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Research Paper

Characterizing and processing a kaolinite-rich water treatment sludge for use as high-reactivity pozzolan in cement manufacturing

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ABSTRACT

This paper evaluates the potential of water treatment sludge ash (WTSA) as high-reactivity pozzolan using strength tests in mortars and techniques to evaluate the calcium hydroxide consumption in pastes with hydrated lime. The sample was dried in an oven and then calcined at 600, 700, and 800 °C in a muffle. A high-reactivity metakaolin sample was used as a pozzolan reference. The properties of the samples assessed were fineness (Blaine), density (Le Chatelier), and chemical and mineralogical composition using X-ray diffraction (XRD), X-ray fluorescence (XRF), and thermogravimetric analysis (TG). The assessment of the pozzolanic properties employed standardized tests such as compression tests with hydrated lime (NBR 5751) and Portland cement (NBR 5752) in mortars. Also, non-standardized techniques, such as XRD, TG, and electrical conductivity, were utilized to evaluate the consumption of calcium hydroxide in pastes and solutions with hydrated lime and pozzolan. The standardized tests showed that mortars with metakaolin (MK) presented higher compressive strength values, while mortars with ash calcined at 600 °C (SA600) obtained the lowest values among WTSA. This behavior occurred because the preparation of mortars was strongly influenced by their consistency, affecting the compressive strength. However, analyzing the consumption of calcium hydroxide in pozzolan-hydrated lime pastes, SA600 presented higher values. Unlike tests on mortars, consistency is not affected in preparing the pastes, allowing a better development of pozzolanic reactions. Based on $Ca(OH)_2$ consumption, it was concluded that the samples of WTSA studied could be used as high-reactivity pozzolans.

1. Introduction

1.1. Water treatment sludge

The use of recycled wastes as supplementary cementitious materials has been increasingly studied worldwide due to the increased awareness of public and private institutions regarding the need to preserve the environment in all aspects. Furthermore, using supplementary cementitious materials from waste is a clever strategy to allocate the waste properly, decrease the clinker content used and, consequently, reduce the release of CO_2 into the atmosphere (Lothenbach et al., 2011; Franco de Carvalho et al., 2019; Juenger et al., 2019; Gupta and Chaudhary, 2020).

Particularly in Brazil, this concern has led state and city governments to demand that local companies sustainably dispose of their waste. As landfill costs have increased in recent years, companies have been looking for ways to value their waste to reuse it in their production cycle or sell it for other purposes.

In this context, sanitation companies stand out as they generate highly toxic and pathogenic waste. The sludge generated by the water and sewage treatment plants must be disposed of following strict and costly federal regulations. Nonetheless, in the metropolitan region of Recife, it has been reported that most of the water treatment sludge generated by the water treatment plants is irregularly disposed of directly in rivers (da Motta Sobrinho et al., 2019). Therefore, companies in this sector have been studying ways to value their generated waste. The sludge has been used by the chemical and agroindustry, but only in the most developed countries (Cusidó and Cremades, 2012). Research shows that the processed water treatment sludge can be used in construction in a variety of ways, whether as a raw material for red ceramics (Cusidó and Cremades, 2012), as a recycled aggregate in concrete and mortar (de Oliveira Andrade et al., 2018), or as supplementary cementitious material to Portland cement (Tantawy, 2015; de Azevedo Basto et al., 2019; de Godoy et al., 2019).

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Toward the water treatment sludge ash (WTSA), it is a consensus that this material can be used as an admixture for Portland cement. However, its chemical composition depends on the local water treatment process and geological aspects. The literature (Huang and Wang, 2013; Pinheiro et al., 2014; Gastaldini et al., 2015; Ahmad et al., 2016; de Oliveira Andrade et al., 2018; de Carvalho Gomes et al., 2019; de Godoy et al., 2019; Santos et al., 2019; Bohórquez González et al., 2020; Liu et al., 2021) exhibits significant deviation in SiO_2, Al_2O_3, and Fe_2O_3 contents. As these oxides are essential for pozzolanic reactions, it is necessary to characterize the chemical properties of WTSA in each situation. However, for better dissolution and occurrence of pozzolanic reactions, these oxides need to be presented in an amorphous form. Thus, a crystallographic study must also be required, using X-ray diffraction. This technique can also be used to measure the consumption of calcium hydroxide due to the pozzolanic reaction. Although there are no standardized mineralogical requirements for evaluating pozzolanic activity, the literature shows that pozzolans must consume a reasonable amount of calcium hydroxide in pastes with hydrated lime (Torres et al., 2020; de Azevedo Basto et al., 2022).

The pozzolanic activity also depends on the fineness of the material. The specific surface influences the early age compressive strength, whereas the glassy phase chemical and mineralogical compositions are more relevant for the performance at later ages (Tironi et al., 2013; Mohammed, 2017). This property can be assessed through sieving, laser granulometry, and indirect determinations by air permeability (Blaine) or gas adsorption (BET). The requirements of the Brazilian standard ABNT NBR 15894-1 (2010) for metakaolin stablishes that the material must present at least 15 $m^2.g^{-1}$ on BET and a maximum of 10% retained material on 45 μm sieve. According to the Brazilian standards for regular pozzolans (ABNT NBR 12653:, 2015), the amount of material retained on 45 μm sieve must not be superior to 20%.

For clayed materials such as WTS, the amorphous phase is derived from the bound water evaporation (dehydroxylation of kaolinite), which corresponds to the destruction of kaolinite's crystallinity resultant from efficient calcination (Mohammed, 2017; de Godoy et al., 2019, 2020). Consequently, thermal treatment of WTS is necessary, obtaining water treatment sludge ashes (WTSA).

The studies show that WTSA has pozzolanic properties. Gastaldini et al. (2015) used WTSA calcined between 400 and 700 °C. The authors found that 700 °C for 1 h was the best calcination condition to obtain the highest compressive strength values with cement. Also, they concluded that replacing 25% of cement with WTSA can increase the compressive strength of concrete (water/binder = 0.35) at 28 days to 29.7% compared to the reference mixture (without WTSA). In another study, de Godoy et al. (2019) varied the calcination temperature at 600, 650, 700, 750, and 800 °C for 1 and 2 h. Also, flash calcination was conducted as a rapid, economical, and feasible alternative compared to traditional calcination. The highest results of pozzolanic activity were found for WTSA calcined at 800 °C for 1 h at 7 days, and at 600 °C for 1 h and 650 °C for 2 h at 28 days. The flash calcined ash presented a pozzolanic activity index with Portland cement of 94% at 7 curing days and met the requirement of the Brazilian standard. Bohórquez González et al. (2020) evaluated the replacement of cement by WTSA calcined at 600 and 800 °C. The authors concluded that the calcination at 600 °C for 3 h in the content replacement of 10% presented the highest compressive strength values. Furthermore, de Carvalho Gomes et al. (2019) concluded that WTS could be useful for civil construction since it is appropriately processed to eliminate organic matter and reduce its porosity.

