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Evaluation of the use of silica-alumina refractory waste as a supplementary cementitious material

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Abstract

The use of supplementary cementitious materials (SMCs) is one of the three essential factors for the sustainability of the cement industry. To reduce CO_2 emissions, several studies have introduced new methods for the utilization of industrial wastes such as the addition into cementitious materials. The objectives of this study were to chemically and physically characterize refractory ceramic industry waste and determine its viability as an SMC. The pozzolanic characteristics or SMC characteristics of the refractory waste were investigated using standard tests. The results revealed that the refractory waste was mainly composed of Al_2O_3 and SiO_2. The mineralogical analysis revealed that the mullite was the major phase of the waste, followed by small amounts of cristobalite and quartz phases. The results of the modified Chapelle test and pozzolanic activity index with lime after 7 days indicated that the waste did not meet the minimum pozzolanicity required by the standard. However, the waste improved the mechanical resistance of the cement specimens containing the waste after 28 days compared with the reference sample, indicating the promising potential of the material for this application. This result indicated that the refractory waste shows a filler type effect, and thus, can be employed as an SCM.

Keywords: refractory waste, pozzolanicity, sustainability, supplementary cementitious material.

INTRODUCTION

Concrete is now the second most consumed material by humans, the first being water. The high consumption of concrete has a positive effect on the current socio-economic growth and development of the human population. In recent years, the global gross cement production has increased by 2.5% annually [1]. However, cement production processes have negative impacts on the environment. For example, according to the British research institute Chatham House, more than 4 billion tons of cement are produced each year, which results in approximately 8% of the global CO_2 emission [2]. This indicates that cement production contributes significantly to global warming. Brazil, one of the major producers of Portland cement, produced approximately 71 million tons of cement in 2013. However, due to the economic crisis, the cement production reduced to 54 million tons in 2018, of which 90% of the cement contained some addition [3]. Due to the decrease in the natural resources used for the production of cement, the use of supplementary cementitious materials (SCMs) such as minerals and industrial residues has emerged as an alternative [4]. SCMs are used as filling materials in cement to improve the technical properties of cementitious materials, reducing the quantity of Portland cement in concrete. Based on the chemistry of the hardening process, mineral additions used in concrete or mortars can be classified as hydraulic materials, pozzolanic material, and fillers [5, 6].

Granulated blast furnace slag is the major source of hydraulic additions. It exhibits inherent hydraulic properties, indicating that they exhibit setting and hardening properties under water, similar to that in Portland cement. In addition, the chemical reaction between slag and water forms hydrated phases that are similar to those found in Portland cement, with the dominating C-S-H phase [6]. According to the American standard ASTM C618-19 [7] and the Brazilian standard NBR 12653:2015 [8], pozzolanic materials can be defined as siliceous or siliceous and aluminous materials that, by themselves, possess little or no cementitious value, but, in the finely powdered form and in the presence of moisture, react chemically with calcium hydroxide (lime) at ordinary temperature to form compounds with cementitious properties. Fillers are additives that remain inert in cement. In traditional literature, limestone has been commonly regarded as an ‘inert filler’; however, compared with the mineral additions of the first two groups, the degree of reaction of $CaCO_3$ in cement paste is low. The contribution of limestone to the hydration reactions is significant when it is co-milled with the clinker, although in relatively small amounts. Nowadays, limestones are recommended in the European standard EN 197-1 as mineral additions, together with pozzolanic additives, and can be classified as an SCM similar to granulated blast furnace slag and fly ash [6, 9]. The use of additives in concrete significantly reduces the environmental impacts of concrete and shows economic and geological benefits [10]. The use of pozzolanic materials from various industrial or natural sources as additives has been reported to show satisfactory results for the manufacture of cementitious materials. Studies have reported excellent results with the use of red ceramic waste [11], rice husk ash [12, 13], sugarcane bagasse ash [14, 15], waste from kaolin industries [16], metakaolin [17, 18], and several others

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[19, 20] as additives in cement. Accordingly, studies have observed that cement matrices allow a certain diversity of materials from residues or other sources that are not natural, provided that these materials contain aluminosilicates and possess a certain degree of reactivity.

