest that the material is capable of being used as a partial replacement for Portland cement. Thus, the objective of this work was to evaluate the potential application of calcined clays submitted to calcination thermal treatments for use as supplementary cementitious material. To achieve this objective, the clays were characterized and subsequently subjected to calcination in a muffle at temperatures equal to 500, 600, 700 and 800 °C, carrying out tests regarding the pozzolanic activity potential, performance as metakaolin and electrical conductivity. It was observed that for the proposed temperatures, the performance indices with Portland cement and use as metakaolin were met, demonstrating a high potential for use. Upon verifying the data found, the sample calcined at 800 °C showed the best results, both in mechanical performance (performance index with Portland cement and performance as metakaolin) and in the electrical conductivity test. In this sense, it appears that the use of calcined clays has the possibility of application as a material to minimize the adverse effects caused by the manufacture of Portland cement.

Keywords Pozzolan · Portland cement · Metakaolin · Electric conductivity

1 Introduction

The construction industry strongly contributes to economic growth in many countries. Among the main reasons for this activity to be considered important, the virtues start from the generation of jobs in the most diverse interconnected sectors, linked to the high demand for construction materials, in addition to the various projects built to provide the solution of infrastructure problems. In this sense, the activities carried out require the use of various materials that are produced and used on a large scale, such as Portland cement, which is the second most used material in the world, with manufacturing data exceeding 4.1 billion tons. of cement [1].

Together with these data, it was identified that this activity has negative effects on the environment. The cement industry is responsible for about 8% of all carbon dioxide (CO_2) emitted from human activities [2] and this figure could reach 25% by 2050 [3], if interventions are not carried out, both in terms of use of raw materials, as well as technological innovations for calcining materials. Linked to these factors, cement production demands high energy consumption, which exceeds 7% of all energy used in industrial sectors [4].

Due to the need for innovations, actions were listed to reduce these adverse effects. Among the proposed activities, the replacement of clinker by supplementary cementitious materials has gained prominence in recent years. [5]. This

✉ Gustavo Gutierrez de Oliveira Rodrigues
gustavogutierrez01@hotmail.com

Natália Salamoni
nasalamoni@gmail.com

Abrahão Bernardo Rohden
abrcivil@gmail.com

[1] Blumenau Regional University Foundation, PPGEA, Blumenau, Brazil

reason is mainly due to reducing the proportion of the main compound that emits CO_2 in the manufacturing process [6] and replacing it with materials from industrial by-products or those that require a lower percentage of energy for thermal activation [7].

Currently, several materials are used that partially replace cement, especially blast furnace slag and fly ash [8]. These materials come from industry by-products, which would be destined for landfills [9]. It should be noted that the application of these materials is seen as a great alternative in the quest to promote reductions in the negative environmental impacts of cement production. However, a fact to be considered is related to the availability of these inputs against the demand for cement. In 2019, cement manufacturing, in national terms, was approximately 54.3 million tons [1].

On the other hand, the availability of the main materials used in the manufacture of cement, which are blast furnace slag, around 7.38 million per year, in addition to fly ash, disposed mainly in Santa Catarina and Rio Grande do Sul, with close data to 2.2 million tons/year, do not show increasing rates when compared to cement production [10]. By correlating the use limits provided for by NBR 16697 [11] which allows the replacement of clinker in up to 75% of mineral additions, it is noticed that the available volume of materials with application potential is around 17% [10]. As these are materials that in production scales grow in quantities smaller than the cement production, there is a need to identify and apply new materials capable of keeping up with the growing data of cement.

Therefore, among the materials identified as suitable for use, calcined clays are identified as one of the only materials available, in terms of quantities, to accompany the growing number of cement manufacturing [12]. The use of calcined clays has been increasing in recent years. For its application, it is necessary that they undergo calcination thermal treatments, between 500 and 900 °C, so that their structure, previously crystalline, becomes amorphous and presents reactivity when inserted in cementitious matrices [13].

Linked to demanding quantitative energy for submission of calcination, when compared to the manufacture of cement, clays appear as an important alternative for use as a cementitious material, mainly because it does not emit CO_2 in the calcination process. In view of this, this work aimed to evaluate the potential application of calcined clays subjected to thermal calcination treatments for use as a supplementary cementitious material.

2 Supplementary cementitious materials

Supplementary cementitious materials (SCM) are soluble siliceous, aluminosicious or calcium aluminosilicate powders that can be used as partial replacements for Portland cement in concrete mixes and for clinker in cements. Its use can improve properties in the fresh and hardened states of concrete, durability, in addition to reducing costs and environmental impact [14, 15].

The SCM have favourable characteristics according to their composition and origin, and may be a filler effect, contributing to the reduction of pores, increase of mechanical resistance and, also, activation of pozzolanic reactions in alkaline aqueous conditions or in contact with the calcium hydroxide (CH) forming hydration products such as hydrated calcium silicate (C–S–H) [16, 17].