1.2. Non-standardized tests for assessing pozzolanic activity

The pozzolanic activity corresponds to the decrease of calcium hydroxide (CH) or Portlandite (in cementitious mixtures) content and the consequent increase in the amount of hydrated products (C-S-H, C-A-S-H or C-A-H) (Mohammed, 2017). The pozzolanic reaction is dependent on the maximum amount of CH consumed by the pozzolan, which is related to the mineralogical composition of the material, and the combination rate between pozzolan and CH, because of the pozzolan fineness, water to solid ratio, and curing temperature (Massazza, 2003). Because of this, Tironi et al. (2013) recommend that the pozzolanic reactivity of calcined clays be verified by a combination of two types of tests, allowing a complete evaluation. In one test, the contribution of the pozzolanic material to the improvement of mechanical properties by the densification of microstructure is assessed (e.g., strength activity index test). The other test should precisely quantify the $Ca(OH)_2$ consumption, for example, chemical titration, XRD, and TGA. According to de Carvalho Gomes et al. (2019), based on a review of different types of WTS and WTSA, the calcined sludge can be classified as an artificial pozzolan because it consumes $Ca(OH)_2$ and produces a significant amount of hydrates.

Frías et al. (2014) quantified the fixed CH after 1, 7, 28, and 90 days of reaction of a WTSA-CH system. Although the studied material presented high pozzolanicity at 1-day (fixed lime of 70%), the increase in fixed lime was not significant with age, representing 79.8% at 7 days and 84.1% at 90 days. From the XRD, the main hydration product verified in all ages was strätlingite (C_2ASH_8), followed by tetracalcium aluminate hydrate (C_4AH_{13}), confirmed by the thermodynamic model obtained using the PHREEQC geochemical software program. Liu et al. (2021) used cement pastes with WTSA replacement of 10%, 20%, and 30% to measure by TGA the amount of chemically bound water (H) present in the hydration products and calcium hydroxide per gram of cement at 7, 28, and 90 days. The results showed that the filler effect is predominant at 7 days, evidenced by an increase in the CH content with either replacement level (compared to the reference cement paste) and a significant increase of H content. The pozzolanic activity was more pronounced for the later ages, and the system with 10% WTSA achieved the highest pozzolanic activity. Ruviaro et al. (2021) investigated cementitious matrices containing 15%, 20%, 30%, 40%, and 45% of WTSA. The isothermal calorimetry conducted for 48 h allowed noticing that, for all levels of WTSA, the induction period was reduced due to the filler effect, and the cumulative heat from 12 h onward decreased with increasing replacement level. Additionally, the use of WTSA causes the anticipation of the aluminate peak compared to the reference specimen, resulting from the undersulfation of the system and the content of Al_2O_3 present in the calcined sludge. The CH content at 1, 3, 7, 28, and 90 days determined by TGA showed that, in general, the pozzolanic reaction is low until 7 days. Between 7 and 28 days, an expressive decrease of CH content occurred with increasing WTSA levels. However, similar quantities of CH were found between 28 and 90 days. The same trend was observed previously by Frías et al. (2014). The XRD pattern at 28 days of hydration showed the reduction of Portlandite peaks with the incorporation of WTSA, as evidenced by TGA.

Electrical conductivity presented a good relationship with the specific surface of calcined clays, constituting a rapid indirect test to evaluate the short-term activity (Tironi et al., 2013). The measurement of loss in the electrical conductivity can evaluate the consumption of CH from the first time of contact. According to Tironi et al. (2013), the densification of the cementitious matrix and, consequently, porous refinement are equally significant and are better distinguished by the SAI test. In a previous work (de Azevedo Basto et al., 2019), the loss in conductivity after 1000 s was effective to evaluate the pozzolanicity of sewage sludge ashes (SSA) calcined at different temperatures (600, 700, 800, and 900 °C). Therefore, the tests cited above to enable us to investigate the pozzolanic activity of WTSA. It is important to notice that different quantifications of reactivity may occur from one test to another (Mohammed, 2017).

In this scenario, this article proposes to produce a high-reactivity pozzolan from the WTS, whose pozzolanic properties were studied through strength and chemical-microstructural properties. In this study, analysis of calcium hydroxide consumption due to the pozzolanic reaction was carried out in pastes with hydrated lime. Generally, the

microstructural study of pozzolanic activity is conducted with Portland cement pastes. However, utilizing pastes with hydrated lime can be an interesting alternative, since it can provide quantification data of calcium hydroxide consumption. Also, most publications found in the literature show that ash could be used as a regular pozzolan. However, during the characterization phase, it was observed that the sludge had a considerable amount of kaolinite, which could increase its pozzolanic potential after being calcined.

It is essential to mention that this evaluation presents some limitations to simulate the environment of a hydrated cement past, such as the pH that is slightly different from the pH found in cement pastes and the lack of sulfates that may react with calcium hydroxide. The authors believe that studies such as this one can bring discussions about adding value to waste like WTS and safely incorporate it into the civil construction chain after incineration.

2. Experimental program

2.1. Materials

2.1.1. Water treatment sludge ash

The raw sludge used in this experimental program was obtained from the Botafogo Water Treatment Plant, located in Igarassu, Pernambuco, Brazil. In this treatment plant, the flow of treated water reaches 1500 L/s, where about 14 tons of sludge is produced daily. The sludge was collected in the settling stage, formed by the material deposited at the bottom of settling tanks. The removal of excess sludge in the decanters was performed daily; however, a deeper cleaning is carried out quarterly, where all the sludge is removed. After removal, the sludge was stored in bags with a geotextile blanket, where excess water was removed and reinserted into the treatment process. Then, the sludge was transferred to an area where outdoor drying takes place, and at the end, it was sent to licensed sanitary landfills.

After being collected, the sludge was stored in the facilities of the Civil Construction laboratory at UFPE, where it was placed for air-drying for 7 days. Then, the sludge was dried in an oven at 100 °C for 72 h. In a preliminary investigation through X-ray diffraction, kaolinite was detected in the sample of WTS; therefore, it was decided to calcine the samples to turn the kaolinite into reactive Al_2O_3 and SiO_2. In this context, three calcination temperatures were used: 600, 700, and 800 °C, where samples SA600, SA700, and SA800 were obtained, respectively. It was decided to use such temperatures because it was observed in the literature that works use calcination temperatures between 600 and 800 °C for calcination of water treatment sludge (Snellings et al., 2012; Gastaldini et al., 2015; de Godoy et al., 2019; Hagemann et al., 2019; Santos et al., 2019; Bohórquez González et al., 2020). Above 800 °C, crystallization can occur from the sludge fusion process, which is undesirable since the objective is to make the sludge viable as a pozzolan. The duration of calcination was 3 h.

As the samples showed coarse granulometry after drying and calcination, it was decided to carry out milling for 1 h in a ball mill to homogenize the material and promote the particle size reduction, increasing its pozzolanic potential. The effect of particle size on pozzolanic reactivity is well discussed in a paper by Walker and Pavía (2011). Fig. 1 presents the appearance of the samples after the processes are performed. The flowchart in Fig. 2 summarizes the processing performed for obtaining the samples.

2.1.2. Other materials

A high reactive metakaolin was used as reference pozzolan to compare the performance with WTSA. This sample of metakaolin has been used previously and showed high pozzolanic activity (de Azevedo Basto et al., 2019, 2022; Torres et al., 2020). The main mineralogical phases detected were quartz and kaolinite. Its chemical composition can be found in Table 3.

In the pozzolanicity tests with hydrated lime, calcium hydroxide with 99% purity was used to minimize the presence of other chemical elements. Quantitative analysis using Rietveld refinement showed that the hydrated lime had 95.9% calcium hydroxide and 4.1% calcite.