The South of Brazil is a region that has numerous factories producing ceramic tiles and refractories with high waste generation. Refractory materials are mostly shaped ceramic materials that maintain their chemical and physical characteristics even when subjected to high temperatures or thermomechanical stresses. Generally, these refractory materials are used as insulating coatings in furnaces for metal melting, glass making, metallurgical heat treatments, among others, or for more common purposes such as thermal insulators used in fireplaces [21, 22]. Approximately 70% of the refractory materials produced are used by the steel industries as internal linings for ovens and transfer vessels. Refractory waste is commonly disposed of in landfills [23]. According to one study, approximately 28 million tons of refractory waste is generated annually on a global scale, of which only 7%-10% is recycled [24]. Several studies have reported new methods for the efficient utilization of refractory waste to reduce its environmental impacts. For example, one study proposed the use of refractory brick waste as supplementary material in cement for use in concrete. In addition, the use of three types of refractory waste substitutes for silica fume was highlighted, and the performance of refractory-added cement in concrete was found to be satisfactory [25]. Another study demonstrated the use of refractory waste in concrete mortars as an aggregate material to replace one of the aggregates normally used [26]. Furthermore, a recent study on the production of calcium aluminate cement containing 50%-70% slag from the steelmaking process in the clinker mixture revealed that the cement containing 60% of the residue showed superior resistance to commercial cement [27].

These findings indicate the promising potential of refractory waste as SCM in cements owing to their composition and physicochemical characteristics to reduce CO_2 emissions, and thus, make the planet more sustainable. The silica-alumina refractory residues are acidic (rich in silica and alumina), indicating that it can be used at lower operating temperatures than other refractory materials and has a lower production cost than that of basic refractory materials [28]. In addition, the chemical composition of this type of silica-alumina refractory residues meets the requirements for pozzolanic materials required by a Brazilian standard [8]. Based on the findings of previous studies on refractory waste and its application as SCM in cements, the aim of this study was to evaluate the potential of a ceramic industrial residue generated in the production of silica-alumina refractories as an SCM in cement through standard tests of SCMs and pozzolanic activity and compare it with other contemporary SCMs. This study aims to provide information on the reuse and recycling of wastes that are not widely used, thus reducing the amount of waste and at the same time partially replacing materials, such as cement, and consequently reducing environmental impacts.

EXPERIMENTAL

Molded silica-alumina refractory waste supplied by the company Librealto Refratarios located in the city of Lauro Müller, in the south of Santa Catarina, Brazil was used as the primary material. Kaolinite is the major mineralogical component in the silica-alumina refractory. The constituent oxides of kaolinite undergo chemical reactions during heat treatment, which favors the nucleation and growth of mullite crystals. As the refractory manufacturing temperature increases (below melting temperature), the mullite phase content in the material also increases, and it is the main phase responsible for the mechanical strength [29]. The silica-alumina waste was generated after firing the refractory at 1500 °C as defective bricks (by cracks, breaks, unusual sizes); typically, they are sent to a disposal pile and ground in a hammer mill. Part of the generated waste is returned to the process and the rest is disposed of. The waste was characterized by the company and classified according to its hazardousness as waste Class II A - non-inert, according to the Brazilian standard NBR 10004 [30]. The supplied material was already finely ground. Approximately 50 kg of waste was purchased. Then, the waste was divided into 4 parts, and the sample was reduced to 10 kg to carry out the work on a laboratory scale. After this, the sample was dried in an oven (30 L, El 1.1, Odontobras) at 70±5 °C for 24 h.