Traditionally, blast furnace slag, fly ash, silica fume and metakaolin have been used as SCM. Despite the great knowledge already achieved about the impacts on cement hydration and concrete properties, it is important to analyse and research new SCM sources that aim to meet the increased demand for cementitious materials and the varied availability of raw materials for each region, cooperating for the sustainable development of the sector [14, 15, 17].

With the discontinuity of coal plants in the world, for environmental reasons, for example, the amount of fly ash currently available will tend to reduce [17]. On the other hand, metakaolin, despite its good performance in concrete, has limited use due to its price being generally higher than that of Portland cement [18].

Other possible sources of SCM that contain calcareous, siliceous, and aluminous materials are sugarcane bagasse ash, rice husk ash [15], calcined clays [18, 19] and red ceramic waste [20].

Due to the use of alternative raw materials to replace clinker, the Brazilian cement industry is responsible for emitting 564 kg of CO_2 per ton of cement, around 11% less than the world average. The use of materials and by-products from other production processes also contributes to the preservation of non-renewable natural resources [21].

The use of calcined clay as a SCM has gained prominence. Allied to its abundance and low production cost [17], the mechanical and durability properties of its use in concrete highlight the potential use of the material as a SCM [18, 19]. Clays contain clay minerals that are thermally activated, such as kaolinite, illite, montmorillonite and mica. Thermal activation usually occurs through calcination, making it reactive with Portland cement in its hydration phase. Calcined clays with up to 25% (by weight) of kaolinite offer greater potential for application as SCM [18, 19].

In addition to the origin and chemical composition of the clay, the amount and type of impurities also interfere with its use as an SCM. According to these factors, each clay will have an ideal calcination temperature, which will be directly related to its reactivity [19]. Depending on its composition and impurities, the activation of calcined clays occurs in the temperature range of 550 to 950 °C, below the sintering temperature, where vitreous structures would form. Because of this, calcined clays have physicochemical and morphological properties different from conventional SCM [18].

As SCM can be applied both in the partial replacement of clinker in cement production and in the partial replacement of cement in concrete, its use has become an important tool for reducing CO_2 emissions in the sector [14].

In addition to reducing the clinker factor and CO_2 emissions in the cement industry, the use of calcined clays can match and even surpass the binding properties of concrete [18].

Based on the growing use of clays such as SCM, Bediako et al. [19] calcined clays from the Ghana region, at temperatures of 600, 700, 800, 900 and 1000 °C, for 3 h in an electric muffle. Material characterizations were carried out with thermogravimetric tests (TGA) and Fourier Transformed Infrared Spectroscopic techniques (FTIR), and compressive strength tests to determine the pozzolanic activity with 20% replacement of Portland cement by calcined materials. With the results obtained, it was verified that the clay used had a predominance of kaolinite, with an ideal calcination temperature that produced more reactive pozzolanic phases and greater mechanical resistance was 800 °C. Furthermore, the 20% replacement indicated that the clays behaved both as a filling material and as a pozzolanic material, helping in the initial and final resistance of the cementitious matrix.

Arslan et al. [22] evaluated mortars with substitutions of 0, 5, 10, 15 and 20% of Portland cement by calcined kaolin and metakaolin exposed to temperatures of 300, 600 and 900 °C, for two hours. 15% replacement results in improved mechanical performance and durability.

Similarly, Rakhimov et al. [23] replaced Portland cement with 5, 10, 15 and 20% of thermally activated clays (400, 600 and 800 °C) in concrete, demonstrating superior results in terms of mechanical strength and density in concretes with the addition of 5 to 15% of clays calcined at temperatures of 400 and 600 °C and milled to a specific surface area of 250 to 500 m^2/kg.

3 Materials and methods

3.1 Materials

The clay used as raw material in this study was collected in a ceramic industry in the interior of the State of Santa Catarina. Its collection took place in the form of extruded blocks (Fig. 1), which had moisture content equal to 1%, and they had not been submitted to any thermal calcination treatment.

Fig. 1 Clay in natura

As for the use of fine aggregate, the ranges determined by NBR 7214 [24] were used, which corresponded to 0.15, 0.30, 0.60 and 1.20 mm. The initial procedures for preparing the aggregates consisted of washing to remove any organic matter contained, in addition to subsequent drying and granulometric separation for use.

As indicated by NBR 5752 [25] and 15894-2 [26], the cement used is of the CP II-F 32 class. This choice is because this cement does not have pozzolanic addition, which could influence the quality of the studied product.

3.2 Methods

After collection, the clay samples were dried, ground and calcined in an electric muffle at temperatures corresponding to 500, 600, 700 and 800 °C for 2 h [27, 28]. Then, the clays went through the milling process again.

To evaluate the performance of these alternative materials with Portland cement, tests were carried out to assess fineness (#0.045 mm), specific mass, humidity content, loss on fire, performance index with Portland cement, performance with metakaolin and electrical conductivity. Figure 2 presents the flowchart of the experimental procedure.