A limestone powder Portland cement was employed for the pozzolanic tests with cement. This cement fits in CP II-F 32 class according to the Brazilian standard ABNT NBR 16697 (2018). Results of characterization of this cement were: Blaine fineness = 403.6 $m^2.kg^{-1}$, LOI = 10.5%, specific gravity = 2.97, and the contents of CaO, SiO_2, Al_2O_3, Fe_2O_3, Na_2O, and MgO by XRF were 63.18, 11.84, 3.26, 2.46, 0.13, and 1.35%, respectively.

The fine aggregate used to prepare the mortars were standardized sand according to ABNT NBR 7214 (2015). This sand is composed of four fractions with different sizes: 25% 0.15–0.30 mm, 25% 0.30–0.60 mm, 25% 0.60–1.20 mm, and 25% 1.20–2.40 mm.

2.1.3. Characterization of anhydrous materials

Here, the characterization of the WTS and MK samples is detailed. The physical analyses were conducted from density test through the Le Chatelier flask (ABNT NM 23:, 2000), specific surface by the Blaine method (ABNT NBR 16372:, 2015), and laser granulometry. The laser granulometry test aimed to detail the particle size distribution of the pozzolans. For this test, it was used a Malvern Instruments equipment model Mastersizer 2000. Another parameter for assessing the fineness is to weigh the material retained on a 45 μm sieve. To be considered metakaolin (high reactive pozzolan), the amount of retained material must be below 10% of the total sample, as per ABNT NBR 15894-1 (2010).

The chemical and mineralogical analysis of the anhydrous materials was performed using X-ray fluorescence (XRF), X-ray diffraction (XRD), and thermogravimetry (TG) with differential thermal analysis (DTA) tests. XRD was carried out using a Bruker D2 Phaser diffractometer with the following parameters: 0.05° increment, 5.1°/min scan speed, 5–80° (2θ) scan range, 15 rpm goniometer speed, 10 mA current, 30 kV tension, and Cu anode tube with λ = 1.54 Å. The lattice parameters of this kaolinite were a = 5.2077 Å; b = 8.9537 Å; c = 7.4147 Å and the angles α = 93.028°, β = 104.15° and γ = 89.317°.

Finally, for TG analyses, it was used Shimadzu DTH60 equipment. The temperature range was 30 to 1000 °C with 15 °C.min^{-1} temperature rate and 50 mL.min^{-1} N_2 gas rate.

Fig. 1. Appearance of dried (DS100) and calcined (SA600, SA700, and SA800) water treatment sludge after grinding.

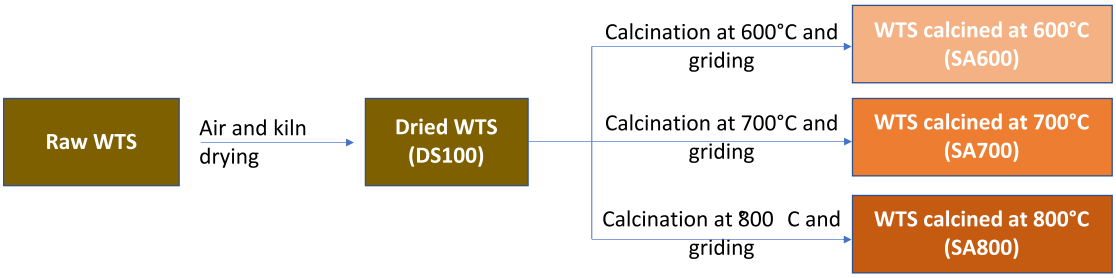

Fig. 2. Flowchart of water treatment sludge (WTS) processing.

2.2. Mortars for compressive strength tests

The preparation of mortar mixes followed the Brazilian standards of strength activity tests with hydrated lime at 7 days (ABNT NBR 5751:, 2015) and Portland cement at 28 days (ABNT NBR 5752, 2014). In the compressive strength test with hydrated lime, the mortars were prepared with pure calcium hydroxide, pozzolan, and standardized sand. The hydrated lime:pozzolan proportion is established according to Eq. (1):

$$m = 2 \bullet \frac{\delta_{poz}}{\delta_{CH}} \bullet 104g \quad (1)$$

where m is the mass of pozzolan necessary to cast three ø5x10 cm cylindrical specimens, δ_{poz} and δ_{CH} are the specific gravities of pozzolan and hydrated lime, respectively, and 104 g is the mass of hydrated lime.

The proportion of sand to binder (hydrated lime + pozzolan) is 3 in volume. The standard states that the mixes must be prepared with a water amount necessary to achieve 225 ± 5 mm on the flow table test. However, this rule is not interesting from a scientific perspective because it is not appropriate to compare mixtures with different water/binder ratios. Thus, a superplasticizer admixture was employed to prepare mortars with a water/binder fixed at 0.65. This w/b ratio was based on previous experiments, which was noticed that this was the minimum w/b necessary to cast the mortars (de Azevedo Basto et al., 2019; Torres et al., 2020). Also, Taylor (1998) reports that this w/b allows higher hydration of the pastes compared to lower w/b ratios. After mixing, the mortars were maintained in the molds for 24 h at 23 °C and then placed in an oven at 55 °C for 6 days. The minimum compressive strength to be achieved for the tested material to be considered pozzolan is 6 MPa, according to the Brazilian standard NBR 12653 (ABNT NBR 12653, 2015).

In the strength activity test with Portland cement, the mortars are prepared with 75% cement and 25% pozzolan in mass. Here, the sand: binder proportion is 3 in mass. After mixing, the mortars remained in the molds for the first 24 h at 23 °C and were then introduced into a saturated solution with lime for a submerged cure. To be considered pozzolan, the mixes with the material tested must achieve 90% of the compressive strength of a mortar elaborated with only Portland cement (ABNT NBR 12653, 2015). The compressive strength tests were conducted in an AGV-X Shimadzu load machine, with preparation and load rate according to NBR 7215 (ABNT NBR 7215, 1996). The amounts of materials utilized in the experimental program are displayed in Table 1.

2.3. Pastes for the microstructural analysis

The preparation of the pastes aimed to assess the pozzolanic activity of WTSA from a microstructural perspective. The mixtures were designed with pure calcium hydroxide, pozzolan, and deionized water, following the exact proportions of the mortars in the strength test with hydrated lime. Using calcium hydroxide instead of cement was a decision based on previous research (Torres et al., 2020) where it is stated that employing cement makes the comparison among mixtures complex since calcium hydroxide is produced continuously from the cement hydration. At the same time, this does not occur in pure calcium hydroxide-pozzolan pastes. Thus, calcium hydroxide-pozzolan pastes are better to comprehend the calcium hydroxide consumed due to the pozzolanic reaction.

Table 1
Mass amounts of the mortars prepared for the compressive strength tests.