First, the chemical and physical characterization of the residue was carried out by a moisture content test according to a standard [31]. For this, we used a scale (AD3300, Mars) and an oven. The residue was determined using a 45 μm (325 mesh) sieve (Bertel) following the standard NBR 15894-3 [32]. The density was calculated using a pycnometer with helium gas (Ultrapycnometer 1200e, Quantachrome), and the samples were dried at 100 °C in the oven for 24 h. The chemical composition of the residue was determined by X-ray fluorescence spectroscopy using a spectrometer (WRFDX Axios Max, Panalytical). For this analysis, the sample was dried in the oven until the mass was constant, after which the sample was sieved through a 45 μm sieve. Subsequently, the sieved material was molded into a molten tablet (a glass disc prepared by mixing the sample with a melting pattern); thereafter, the molten tablet was analyzed in the equipment, where the reading of the previously specified elements was carried out. For identifying the mineralogical phases, the phase quantification was characterized by X-ray diffraction (XRD) using the Rietveld method. The test was performed using a diffractometer (D8 Advance ECO, Bruker). The X-ray source of this equipment was a copper anode tube with a characteristic emission line of 1.54 Å/8.047 keV ($CuK\alpha_1$) operating with a high-speed detector (SSD 160). The analysis was performed in the θ-2θ scan, in the range from 3-100°, at a step size of 0.02°, and speed of 3 $°.min^{-1}$. The identification of the crystalline phases was performed using the Inorganic Crystal Structure Database (ICSD). The amorphous percentage and quantification of the phases were determined using the Rietveld method with an internal standard. For this, the powder sample (particle size <60

μm) was mixed with the same mass of α-Al_2O_3 (99.99%) to obtain a mixture of 50% internal standard. The mixing was carried out in a laboratory mixer, with a container of approximately 250 cm for 4 h. Phase quantification was performed using the GSAS software with the EXPGUI interface. The surface area was measured by the Brunauer-Emmett-Teller (BET) method and was performed with an adsorptometer (Nova 1200e, Quantachrome) using nitrogen adsorption. The residue underwent heat-treatment at 300 °C for 3 h for the degassing process, where any moisture present in the sample and some impurities that may interfere with the adsorption and desorption of nitrogen gas were removed. For the laser particle size distribution analysis, a granulometer (mod. 1064, Cilas) capable of measuring particles between 0.04 and 500 μm was used. A sodium polyacrylate dispersing agent (Disperlan) was used. Differential scanning calorimetry and thermogravimetry (DSC/TG) were performed simultaneously in a thermal analyzer (STA 449 Jupiter, Netzsch) with platinum crucibles under a synthetic air atmosphere up to 1200 °C at a rate of 10 $°C.min^{-1}$ and air flow of 10 $mL.min^{-1}$.

After the characterization of the waste, standard tests were performed to verify the pozzolanicity of the residue by determining the content of fixed calcium hydroxide (modified Chapelle test) [33]. The test was performed using a Dubnoff orbital bath (TE-0532, Tecnal) and a Wagner shaker (TE-160, Tecnal). The pozzolanic activity index with lime after 7 days was performed according to a standard [34], molding 3 cylindrical specimens with a diameter of 50 mm and a height of 100 mm, and adding an amount of water to reach a consistency index of 225±10 mm, measured on a flow table. The evaluation of the mechanical strength with cement after 28 days was carried out according to a standard [35], measuring the compressive strength of reference specimens (without the addition of waste) and specimens containing 15% by mass of the refractory waste. The cement used was CP II-F-32 as required by a standard [36], in addition to the 4 fractions of 468 g of sand (117 g for each fraction). The granulometric profile of the sand was: 25% by mass retained between 2.4 and 1.2 mm sieves; 25% by mass retained between 1.2 and 0.6 mm sieves; 25% by mass retained between 0.6 and 0.3 mm sieves; and 25% by mass retained between 0.3 and 0.15 mm sieves [37]; water was added until a consistency index of 180±10 mm was reached. The 6 specimens were molded in a cylindrical shape, with a diameter of 50 mm and a height of 100 mm following a standard [38]. For both tests, a press (SSH300, Emic) was used with TRD 30 load cell and a crosshead speed of 0.25 $MPa.s^{-1}$ for the mortar specimens. Finally, the microstructure of refractory waste and cementitious specimens with and without the addition of refractory waste ruptured at 28 days was analyzed using a scanning electron microscope (SEM, EVO MA10, Zeiss). The images were taken at 5000x magnification. For the analysis, the samples were subjected to a vacuum metallization process where they were coated with gold using a sputtering (QR 150ES, Quorum). To confirm the phases formed after 28 days of curing, a mineralogical analysis was made by XRD.