3.2.1 Milling

The executive milling stage was carried out using a Los Angeles Abrasion type ball mill with steel balls with a diameter of 17 mm. This activity took place by drying the clay for about 24 h in an oven at a temperature of 110 °C. Linked to these factors, a sphere mass ratio was defined in relation to the mass of the sample to be ground. Therefore, based on studies by Pouey [29] and Cordeiro [30], a ratio of 1:5 was defined.

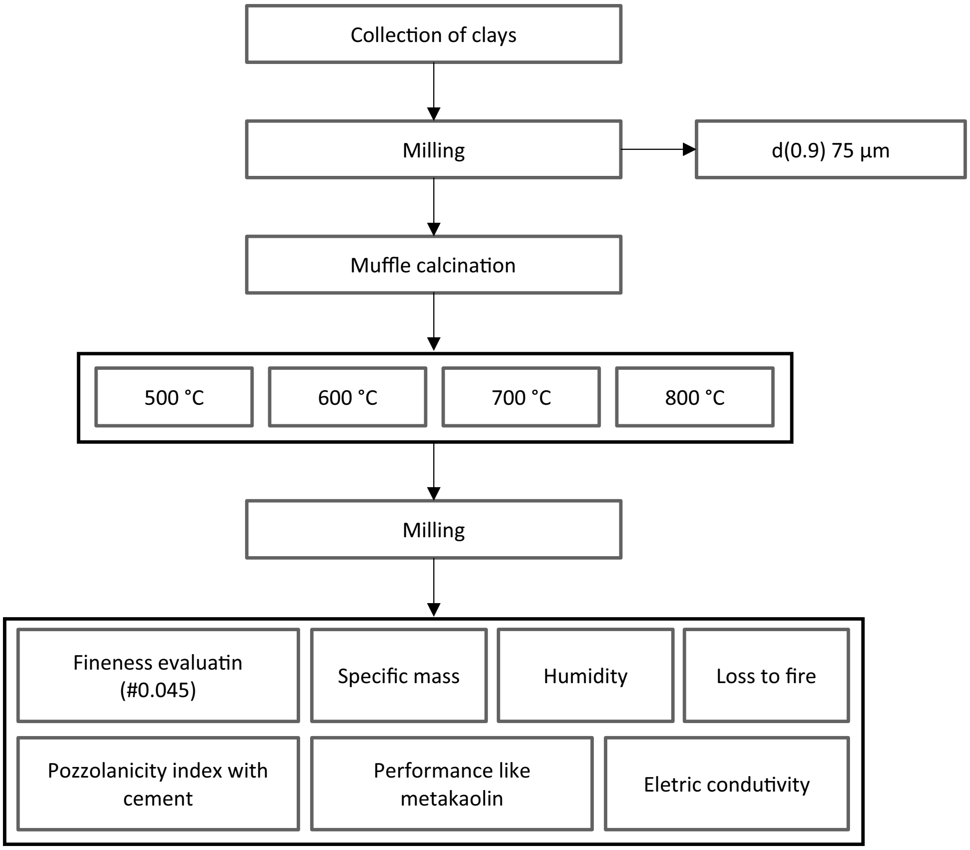

Fig. 2 Methodological procedures

Table 1 Mix proportions of mortars

Material	Reference	Cement + SCM Pozzolan	Cement + SCM High reactivity pozzolan
Cement CPII-F-32 (g)	624	468	530
SCM (g)	–	156	94
Sand (g)	1872	1872	1872
Water (g)	300	300	300
Superplasticizer (g)	–	Adjustable	Adjustable

3.2.2 Characterization

The granulometric composition and evaluation of the fineness (#0.045 mm) of the clays was determined using the Mastersizer 2000 equipment. Afterwards, the clays were further characterized through specific mass tests—DNER-ME 093 [31], humidity content—NBR NM 24 [32] and fire loss—NBR NM 18 [33].

For the evaluation of the performance index of calcined clays with Portland cement and the use as a high reactivity pozzolana, they were evaluated according to the prescriptions determined in NBR 5752 [25] and NBR 15894-2 [26].

After mixing (Table 1), which was carried out in a mechanical mortar mixer, six cylindrical specimens of (50 × 100) mm were moulded, in triplicate, which were submerged in a tank of water saturated with lime for curing until the test of resistance to axial compression at 7 (Cement + SCM High reactivity pozzolan) and 28 days (Cement + SCM Pozzolan). The bases of the samples were ground for the mechanical resistance test. The performance index was then calculated by the ratio of the average resistance of the mortars with the alternative materials by the average resistance of the reference mortar.