Strength test with hydrated lime (adapted NBR 5751) – 3 cylindrical specimens (ø 5 × 10 cm)

Nomenclature	Pozzolan mass (g)	w/b ratio	CH mass (g)	Water (g)	SP (g)	Sand (g)
DS100	219.14	0.65	104	210.04	3.17	936
SA600	247.93	0.65	104	228.75	3.45	936
SA700	250.71	0.65	104	230.56	3.48	936
SA800	254.43	0.65	104	232.98	3.51	936
MK	240.50	0.65	104	223.93	3.38	936

Strength test with cement (NBR 5752) - 6 cylindrical specimens (ø 5 × 10 cm)

Nomenclature	Pozzolan mass (g)	w/b ratio	Cement mass (g)	Water (g)	SP (g)	Sand (g)
Reference	0	0.65	624	300	6.12	1872
DS100	156	0.65	468	300	6.12	1872
SA600	156	0.65	468	300	6.12	1872
SA700	156	0.65	468	300	6.12	1872
SA800	156	0.65	468	300	6.12	1872
MK	156	0.65	468	300	6.12	1872

After mixing, the pastes were cast in plastic containers and then stored in a desiccator for the first 24 h. Next, they were placed in an oven at 55 °C for 6 days. After thermal healing, the hydration stoppage was carried out by freezing the pastes in net nitrogen and then lyophilized for 24 h. Before each test, the pasted were ground and sieved through an n° 200 mesh, where only the passing material was collected for the tests. It is worth mentioning that precautions against carbonation were taken, including storing the samples in a vacuum desiccator with silica gel and barium hydroxide as sacrificial carbonator.

The microstructural tests to evaluate the pozzolanic reaction were XRD and TG. The configurations of those tests were the same used for anhydrous materials. In the XRD test, the assessment of calcium hydroxide consumption is based on the decrease of CH peak intensities, especially at 18° and 34° (2θ). The calculation methodology was the same used by Torres et al. (Torres et al., 2020) and Basto et al. (de Azevedo Basto et al., 2022). The calculation of the relative decrease of peak intensity ($RDPI_{18,34°}$) followed Eq. (2). Also, the consumption of CH (C_{CH}) was calculated according to Eq. (3):

$$RDPI_{18,34°}(\%) = 100 \times \frac{I_{CH+poz}}{I_{CH}} \quad (2)$$

$$C_{CH}(\%) = 100 \times \frac{RPDI_{18,34°} - c_{poz}}{C_{CH}} \quad (3)$$

where I_{CH} is the net peak intensity (peak intensity – background intensity) of CH in a pure hydrated lime paste, I_{CH+poz} is the net peak intensity of CH in a CH-pozzolan paste, and c_{poz} and c_{CH} are the content of

pozzolan and CH in a CH-pozzolan paste, respectively.

On TG analysis, the CH consumption was evaluated by its quantification on mass loss peak between 370 and 500 °C due to the dehydroxylation of $Ca(OH)_2$. The quantification of $Ca(OH)_2$ was performed through stoichiometric calculation. Lastly, the consumption of CH was calculated by the percentage decrease of $Ca(OH)_2$ in the paste with CH-pozzolan in relation to the same paste, considering that there was no consumption of calcium hydroxide. The CH content was corrected according to the pure hydrated lime paste, considering a slight occurrence of carbonation. The lattice parameters of kaolinite were also calculated by Rietveld refinement using G-SAS 2 v. 5072.

2.4. Electrical conductivity

In addition to compressive and microstructural tests, the pozzolanic activity of WTSA was assessed by employing the electrical conductivity test. Raask and Bhaskar (1975) and Luxán et al. (1989) proposed this method to relate the loss of electrical conductivity in a solution with pure CH and pozzolan with the consumption and CH, hence quantifying the pozzolanic activity.

The methodology used followed the conductivity tests conducted by Payá et al. (2001) and de Azevedo Basto et al., 2019. The tests were performed using a Digimed DM-32 v 2.0 conductivity meter and a Digimed stainless steel conductivity cell model DMC-001 XTX. A Raspberry Pi 3 was connected to the conductivity meter and responsible for collecting and storing the data every second. The solution was prepared with 200 mL of deionized water, 160 mg of calcium hydroxide, and 4 g of pozzolan, and the conductivity was measured during 1000 s. As some pozzolans present a significant contribution to electrical conductivity, the corrected electrical conductivity (C_{pozc}) was the result of subtracting the absolute value of the conductivity of the solution with CH + pozzolan (C_{pozCH}) with the conductivity of a solution with only the pozzolan (C_{poz}) (Eq. (4)). Finally, the loss of conductivity (%LC) was obtained by the relative subtracting between the initial conductivity (C_0) and the corrected electrical conductivity (C_{poza}), according to Eq. (5).

$$C_{pozc} = C_{pozCH} - C_{poz} \tag{4}$$

$$(\%LC)_t = \frac{C_0 - C_{pozc}}{C_0} \tag{5}$$

3. Results and discussion

3.1. Physical, chemical, and mineralogical characterization of anhydrous materials

3.1.1. Density and particle size

Table 2 shows the Blaine fineness data and the characteristic diameters (D[4,3], D[3,2], d_{10}, d_{50}, and d_{90}) obtained in the laser granulometry test. The graphics of particle size distribution can be found in Supplementary Material, Section 2.

It is observed in Table 2 that the ash fineness decreased as the temperature increased probably owing to the process of sintering of the waste, a phenomenon that usually occurs in clays. With sintering, the amorphous material turns into crystalline, increasing the hardness of the material. In this way, the material becomes more resistant to grinding. Once the milling time was fixed in this experimental program (1 h), SA800 presented a lower fineness than the other samples. In XRD results, the mineralogical phases resulting from the sintering process were present in SA800, corroborating this explanation.

Another parameter for assessing the fineness is through the sieving test. Table 2 shows that the samples that met the requirements of ABNT NBR 15894-1 (2010) were SA600, SA700, and MK. The reason for SA800 presents the higher value of material retained can be attributed to sintering processes during calcination, which increases its resistance to grinding.

Analyzing the specific gravity results obtained using the Le Chatelier flask in Table 2, a notable increase of specific gravity mass was observed after calcination, which may have occurred due to the removal of carbonaceous material from the sludge.

3.1.2. X-ray fluorescence (XRF)

The chemical composition of the materials tested is displayed in Table 3. From a chemical perspective, the sum of $SiO_2 + Al_2O_3$ over 75% indicates that the samples of WTSA contain a significant pozzolanic potential (ABNT NBR 12653:, 2015). Nevertheless, these oxides need to be preferentially in the amorphous form for a greater pozzolanic activity to occur. In comparison with the literature (Huang and Wang, 2013; Owaid et al., 2014, 2019; Pinheiro et al., 2014; Ahmad et al., 2016; de Oliveira Andrade et al., 2018; Santos et al., 2019; Bohórquez González et al., 2020), it is noticed that the WTSA used in this experimental program has more substantial amounts of Al_2O_3 than the mean values from the literature. This result is attributed to the flocculants agent ($Al_2(SO_4)_3$) used in Botafogo WTP and the presence of kaolinite, which was confirmed by XRD. Also, despite the amount of Fe_2O_3 agreeing with the literature, the sludge from Botafogo WTP presents higher amounts of this oxide than other WTPs in Pernambuco, owing to the high Fe presence in its water spring. Another important aspect to point out is the LOI of DS100, which is significantly greater than the values found in the literature (Huang and Wang, 2013; Pinheiro et al., 2014; Gastaldini et al., 2015; Ahmad et al., 2016; de Oliveira Andrade et al., 2018; de Carvalho Gomes et al., 2019; de Godoy et al., 2019; Santos et al., 2019; Bohórquez González et al., 2020; Liu et al., 2021). This occurred owing to two factors combined: the dehydroxylation of kaolinite and the presence of organic matter, which was also observed in the thermogravimetric analysis.