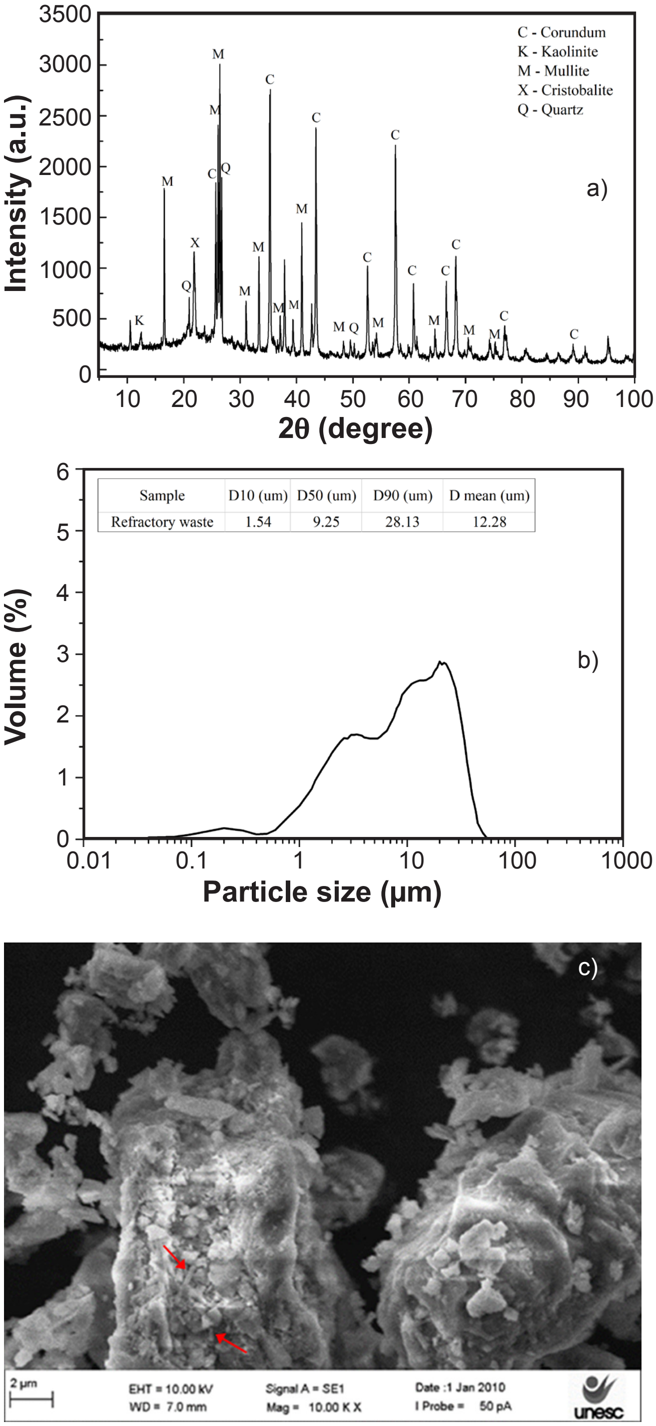
RESULTS AND DISCUSSION

First, the results of the chemical and physical characterization of the refractory waste are presented, followed by the results obtained through the standard tests to verify the pozzolanic activity of the sample. The moisture content of the residue was 2.3%, which met the standard requirements for pozzolanic materials [8], which requires a maximum moisture content of 3%. In addition, the waste retained in the 45 μm sieve was 14.13%, which also met the requirements of the Brazilian standard NBR 12653 [8]. The density of residues has a direct influence on the degree of compaction of the matrix in mortars and concretes; therefore, it is important to analyze the density to understand the properties of residues [39]. The density of the refractory waste was 2.98±0.14 g/cm^3. Generally, insulating refractories are characterized by low density, whereas alumina-rich refractories are denser and stronger. A previous study on Algerian kaolin-based refractories reported a density of 2.75 g/cm^3 [40], while another study reported a density value of 2.95 g/cm^3 for silica-alumina refractories, such as calcined kaolin-based concrete [41]. The density of the refractory waste in this research was consistent with those of previous studies on cement containing SCMs such as acidic slag (2.98 g/cm^3), fly ash (3.01 g/cm^3), and diatomaceous earth (3.03 g/cm^3) [42]. The results of the chemical analysis by X-ray fluorescence spectroscopy are shown in Table I. The obtained results met the chemical requirements of the Brazilian standard [8]. As the residue was a silica-alumina refractory, the residue was mainly composed of SiO_2 (50.51%) and Al_2O_3 (42.62%), with Fe_2O_3 (1.16%) and K_2O (1.83%) as main impurities. In addition, the loss on ignition of the residue was 2.48% because the material was calcined at a high temperature. This value is close to those of the calcined clays used as pozzolanic materials. In addition, the silica, alumina, and iron oxide levels in this study were close to those of two calcined clays investigated in a recent study on the chemical composition of several SCMs [43].

Fig. 1a shows the X-ray diffractogram of the mineralogical analysis of the refractory waste. The mineralogical composition of the refractory waste based on kaolinitic clays consists majorly of mullite ($3Al_2O_3.2SiO_2$) and silica, both in the crystalline form (quartz or cristobalite)