Finally, the electrical conductivity test was performed according to the method proposed by Luxán et al. [34]. This procedure was carried out with the use of 200 ml of a supersaturated solution of calcium hydroxide, submitted to a temperature of 40 ± 1 °C under agitation. After 60 s, the first reading was performed, which obtained the conductivity of the calcium hydroxide solution. Subsequently, 5.0 g of calcined material were added and after 120 s, the sample was subjected to a new reading to obtain the conductivity result of the solution with the calcined clay.

It should be noted that the conductivity meter used reads in milliSiemens per centimeter (mS/cm) at 25 °C. As the sample was at 40 °C, the conversion to the actual conductivity result was performed using Eq. (1)

Table 2 Evaluation of pozzolanic activity by measuring conductivity

Material classification	Conductivity variation (mS/cm)
Non pozzolanic	Less than 0.4
Medium pozzolanic	Between 0.4 e 1.2
Good pozzolanicity	Bigger then 1.2

$$\text{Electrical conductivity at } 40^\circ\text{C} = \text{Elec. conductivity at } 25^\circ\text{C} \times [1 + 0.019 \times (40 - 25)] \tag{1}$$

After the necessary conversions, the calcined clays were classified according to the data proposed by Luxán et al. [34], with the conductivity variation shown in the test, as shown in Table 2.

With the results in hand, the samples were classified according to their degree of pozzolanicity using the electrical conductivity technique. The realization of this essay consisted in the search for informative curtailment regarding the pozzolana developed, in addition to mechanical application with the direct replacement of cement, linked to the responses obtained with the use of electrical conductivity, which is the direct contact of the sample with the hydroxide of calcium.

4 Results and discussion

4.1 Characterization of clays

After the calcination procedures, substantial changes were observed in several aspects, one of the most evident of which was the colour (Fig. 3). The in natura sample had a milder colour, with little colour intensity, however, after the thermal calcination treatments, they showed reddish tones, which corroborate the activation of iron oxide after calcination [35].

Linked to the colour change, the calcined samples tend to present variations in diameters, mainly regarding the increase, due to the chemical reactions that took place. This way, laser granulometry analysis was carried out to identify possible variations in clay diameters. Figure 4 presents the granulometric profiles of the clays before and after the calcination procedures.

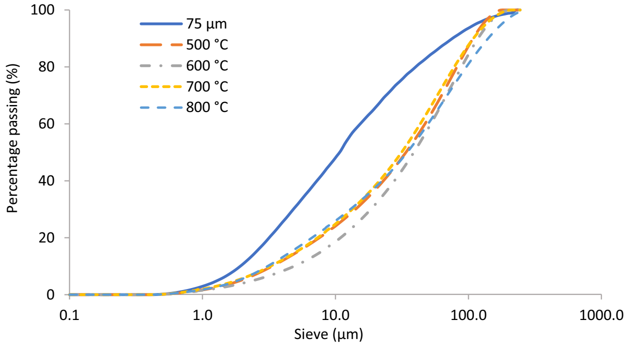

Fig. 4 Granulometric composition of clays

Table 3 $D_{(90)}$ of the particles after thermal calcination treatments

Diameter	Sample			
	500 °C	600 °C	700 °C	800 °C
$d_{(90)}$ (µm)	102	110	105	116

By observing the development of the granulometric curves, due to the thermal treatment, the samples presented a progressive increase in the profiles. Similar facts are portrayed in literature [5, 36, 37]. One of the factors that must be observed in relation to the granulometric profiles is the ratio of the percentage corresponding to 90% of the particles (d90) (Table 3). This data is of great importance, especially for SCM there are specific criteria corresponding to d90, to verify the use as pozzolana and metakaolin.

The values expressed in Table 3 verify the observed increases in the granulometric profiles and corroborate with the thesis of the change in diameters when the samples are submitted to thermal treatments. Linked to these factors, the aspects demonstrated by NBR 5752 [25] and NBR 15894-2 [26] stipulate maximum values that must be respected for use as pozzolan and metakaolin, for the latter corresponding to a maximum of 10% of material retained on the 45 µm sieve.

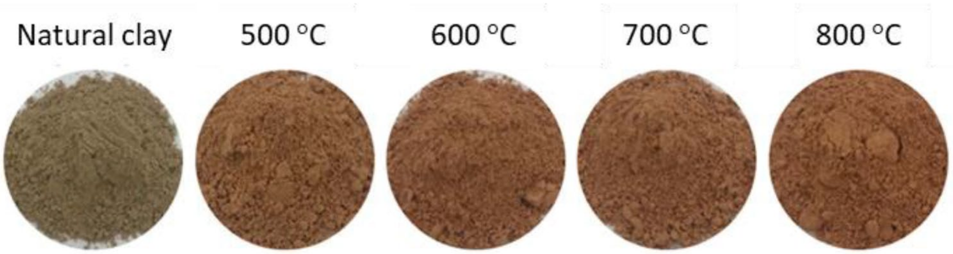

Fig. 3 Samples before and after calcination heat treatments

Based on the limits established by regulatory standards, the samples required milling for use as SCM. Grinding techniques followed the same operational procedures as the initial milling and the milling stop criterion was when the samples met the requirement of 10% of material retained on the 45 μm sieve. The milling time of the samples was around 4 h to meet the necessary granulometries.