Regarding the presence of alkalis, it was not detected Na_2O and the values of K_2O are lower than that found in the literature. It is essential to state that the amount of those oxides is an important index of the quality of the pozzolan because they are expansive and react with some hydration products of cement, impairing the pozzolanic reaction (Berenguer et al., 2021).

3.1.3. X-ray diffraction (XRD) and thermogravimetric analysis (TG)

The dried raw sludge (DS100) presented kaolinite ($Al_2(Si_2O_5)(OH)_4$), which is a mineral commonly found in clays and provides a good indication that the sludge can change into a pozzolan after calcination, owing to the transformation of kaolinite into amorphous material. The percentages of the phases identified using Rietveld refinement were: 66.4% kaolinite, 2.8% quartz, and 30.8% amorphous material.

After calcination, the phases found were: quartz (SiO_2), which was also found in DS 100, grossite (Gr) ($CaAl4O_7$), hematite (Fe_2O_3), and

Table 2
Fineness indexes of the pozzolans used in the experimental program.

Material	Spec. gravity	Blaine fineness (m^2/kg)	D [4,3]	D [3,2]	d_{10} (μm)	d_{50} (μm)	d_{90} (μm)	Material retained on 45 μm sieve (%)
DS100	2.36	1961.01	44.211	10.678	3.592	30.684	104.618	9.85
SA600	2.67	2650.48	40.984	10.779	3.775	27.154	97.94	9.16
SA700	2.70	2430.29	48.848	11.911	4.091	33.014	116.751	9.84
SA800	2.74	1682.58	64.714	0.921	0.189	36.767	170.237	10.55
MK	2.59	2420.08	23.137	9.684	3.739	18.620	49.530	7.69

Table 3
Chemical composition results for raw and calcined water treatment sludge from this experimental program and the literature.

Oxide	This study					Other WTS/WTSA[a]							
	DS100	SA600	SA700	SA800	MK	Mean		Max		Min		Deviation	
						Raw	Calcined	Raw	Calcined	Raw	Calcined	Raw	Calcined
SiO_2	30.30	39.90	41.17	41.72	50.73	54.27	55.78	66.90	66.20	26.84	40.73	12.40	5.43
Al_2O_3	27.79	39.60	40.11	40.48	42.20	25.32	26.34	47.68	42.39	17.83	17.70	7.44	6.50
Fe_2O_3	10.25	13.13	12.76	13.41	3.62	10.04	10.13	24.00	14.60	4.91	4.86	4.29	2.29
CaO	0.06	0.10	0.09	0.10	0.18	1.34	0.73	4.32	1.99	0.13	0.30	1.48	0.53
MgO	N.D.	N.D.	N.D.	0.13	0.18	1.40	1.21	1.99	1.69	0.30	0.40	0.53	0.34
K_2O	0.37	0.46	0.48	0.55	0.20	1.84	2.76	4.20	4.40	0.34	0.99	1.29	1.30
SO_3	0.40	0.30	0.18	0.06	N.D.	0.63	0.09	3.39	0.25	0.00	0.00	1.02	0.11
P_2O_5	0.19	0.25	0.24	0.23	N.D.	0.49	0.45	0.92	0.63	0.26	0.28	0.28	0.17
LOI	29.29	4.75	3.45	1.76	2.79	10.87	3.24	19.32	4.20	6.15	1.90	4.25	0.84

[a] References used for raw WTS (non-calcined): (Huang and Wang, 2013; Owaid et al., 2014, 2019; Pinheiro et al., 2014; Ahmad et al., 2016; de Oliveira Andrade et al., 2018; Santos et al., 2019; Bohórquez González et al., 2020) and WTSA (calcined WTS): (Huang and Wang, 2013; Owaid et al., 2014, 2019; Gastaldini et al., 2015; de Godoy et al., 2019; Santos et al., 2019; Bohórquez González et al., 2020; Liu et al., 2021).

muscovite (M) ($KAl_2(Si_3Al)O_{10}(OH,F)_2$). The amorphous content increased at 600 °C due to the destruction of the kaolinite's structure and its transformation into metakaolinite or amorphous SiO_2 and Al_2O_3. The amorphous content presented a clear decrease at 800 °C owing to crystallization processes, justifying the appearance of the crystalline phases such as hematite. The diffractograms of the anhydrous materials can be found in Supplementary Material, Section 3.

Regarding the presence of kaolinite, another test where its formation can be observed is through thermogravimetry. The first mass loss, observed as an endothermic peak from 30 °C to 85 °C, is related to the loss of physically adsorbed water, representing a weight loss of 2.88%. The mass loss of 8.77%, which occurred between 200 °C and 370 °C, is resultant of the decomposition of organic matter, which represents an exothermic peak. Using equations elaborated by Kristl et al. (2016) to predict the organic matter content in soils, the organic matter content in DS100 can be estimated between 8.55 and 9.56%. The dehydroxylation of kaolinite occurs between 400 °C and 600 °C (Avet et al., 2016; Mohammed, 2017; de Godoy et al., 2019) and grants an amorphous state to the calcined sludge. The endothermic peak at 480 °C results in a weight loss of 11.79%. TG curves of DS100 are detailed in Supplementary Material, Section 4.

3.2. Compressive strength tests

Compressive strength tests were conducted in mortars prepared with hydrated lime and pozzolan (adapted ABNT NBR 5751 (2015)), and cement and pozzolan (ABNT NBR 5752 (2014)). In the strength test with hydrated lime, the samples need to achieve at least 6 MPa at 7 days to be considered suitable for application in concrete and pastes (ABNT NBR 12653:, 2015). Fig. 3 shows that the water treatment sludge ash samples met this requirement. Compressive strength means were 12.74, 15.88, and 14.40 MPa for SA600, SA700, and SA800. The mortar with DS100 presented 1.12 MPa due to the lack of material to react with calcium hydroxide and produce hardened products. The mortar with metakaolin (MK) showed a higher compressive strength mean (19.64 MPa). This was an expected result because this sample of metakaolin is highly reactive. The decrease in compressive strength between SA700 and SA800 occurred owing to the decrease of amorphous material for the pozzolanic reaction and the lower fineness.

Although the mortars with WTSA presented lower compressive strengths than MK, their values of compressive strength were at least twice the minimum required by the Brazilian standard (6 MPa).

There was a similar behavior in pozzolanic activity mortar testing with Portland cement compared to mortars with calcium hydroxide (Fig. 4). The compressive strength of the reference mortar (without inserting a pozzolanic material) at 28 days was 30.38 MPa. This reference value is used to calculate the pozzolanic activity index (PAI), which must be 90% of this reference value for the tested material to be considered a pozzolanic material for use in Portland cement (ABNT NBR 12653:, 2015). MK showed a higher compressive strength (40.83) which represents a PAI of 134%. Concerning the samples with WTSA, the PAIs were 89.63, 112.94, and 108.35% for SA600, SA700, and SA800, respectively. Although the compressive strength of SA600 did not surpass 90% of the reference mixture, it is worth stating that the Brazilian standard is more rigorous than other standards, such as ASTM C618-19 (2019).