Table I - Chemical composition (wt%) of the refractory waste.

SiO_2	Al_2O_3	Fe_2O_3	K_2O	Na_2O	CaO	MgO	TiO_2	LOI
50.51	42.62	1.16	1.83	0.43	0.39	0.27	0.37	2.48



Sample	D10 (um)	D50 (um)	D90 (um)	D mean (um)
Refractory waste	1.54	9.25	28.13	12.28

Figure 1: XRD pattern (a), particle size distribution curve (b), and SEM image of the particles (c) of refractory waste.

and in the amorphous phase. The mullite phase results from the transformation of kaolinite ($Al_2O_3.2SiO_2.2H_2O$) during the thermal cycle at high temperatures [44]. Therefore, because it is a material processed at a high temperature (approximately 1500 °C), the diffractogram of the refractory waste was mainly composed of the mullite phase (ICSD 74008). The Rietveld method revealed that the residue comprised 83% of the mullite phase. The presence of the mullite phase significantly improved the properties of the residue such as high-temperature stability and chemical stability, high refractoriness, good strength, and toughness [45]. The cristobalite (ICSD 34923) and quartz (ICSD 24851) phases were also found in contents of 10% and 7%, respectively, and the quartz phase was considered an impurity. The quality of the refinement was observed by the weighted index (R_{wp}) of 12.11 and the equivalent chi-square index (GOF) of 3.48. The phases observed in this residue have been reported in studies as the 3 main phases found in silica-alumina refractories [29]. The corundum peaks indicated in the diffractogram referred to the mixture with alumina standard for phase quantification. This phase was neglected in the Rietveld method. An additional kaolinite peak was also observed at 13°, which could be attributed to the poor sintering of some bricks during the firing process. However, this phase has not been quantified, possibly due to its low quantity in comparison to other phases. The mullite and cristobalite phases have also been observed as major phases in refractory concrete in previous studies [46]. However, we did not observe an amorphous phase in the Rietveld quantification. The presence of an amorphous phase is one of the main characteristics of pozzolanic materials because it affects the degree of reactivity of the mineral addition [47]. However, it is important to analyze the