Regarding the physical and chemical characteristics, as well as the necessary requirements for classification as pozzolans, they were followed according to the aspects provided for in current regulations, summarized in Table 4. Regarding the specific mass, the in natura sample presented a result equal to 2.51 g/cm^3. When subjected to calcination heat treatments, the samples demonstrated superior results compared to the input. This increase factor may be linked to the change in the diameter of the particles because of the chemical reaction that took place [22, 38].

Another aspect observed in the characterization of the samples in physical terms is with respect to the humidity content. Brazilian regulations provide for a maximum value of 2% for highly reactive pozzolans (metakaolin), a value that did not come close to any of the analysed samples.

For the results obtained in the fire loss test, the national regulations determine the data of 3% as the maximum observed limit for classifying the material as metakaolin. The exposed results prove that all the samples submitted to calcination are classified as pozzolana and metakaolin. When relating the reduction in the fire loss data, it is observed that the samples submitted to higher temperatures had a lower fire loss value, when compared to the lower temperatures, an effect already observed in calcination of clays for use as SCM [39, 40].

4.2 SCM performance

Data regarding performance with cement at 28 days were obtained by virtue of the treated combinations, correlating both the granulometry (75 μm) and the temperatures used (500, 600, 700 and 800 °C). In this phase, the main search consisted of punctual observation in which it was considered whether the variation in temperature exerted impacts on the performance of the samples. In view of these aspects, Fig. 5 depicts the results obtained by breaking the moulded specimens. Together, the data related to compressive strength and the pozzolanic activity index are available.

NBR 12653 [41] and NBR 16697 [11] determine the minimum values that materials must meet to be classified as pozzolan, two percentages of reactivity are observed, which differ only in class in application. When observing the results found, the samples calcined at 500 and 700 °C showed the lowest activity indices when compared to the other data. However, despite the portrayed difference, all samples reached values above the minimum required for classification and use as pozzolans.

The heat-treated sample at 800 °C showed the best result compared to the other results. In this circumstance, the clay may have shown a higher degree of reactivity, due to having been subjected to a favourable temperature together with the time that provided a greater degree of reactivity, reducing its crystalline content [42].

In a performance analysis, the samples were subjected to analysis regarding use as metakaolin, which is analysed for its performance after seven days. If it is a shorter time when compared to use as pozzolan, the results must present at least 105% of the reference resistance for classification as possible use as metakaolin.

Table 4 Characterization and requirements of calcined samples

Test	Sample (°C)	Results	Pozzolan (%)	Metakaolin (%)
Fineness (#0.045 mm)	500	9.9	≤20	≤10
	600	8.9		
	700	9.2		
	800	8.8		
Specific mass (g/cm^3)	500	2.52	–	–
	600	2.52		
	700	2.54		
	800	2.56		
Humidity (%)	500	0.63	≤3.0	≤2.0
	600	0.61		
	700	0.52		
	800	0.46		
Loss to fire (%)	500	3.70	≤10	≤4.0
	600	3.60		
	700	3.15		
	800	2.90		