It is worth mentioning that variations in fineness of the admixtures influenced the pozzolanic activity, which may have occurred in the comparison of the mortars with SA700 and SA800. As the water/binder ratio was fixed, variations in consistency occurred. The mortars with SA600 presented a no-slump consistency due to its high specific surface.

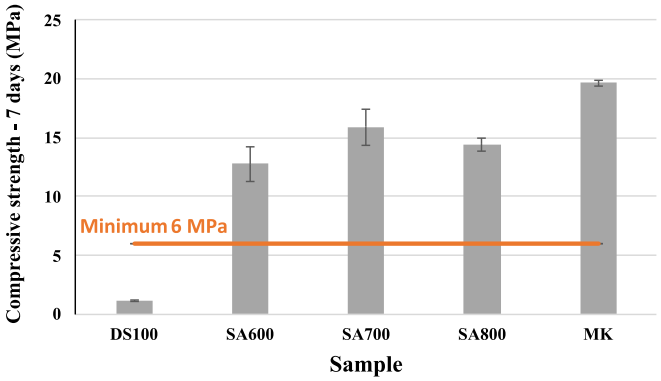

Fig. 3. – Compressive strength results at 7 days for mortars with hydrated lime and pozzolan.

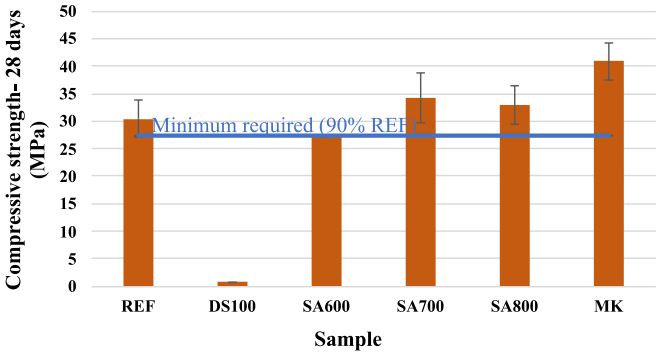

Fig. 4. – Compressive strength at 28 days for mortars with 75% cement and 25% pozzolan by mass.

This impaired consistency led to a less efficient compaction at the fresh state. However, this problem did not occur with the mortars with MK, which possess similar fineness to SA600. Although the consistency problem could be resolved using superplasticizer, the amount of polycarboxylate was already 0.98%, and increasing its content would retard the pozzolanic reactions and influence the compressive strength. Therefore, it was decided to maintain the superplasticizer content in 0.98%.

A linear equation can demonstrate the relationship between the results of compressive strength tests. Fig. 5 shows that MK, SA700, and SA800 met the requirements of both standards and can therefore be used as pozzolans. Although SA600 did not meet the requirement of NBR 12653, it does not hinder its use as a pozzolan. The only sample that cannot be used as pozzolan is DS100 due to its low reactivity.

3.3. Tests on pastes

3.3.1. X-ray diffraction

Analyzing the hydration products of the pastes with calcium hydroxide and pozzolan (Fig. 6), it is noticed the presence of gismondine ($CaAl_2Si_2O_8.4H_2O$), strätlingite ($Ca_2Al_2SiO_7.8H_2O$), and hydrotalcite ($(Mg_{0.667}Al_{0.333})(OH)_2(CO_3)_{0.167}(H_2O)_{0.5}$. The other phases (quartz, kaolinite, and calcium hydroxide) are from anhydrous materials. Strätlingite is the main hydration product because its structure is associated with C-A-S-H gel, also present in pozzolanic reaction in cement pastes.

The X-ray diffraction allows a better comprehension of the pozzolanic activity based on the peak intensities of calcium hydroxide of a paste with pure calcium hydroxide (CH). As the pozzolanic reaction occurs, the peaks of CH decrease, and the reactivity of the pozzolan can be estimated. The results of RDPI and calcium hydroxide consumption are displayed in Table 4.

As shown in Table 4, the calcined samples and metakaolin presented CH consumption over 60% due to the pozzolanic reaction. Because of its low reactivity, the peak intensity of CH was greater than the intensity value discounted by the mass ratio of calcium hydroxide and pozzolan in the paste. The calculated value of consumption was negative, which would be impossible. In this way, negative values were computed as zero. It should be mentioned that the peak intensity depends on several factors; one of them is the intensity. Therefore, errors can occur, such as the occurrence of negative values. Even so, the fact that DS100 had a null or minimal value for CH consumption corroborates the results of compressive strength with both hydrated lime and cement.

Alternatively, the other samples presented remarkable values of CH consumption. However, SA600 showed the greater CH consumption, while it was the sample with lower pozzolanic activity on compressive strength tests. A reasonable explanation for this fact is related to its fineness. The mortars were prepared with sand, which absorbs part of the mixing water and helps to reduce the consistency of the mixture. Since SA600 had the lowest particle size and the water/binder ratio was maintained for all mixtures, the consistency of the mortars was slightly impaired. As a result, the other samples had adequate compaction, which directly interferes with the resistance. This problem does not occur regarding the pastes because sand is not used, and instead of a planetary mixer, a mixer with a speed of 700 rpm was used. In comparison to another study (Torres et al., 2020) in which the same diffractometer was used, the values of RDPI reached 91.06% at 18° and 89.21% at 34° for the sugarcane bagasse ash with the maximum values of PAI on the strength test with hydrated lime and cement, respectively: 13.80 and 109.94%.

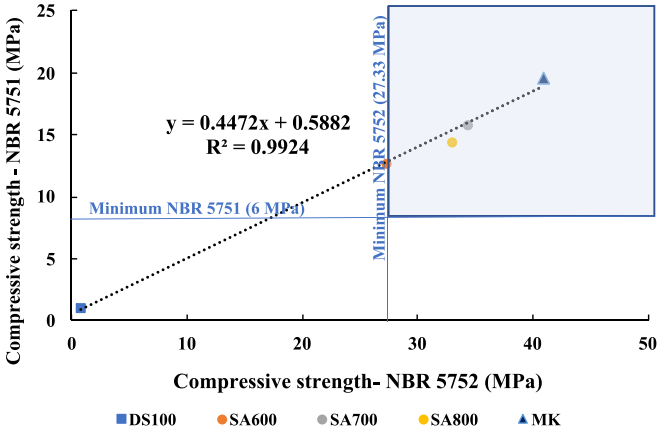

Fig. 5. – Correlation between compressive strengths of mortars on tests with hydrated lime (adapted NBR 5751) and cement (NBR 5752).

3.3.2. Thermogravimetric analysis

Another technique used to measure the CH consumption was thermogravimetric analysis. As shown in Fig. 7, the mass loss due to the dehydration of calcium hydroxide was measured between 370 and 500 °C. Fig. 7 also shows the DTG curves for pozzolan-CH pastes, indicating the peaks due to the dehydration of C-A-S-H, mainly in the form of strätlingite. Also, there are carbonates from CH and pozzolan, but no remarkable content was noticed to evidence carbonation in the pastes.

Table 5 shows the data calculated from the mass losses regarding the quantitative analysis. The results were consistent with those presented in XRD. Again, SA600 presented the highest value of CH consumption, followed by the pastes with MK, SA700, and SA800. Plus, the values of CH consumption varied between 73 and 77% and were close to that presented in XRD.