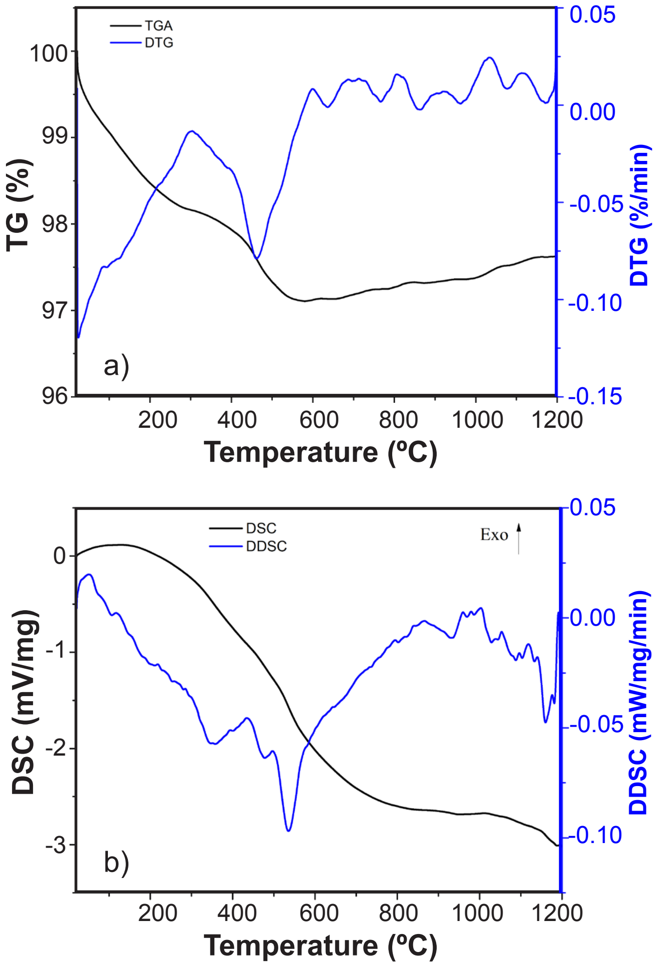


Figure 2: TG/DTG (a) and DSC/DDSC (b) curves of the refractory waste.

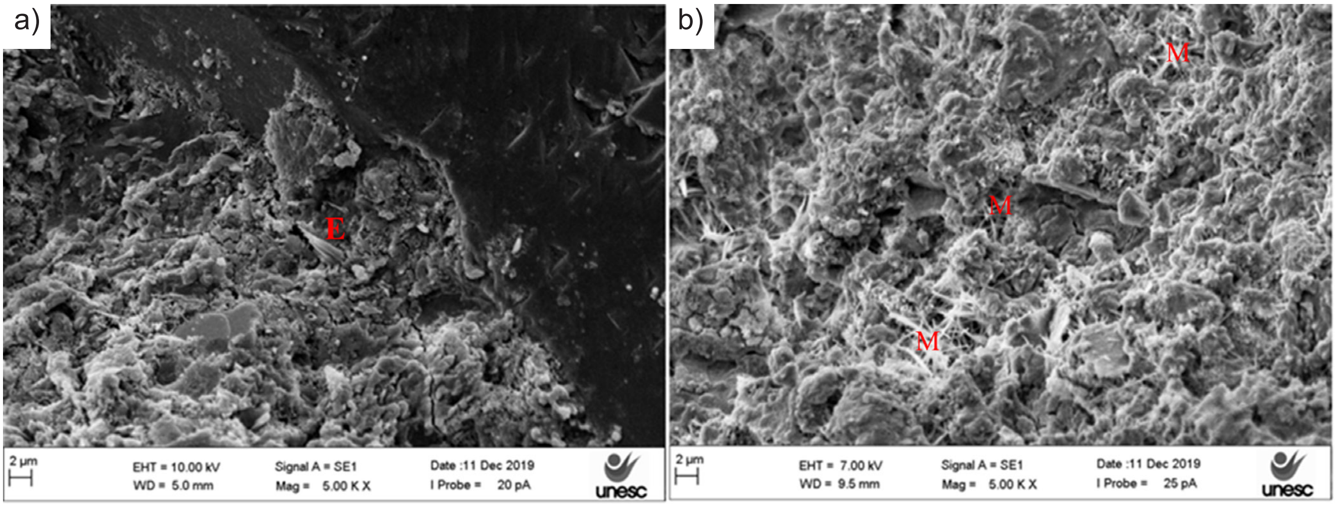


Figure 3: SEM images of the surface of the cementitious specimens: a) sample without refractory waste; and b) sample containing refractory waste. E: ettringite; M: mullite.

interaction of the refractory waste with Portland cement to investigate the potential of the refractory waste to reinforce the mechanical strength of Portland cement owing to its mullite crystals [48].