Fig. 5 Pozzolanicity index with Portland cement

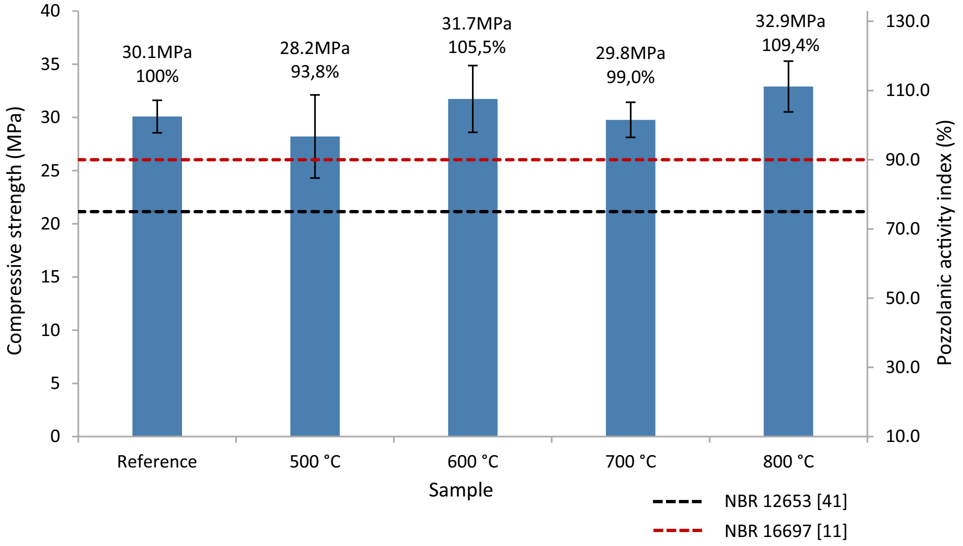

Fig. 6 Performance like metakaolin

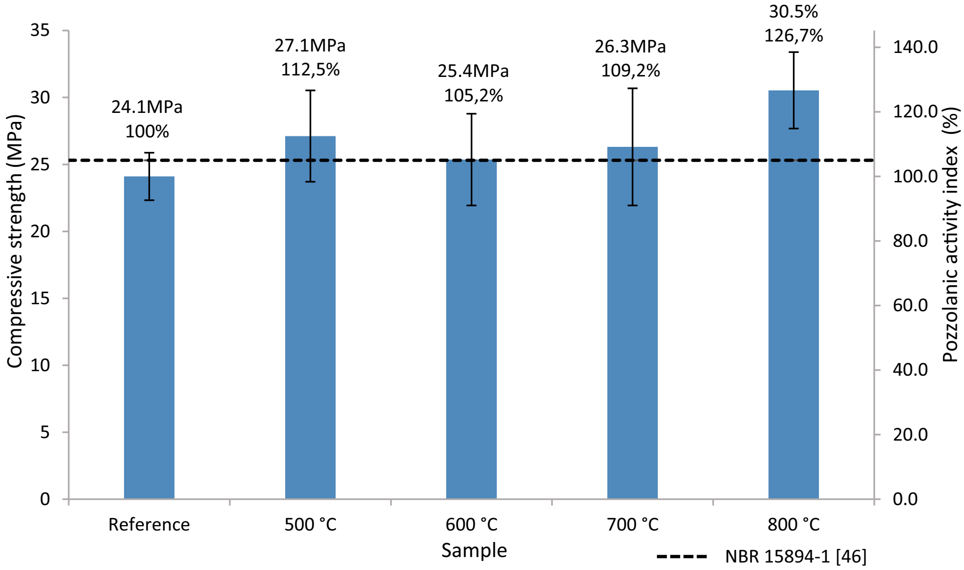

Figure 6 presents the results consistent with the breakage of the specimens for analysis regarding performance as metakaolin. Upon verifying the data obtained, it is noted that all samples met the minimum required by the standard for classification as metakaolin. One should consider using calcined clays that do not always present favourable results for use as metakaolin [43].

In a similar way to what was observed in the analysis as pozzolan, a sample calcined at 800 °C showed the highest reactive percentage for use as metakaolin. In this sense, it was tested that this temperature data corresponded to the best result among those analysed. This respiratory factor, along with the other results, demonstrated that the temperature of 800 °C demonstrates that it is the optimal calcination temperature.

Together with the compressive strength tests to evaluate performance with pozzolan and metakaolin, the electrical conductivity test was carried out. This test directs the calcined sample into direct contact with calcium hydroxide and thus, the change in conductivity exerted by the material can be observed.

Table 5 depicts the results regarding reactivity using the electrical conductivity technique. The results showed that only the sample subjected to heat treatment corresponding to 500 °C did not meet the minimum required for classification as pozzolanic.

For the other results found, the data varied between 0.53 and 0.67 mS/cm, which fit into a classification of average degree of reactivity for pozzolans. The application of this technique has stood out in the medium and recent range in research with the use of clayey materials for use

Table 5 Pozzolanicity by electrical conductivity

Calcined clay (°C)	ΔmS/cm	Classification according to pozzolanicity
500	0.30	No pozzolanicity
600	0.53	Medium pozzolanicity
700	0.64	Medium pozzolanicity
800	0.67	Medium pozzolanicity

as pozzolans [35, 44, 45] which obtained results close to those found in this work.

5 Conclusions

As a result of pressures to reduce the adverse environmental impacts of Portland cement manufacturing, several materials began to be studied and applied in the industry with the aim of providing for their replacement. Among these, calcined clays are identified as one of the only materials with technical and volume capabilities to supply the demand for materials that can replace cement and/or clinker in the manufacturing process, without missing important product characteristics.

In this sense, the results showed that for the time and temperatures adopted, the samples met the minimum requirements for use both as pozzolana and metakaolin. Among the analysed samples, clay calcined at 800 °C showed the best results in all treated aspects.

The mechanical performances were very promising when correlated with the corresponding replacement percentages of each phase. When mechanical results were correlated with electrical conductivity, only the sample subjected to 500 °C did not reach the minimum data for classification as pozzolanic medium.

Based on these factors, it is concluded that the temperatures and calcination time were sufficient to provide calcined material with the necessary properties for use as SCM. Therefore, the continuation of studies with higher temperatures and variable residence times to reduce the energy demand, together with evaluations of other types of clay to enable the characterization of new sources of SCM, becomes propitious.

Author contributions All authors contributed to the conception and design of the study. Material preparation, data collection and analysis were carried out by GC. The main text of the manuscript was written by GGdOR and NS. ABR revised the manuscript. All authors read and approved the final manuscript.