The influence of fineness on CH consumption by TG is demonstrated in Fig. 8. In addition to important aspects as amorphous content and chemical composition, the fineness also influenced the pozzolanic activity. In TG analysis, the CH consumption due to the pozzolanic reaction increases as the fineness increases. This behavior is expected; however, it only can occur since the mixing process and the consistency of the paste are adequate. In this case, the use of a high-speed mixer allowed the fluidity of the paste. On the other hand, the mixing of mortars follows a standardized protocol, in which the speed of the mixer employed is lower. Therefore, when the w/b is maintained, the mixtures with materials with higher specific surface may have presented an inadequate consistency for casting and compacting into the molds. de Medeiros et al. (2015) criticizes the standards used to measure the pozzolanic activity of pozzolans that present high fineness and reactivity, because the water content required by these pozzolans are significantly higher than regular pozzolans, which lead to misleading results.

3.4. Electrical conductivity

Fig. 9 shows an increase in the loss of conductivity (%LC) throughout time. Unlike the other methods, greater CH consumption was noticed for DS100. However, this consumption may not be attributed to pozzolanic reaction. The carbonaceous particles in the DS 100 may have combined with CH and then precipitated. This phenomenon is found in water treatment plants when lime is used as a flocculant agent (Kurniawan et al., 2006).

Regarding the calcined samples and metakaolin, it is noticed that metakaolin presented an LC lower than SA600 and SA700, but in XRD and TG, only SA600 was higher. The low loss of conductivity of metakaolin was also verified in another study (de Azevedo Basto et al., 2019). Nevertheless, when only the samples of calcined sludge are analyzed, the same trend observed in TG and XRD occurs here. Both at 100 and 1000 s, SA600 presented superior values than SA700 and SA800 (Table 6).

Comparing the values of LC and variation of electrical conductivity (Δ) at 100 s, de Azevedo Basto et al. (2019) found the ranges of 42–11%

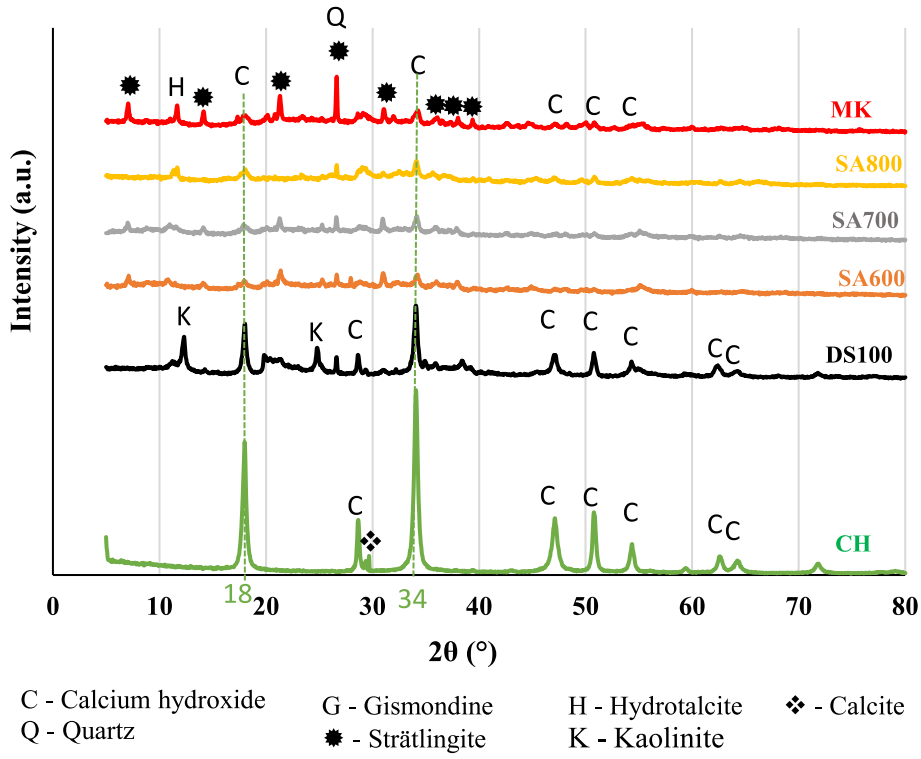

Fig. 6. – Diffractograms of pastes with calcium hydroxide and the samples CH, DS100, SA600, SA700, SA800, and MK.

Table 4
Values of calcium hydroxide peak intensities, RDPI, and calcium hydroxide consumption at 18 and 34° for samples DS100, SA600, SA700, and MK.

Sample	Peak intensity		RDPI		CH consumption (%)	
	18°	34°	18°	34°	18°	34°
DS100	1775.44	2405.80	61.45	63.20	0.00	0.00
SA600	254.92	434.88	94.46	93.35	81.09	77.27
SA700	288.17	554.02	93.74	91.52	78.45	70.82
SA800	355.12	586.36	92.29	91.03	73.17	68.79
MK	288.50	443.63	93.73	93.21	79.05	77.31

for LC and 2.47–0.68 mS/s for Δ_{100}. The ranges obtained here were superior, which can be explained by the superior pozzolanic activity of the WTSA. However, at 1000s, the values found in the same paper achieved 88.3% for LC and 5.11 mS/s for Δ_{1000}, which did not occur for WTSA.

4. Conclusions

From the results, the following conclusions are drawn:

- The sum of the oxides SiO_2, Al_2O_3, and Fe_2O_3 met the Brazilian standard of requirements for pozzolans. Also, the presence of 66.4% of kaolinite in DS100 indicated its pozzolanic potential after calcination. XRD confirmed this once the transformation of kaolinite into metakaolinite was detected. However, a little sintering may have occurred at 800 °C, suggesting that it is the threshold temperature for producing pozzolan;

- The physical characterization of WTSA samples showed that the density increased as the calcination temperature increased. Furthermore, the Blaine fineness varied between 1682 and 2650 $m^2.kg^{-1}$ because the resistance to grinding increased with increasing temperature. In the sieving test, all samples with the exception of SA800 showed an amount of material retained less than 10% on sieve 45 μm.

- In compressive strength activity tests, metakaolin presented the higher values of compressive strength, followed by SA700, SA800, and

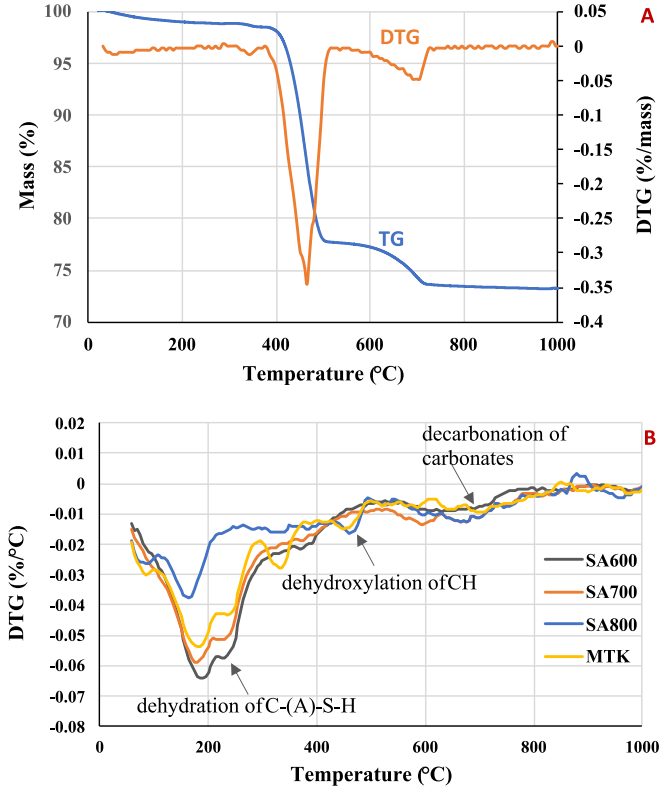

Fig. 7. TG and DTG curves for calcium hydroxide paste (A) and DTG curves for CH-pozzolan pastes.