The particle size distribution curve of the refractory waste (Fig. 1b) showed a trimodal distribution with an average diameter of 12.28 µm, with 90% of the particles having a diameter less than 28.13 µm. This granulometric distribution can be attributed to the calcination of the residue at high temperatures and its high hardness during milling. Similar granulometric distribution ranges have been observed for pozzolans and other SCMs such as calcined clays and limestone fines [43, 49, 50]. The refractory waste in this study had a specific surface area of 3.44 m^2/g, which was significantly higher than the specific surface area of cement (1.94 m^2/g), both measured using the same technique (BET) [51]. The waste showed a significantly higher value, which can increase its reactivity as a pozzolanic material owing to the increase in the contact area. The specific surface area of the residue was consistent with previously reported values for other SCMs such as diatomaceous earth (4.30 m^2/g) [52] and limestone fines (2.97 m^2/g) [50]. Fig. 1c shows the microstructure of the refractory waste. Dense particles and some small needle-shaped particles were observed. These needle-shaped particles are characteristic of the mullite phase [53]. Thermal analysis of the refractory waste was performed by plotting TG/DTG (Fig. 2a) and DSC/DDSC (Fig. 2b) curves. In Fig. 2a, a small mass loss of 2.6% can be observed between 100 and 500 °C. In addition, as shown in Fig. 2b, there were no significant thermal events, as the material had already undergone a firing process at a higher temperature.

One standard test to determine the fixed calcium hydroxide content is the modified Chapelle method [33]. In Brazil, the Chapelle test is not a requirement to classify a material as pozzolanic (except for metakaolin). However, this test was carried out to determine if the refractory waste could have any pozzolanic activity. If the material shows any type of chemical activity with calcium oxide (CaO) in an aqueous medium, it develops cementitious compounds that harden with time, thus, confirming its pozzolanic activity. The refractory waste consumed 230 mg of CaO/g sample. This value was lower than those of common pozzolanic materials such as sugar cane bagasse ash (900 mg CaO/g sample) [54], metakaolin (771 mg CaO/g sample) [55], and iron ore (382.85 mg CaO/g sample) [56]. However, compared with the sugar cane bagasse ash and metakaolin, the value was close to that of the iron ore. Another standard test was the lime pozzolanicity index after 7 days. According to the standard for a material to be considered pozzolanic, it must have a minimum strength of 6 MPa after 7 days [34]. The refractory waste achieved a strength of 5.6±0.4 MPa, which was lower than the minimum required. This indicated that the refractory waste cannot be considered as a pozzolanic material. However, it is important to analyze the waste material for any SCM-related characteristics.

The mechanical strength test with cement after 28 days showed a positive result with the addition of the refractory waste. The mechanical resistance of the sample containing the refractory waste reached an average strength of 23.5±0.1 MPa. This value was similar to the reference specimens (without the addition of waste), which was 23.1±0.4 MPa. According to the standard, the specimens must achieve a 75% resistance in relation to the reference specimens. Although the material did not show pozzolanic activity in other standard tests, the good resistance characteristic of the refractory waste indicated that it can be regarded as a filler SMC. The improved resistance characteristics of the waste can be attributed to the characteristics of particle size and specific surface area, which improved the particle packing of the samples, thereby reducing the empty spaces between the particles and improving the mechanical resistance. To further understand the effect of the residue on cement, the samples were analyzed by SEM. Fig. 3 shows micrographs of the samples with and without the refractory residue after 28 days of curing. Ettringite needles (indicated with letter E in Fig. 3a) were observed in the micrograph of the cement without the refractory waste. This characteristic phase of