Funding The authors have not disclosed any funding.

Data availability The data is arranged in the paper.

Declarations

Competing interests The authors declare no competing interests.

References

1. CEMBUREAU (2020) The European Cement Association. Activity report 2019
2. Akhlaghi O, Aytas T, Tatli B, Sezer D, Hodaei A, Favier A, Scrivener K, Menceloglu YZ, Akbulut O (2017) Modified poly(carboxylate ether)-based superplasticizer for enhanced flowability of calcined clay–limestone–gypsum blended Portland cement. Cement Concr Res 101:114–122. https://doi.org/10.1016/j.cemconres.2017.08.028
3. Provis JL (2014) Geopolymers and other alkali activated materials: why, how, and what? Mater Struct 47:11–25. https://doi.org/10.1617/s11527-013-0211-5
4. Diaz-Loya I, Juenger M, Seraj S, Minkara R (2019) Extending supplementary cementitious material resources: reclaimed and remediated fly ash and natural pozzolans. Cement Concr Compos 101:44–51. https://doi.org/10.1016/j.cemconcomp.2017.06.011
5. Shah V, Bishnoi S (2018) Carbonation resistance of cements containing supplementary cementitious materials and its relation to various parameters of concrete. Constr Build Mater 178:219–232. https://doi.org/10.1016/j.conbuildmat.2018.05.162
6. Scrivener KL (2004) Options for the future of cement. Indian Concr J 88:11–21
7. IEA, WBCSD (2009) Cement technology roadmap 2009, carbon emissions reductions up to 2050
8. World Business Council for Sustainable Development-WBCSD (2016) Cement sustainability initiative, getting the numbers right
9. Battagin AF, da Battagin ILS (2017) Materiais de construção civil e princípios de ciência e engenharia dos materiais, 3rd edn. IBRACON, São Paulo
10. Da Silva MG, Battagin A, Gomes V (2017) Materiais de construção civil e princípios de ciência e engenharia dos materiais, 3rd edn. IBRACON, São Paulo
11. Associação Brasileira de Norma Técnicas (2018) NBR 16697: cimento Portland—requisitos
12. Scrivener KL, John VM, Gartner E (2018) Eco-efficient cements: Potencial, economically viable solutions for a low-CO2, cement-based materials industry. Cement Concr Res 114:2–26. https://doi.org/10.1016/j.cemconres.2018.03.015
13. de Kazmierczak CS (2017) Materiais de construção civil e princípios de ciência e engenharia dos materiais, 3rd edn. IBRACON, São Paulo
14. Juenger M, Snellings R, Bernal S (2019) Supplementary cementitious materials: new sources, characterization, and performance insights. Cement Concr Res 122:257–273. https://doi.org/10.1016/j.cemconres.2019.05.008
15. Nicoara AI, Stoica AE, Vrabec M, Šmuc Rogan N, Sturm S, Ow-Yang C, Gulgun MA, Bundur ZB, Ciuca I, Vasile BS (2020) End-of-life materials used as supplementary cementitious materials in the concrete industry. Materials 13:1954. https://doi.org/10.3390/ma13081954
16. Juenger MCG, Siddique R (2015) Recent advances in understanding the role of supplementary cementitious materials in concrete. Cement Concr Res 78:71–80. https://doi.org/10.1016/j.cemconres.2015.03.018