SA600. In the strength tests with hydrated lime, the samples presented at least twice the minimum value (12.74 MPa) established by the Brazilian standard (6 MPa). In tests with Portland cement, the lowest pozzolanic

Table 5
Mass loss, CH measured, and CH consumption for pozzolan-CH pastes measured by TG.

Sample	Mass loss between 370 and 500 °C (%)	CH measured (mg)	CH consumed (%)
SA600	1.366	5.62	77.21
SA700	1.522	6.26	74.41
SA800	1.552	6.38	73.63
MK	1.475	6.07	75.91

activity index (PAI) was presented by mortar with SA600 (89.63%);

- When XRD and TG were performed in pastes, the pozzolanic potential of SA600 was clear. All tests on pastes showed that SA600 was the sample with the greatest values of CH consumption. All samples, except for DS100, presented the potential to be used as a high-activity pozzolan. The results also show that XRD and TG tests on pastes with pure calcium hydroxide are adequate to compare pozzolanic activity between pozzolans;

- Toward electrical conductivity, this test presented coherent results in pozzolans with low organic matter content. However, in samples with carbonaceous content, there might be a chemical interaction between calcium hydroxide and organic matter, leading to misleading results. Also, different pozzolans may interact differently with calcium hydroxide, so it is appropriate to compare only pozzolans with the same type.

- This study showed that the calcined samples could be employed as high-reactivity pozzolans. Also, this research presented the importance of investigating the pozzolanic activity allying standardized and microstructural/chemical tests.

CRediT authorship contribution statement

Tiago M.S. Agra: Methodology, Formal analysis, Investigation, Formal analysis, Writing – original draft, Writing – review & editing. **Victor M.E. Lima:** Methodology, Formal analysis, Investigation, Formal analysis, Writing – original draft, Writing – review & editing. **Priscilla E.**

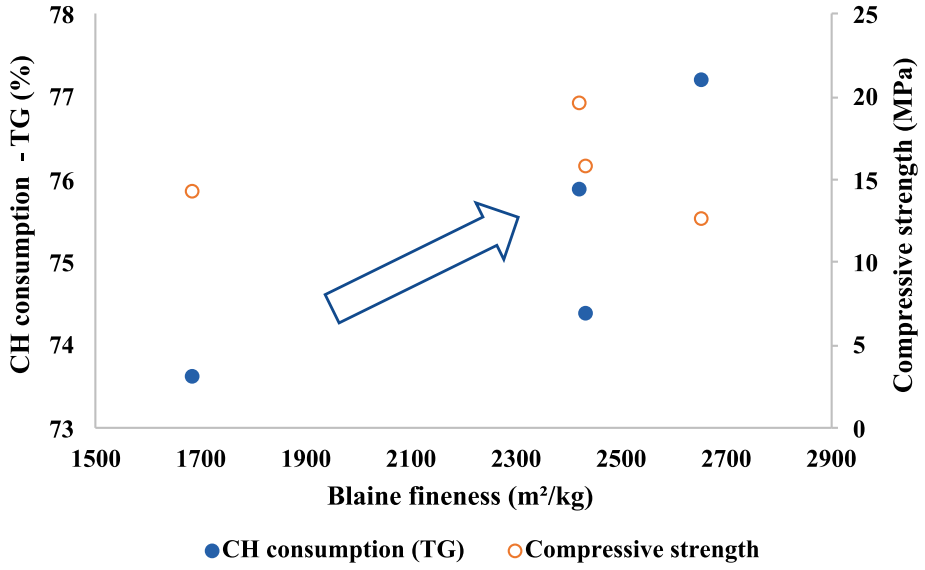

Fig. 8. – Relationship between Blaine fineness and CH consumption by TG and compressive strength with hydrated lime.

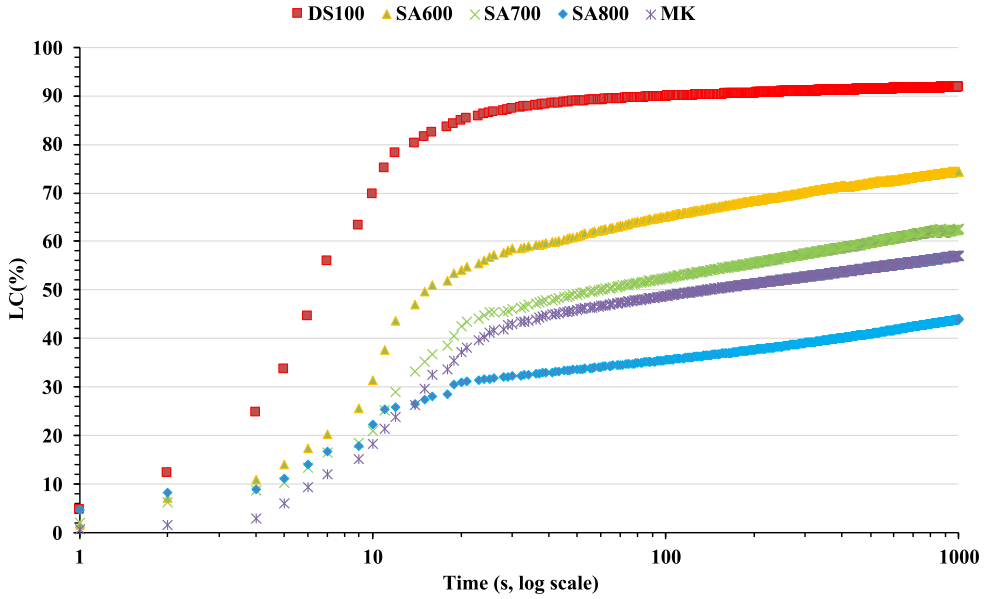

Fig. 9. Loss of electrical conductivity (LC) in calcium hydroxide solutions for DS100, SA600, SA700, SA800, and MK.

Table 6
Values of loss of electrical conductivity (%LC) and variation of electrical conductivity (Δ) in calcium hydroxide solution for samples DS100, SA600, SA700, SA800, and MK.

Time (s)	DS100	SA600	SA700	SA800	MK
100 (%LC)	89.82	65.09	52.41	35.51	48.67
1000 (%LC)	91.69	74.45	62.51	43.97	57.03
100 (Δ)	4.75	3.48	2.50	1.87	1.89
1000 (Δ)	4.85	3.98	2.98	2.32	2.21

A. Basto: Formal analysis, Writing – original draft, Writing – review & editing. **Antonio A. Melo Neto:** Methodology, Formal analysis, Writing – original draft, Writing – review & editing, Supervision.

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

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.clay.2023.106870.

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