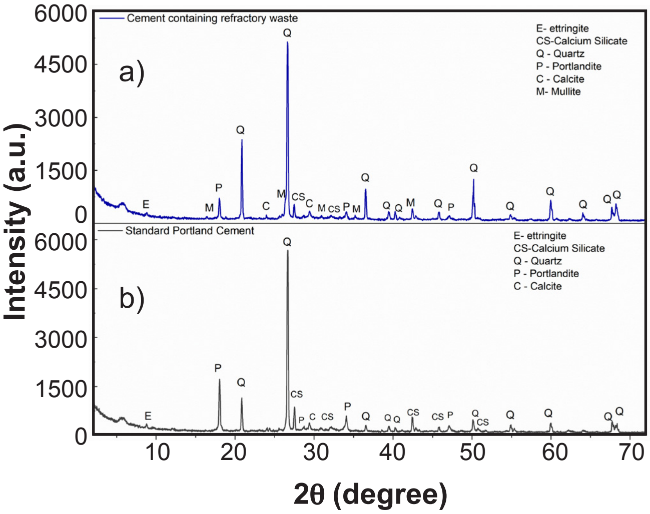


Figure 4: XRD patterns of the cementitious samples with (a) and without (b) refractory waste after 28 days of curing.

the cured cement begins to form minutes after the hydration starts, thus developing the initial resistance, resulting from the hydration of the aluminates in the presence of sulfates. In the micrograph of the cement containing the refractory waste (Fig. 3b), the amount of grains in the form of needles significantly increased; however, they were mullite needles (letter M) from the waste that intertwined the cement particles, forming a network, which increased the mechanical resistance of the cement [46]. In addition, the mullite phase increases the mechanical strength of materials and can be used as reinforcement in several composite materials [45].

Fig. 4 shows the X-ray diffractograms of the mineralogical analysis of the cementitious samples with and without the refractory waste after 28 days of curing. The phases found in the sample containing refractory waste were ettringite (ICSD 155395), portlandite (ICSD 01-0760-571), mullite (ICSD 74008), calcium silicate (PDF 00-033-0302), calcite (ICSD 01-083-0578), and quartz (ICSD 24851). The presence of the mullite phase was coherent with the addition of refractory waste and the needles observed in the microstructure shown by the SEM image (Fig. 3b). The other phases found in both samples were the characteristic phases of the hydration process of Portland cement [57].

CONCLUSIONS

The results of this study indicated that silica-alumina refractory waste can be considered as a potential supplementary cementitious material (SCM). Although it is required that SCMs possess certain pozzolanic activity, which guarantees a better performance when used in mortars and concrete, this silica-alumina refractory waste physically improved the mechanical property of concrete without any direct chemical interaction, indicating that it can be regarded as a filler type SCM. The filler effect of the refractory waste improved the packing of the particles in concrete, thus reducing the pores or empty spaces between the particles, which in turn increased the mechanical strength of the concrete due to a decrease in stress concentrators, such as pores. The modified Chapelle test, which was carried out to investigate the pozzolanic characteristics of the material, revealed that the refractory waste consumed 230 mg CaO/g sample. In addition, according to the Brazilian standard NBR 12653, refractory waste met the chemical requirements of moisture content, which was less than 3%, a loss on ignition of less than 10%, and the sum of silica, alumina, and iron oxide greater than 70%. Regarding the physical characteristics, the waste met the requirements of the material retained in the 45 μm sieve and the pozzolanic activity index with cement after 28 days. However, the sample did not show good performance in relation to the lime test after 7 days, as it did not achieve the minimum requirement of the standard. These tests are based on chemical interactions and are strongly linked to the reactivity of the material. As this material is generated during a high-temperature firing process, even if the firing does not happen properly, the material is totally crystalline, which makes this material practically inert in processes that occur at room temperature or relatively low temperatures such as the curing of cementitious materials. Finally, the refractory waste showed a filler effect, indicating its promising potential as an SCM. The findings indicated that refractory waste can be used efficiently as a filler in Portland cement-based materials, i.e. utilizing waste generated in the refractory industries.

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