17. Skibsted J, Snellings R (2019) Reactivity of supplementary cementitious materials (SCMs) in cement blends. Cement Concr Res 124:105799. https://doi.org/10.1016/j.cemconres.2019.105799
18. Beuntner N, Sposito R, Thienel K (2019) Potential of calcined mixed layer clays as pozzolans in concrete. ACI Mater J 116: 19–29. https://doi.org/10.14359/51716677
19. Bediako M, Purohir S, Kevern J (2017) An investigation into Ghanaian calcined clay as a supplementary cementitious material. ACI Mater J 114(6): 889. https://doi.org/10.14359/51700896
20. Hoppe Filho J, Pires CAO, Leite OD, Garcez MR, Medeiros MHF (2021) Red ceramic waste as supplementary cementitious material: Microstructure and mechanical properties. Construct Build Mater 296:123653. https://doi.org/10.1016/j.conbuildmat.2021.123653
21. Sindicato Nacional da Indústria do Cimento-SNIC (2020) Relatório anual 2020, São Paulo
22. Arslan F, Benli A, Karatas M (2020) Effect of high temperature on the performance of self-compacting mortars produced with calcined kaolin and metakaolin. Construct Build Mater. https://doi.org/10.1016/j.conbuildmat.2020.119497
23. Rakhimov RZ, Rakhimova NR, Gayfullin AR, Morozov VP (2018) Effect of the addition of thermally activated heavy loam to portland cement on the properties of cement stone. Inorg Mater Appl Res 9:679–686. https://doi.org/10.1134/S2075113318040330
24. Associação Brasileira de Norma Técnicas (2015) NBR 7214: areia normal para ensaio de cimento—especificação
25. Associação Brasileira de Norma Técnicas (2014) NBR 5752: materiais pozolânicos—determinação do índice de desempenho com cimento Portland aos 28 dias
26. Associação Brasileira de Norma Técnicas (2010) NBR 15894-2: metacaulim para uso com cimento Portland em concreto, argamassa e pasta—Parte 2: Determinação do índice de desempenho com cimento aos sete dias
27. Alujas A, Fernandéz R, Quintana R, Scrivener KL, Martirena F (2015) Pozzolanic reactivity of lowgrade kaolinitic clays: influence of calcination temperature and impact of calcination products on OPC hydration. Appl Clay Sci 108:94–101. https://doi.org/10.1016/j.clay.2015.01.028
28. Danner T, Norden G, Justnes H (2018) Characterisation of calcined raw clays suitable as supplementary cementitious materials. Appl Clay Sci 162:391–402. https://doi.org/10.1016/j.clay.2018.06.030
29. Pouey MTF (2006) Beneficiamento da cinza de casca de arroz residual com vistas à produção de cimento composto e/ou pozolânico. Tese de Doutorado. UFRGS, Porto Alegre
30. Cordeiro LNP (2009) Análise da variação do índice de amorfismo da cinza de casca de arroz sobre a atividade pozolânica. Dissertação de Mestrado. UFRGS, Porto Alegre
31. Ministério dos Transportes (1994) DNER-ME 093: determinação da densidade real
32. Associação Brasileira de Norma Técnicas (2003) NBR NM 24: materiais pozolânicos—determinação do teor de umidade
33. Associação Brasileira de Norma Técnicas (2012) NBR NM 18: cimento Portland—análise química—determinação de perda ao fogo
34. Luxán MP, Madruga F, Saavedra J (1989) Rapid evaluation of pozzolanic activity of natural products by conductivity measurement. Cement Concr Res 19:63–68. https://doi.org/10.1016/0008-8846(89)90066-5
35. Rodrigues GGO (2022) Estudo da calcinação “flash” de argila ilítica para produção de material cimentício suplementar. Dissertação de Mestrado. FURB, Blumenau
36. Tironi A, Trezza MA, Scian AN, Irassar EF (2013) Assessment of pozzolanic activity of different calcined clays. Cement Concr Compos 37:319–327. https://doi.org/10.1016/j.cemconcomp.2013.01.002
37. Tironi A, Trezza MA, Scian AN, Irassar EF (2014) Thermal analysis to assess pozzolanic activity of calcined kaolinitic clays. J Therm Anal Colorim 117(2):547–556. https://doi.org/10.1007/s10973-014-3816-1
38. Chen Y, He S, Zhang Y, Wan Z, Oğuzhan Ç, Schlangen E (2021) 3D printing of calcined clay-limestone-based cementitious materials. Cement Concr Res. https://doi.org/10.1016/j.cemconres.2021.106553
39. Hung W, Kazemi-Kamyab H, Sun W, Scrivener K (2017) Effect of replacement of silica fume with calcined clay on the hydration and microstructural development of eco-UHPFRC. Mater Des 121:36–46. https://doi.org/10.1016/j.matdes.2017.02.052
40. Taylor-Lange SC, Lamon EL, Riding KA, Juengen MCG (2015) Calcined kaolinite–bentonite clay blends as supplementary cementitious materials. Appl Clay Sci 108:84–93. https://doi.org/10.1016/j.clay.2015.01.025
41. Associação Brasileira de Norma Técnicas (2015) NBR 12653: materiais pozolânicos—requisitos
42. Garcia E, Cabral Junior M, Quarcioni VA, Chotoli FF (2016) Avaliação da atividade pozolânica dos resíduos de cerâmica vermelha produzidos nos principais polos ceramistas do Estado de S. Paulo Cerâmica 61:251–258. https://doi.org/10.1590/0366-69132015613581847
43. Pavesi TB (2020) Emprego de resíduos da construção civil de cerâmica vermelha (RCCCV) em substituição parcial do cimento Portland no concreto convencional. Dissertação de Mestrado. FURB, Blumenau
44. Matos Neto JA (2013) Caracterização de estéril argiloso para utilização como pozolana. Dissertaçãode Mestrado, Universidade Federal de Ouro Preto, Ouro Preto
45. Sebastiany LD (2014) Avaliação de métodos de determinação do potencial pozolânico de resíduos da indústria cerâmica vermelha. Dissertação de Mestrado, Universidade do Vale do Rio dos Sinos, São Leopoldo
46. Associação Brasileira de Norma Técnicas (2010) NBR 15894-1: metacaulim para uso com cimento Portland em concreto, argamassa e pasta—Parte 1: requisitos. Rio de Janeiro